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THE 7th INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS,

ROCAM 2012

28-31 AUGUST 2012, BRASOV, ROMANIA

Held under the auspices of
International Organization for Crystal Growth

An International Conference on Advanced Materials
and Crystal Growth with special topics
on nano-, multifunctional and photovoltaic materials

ABSTRACT BOOK

EDITURA UNIVERSITATII DIN BUCURESTI 2012

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FOREWARD

The **7th INTERNATIONAL CONFERENCE ON ADVANCED MATERIALS, ROCAM 2012** will be held at the International Conference Centre of the “Transilvania” University, Brasov, August 28-31, 2012, under the auspices of the **International Organization for Crystal Growth.** The **International Summer School on Crystal Growth and Photovoltaic Materials** will be a parallel event, under the auspices of the **International Union of Crystallography**, Aug 27 - Sept 1, 2012, at the same location.

The main organizer of **ROCAM** events is the Romanian Materials Science - Crystal Growth Society**,** a non-profit association, affiliated in 1994 as a National Association at the International Organization for Crystal Growth. Other important co-organizer institutions of **ROCAM 2012** are Transilvania University of Brasov, Romania, Leibniz Institute for Crystal Growth (IKZ), Germany, University of Bucharest, Faculty of Physics, Romania, National Institute of Materials Physics (NIMP), Romania and National Institute for R&D in Electrical Engineering ICPE-CA, Romania.

ROCAM 2012 conference covers four days in three parallel sections, including eight topics: 1. Crystal Growth Fundamentals and Applications, 2. Organic Materials: Advanced Methods, Advanced Applications, 3. Biocompatible Materials and Applications, 4. Thin Films of Multifunctional Materials for Advanced Applications, 5. Advanced Ceramics: From Micro- to Nanoscale, 6. Advanced Materials for Photovoltaics and Applications, 7. Modelling and Characterization of Advanced Materials, 8. Advances in Surface Science and Engineering.

There are four plenary lectures delivered by the following distinguished personalities: Prof. Roberto FORNARI, President of the International Organization. for Crystal Growth, Germany, Thomas KUECH, Ed.-in-Chief J Crystal Growth, University of Wisconsin - Madison, USA, Prof. Joseph GREENE, Ed.-in-Chief Thin Solid Films, University of Illinois, Urbana, Illinois, USA, Prof. Martin GREEN, Ed-in-Chief Progress in Photovoltaics: Research and Applications, University of New South Wales, Sydney, Australia.

Organizing Committee of the Conference gratefully acknowledge the active contribution of the following personalities: Prof. Roberto FORNARI, director of IKZ-Leibniz Institute for Crystal Growth and Humboldt University Berlin, Germany, Prof. Hanna DABKOWSKA, McMaster University, Canada, Prof. Thierry DUFFAR, Grenoble Institute of Technology, France, Prof. Sefik SUZER, Bilkent University, Turkey, Prof. Peter RUDOLPH, Crystal Technology Consulting (CTC), Schoenefeld, Germany, Jochen FRIEDRICH, Fraunhofer IISB, Department Crystal Growth, Erlangen, GERMANY, Tatau NISHINAGA, Professor Emeritus, The University of Tokyo, Koichi KAKIMOTO, general secretary of the International Organization for Crystal Growth, Wolfram

MILLER, Leibniz Institute for Crystal Growth (IKZ), Berlin, Jeffrey DERBY, University of Minnesota, Minneapolis, U.S.A, Prof. Stefan ANTOHE, the Dean of the Faculty of Physics, University of Bucharest, Romania, Prof. Lucian PINTILIE, the director of the Institute of Materials Physics, Bucharest-Magurele Romania and Prof. Wilhelm Kappel the director of the National Institute for R&D in Electrical Engineering ICPE-CA, Romania.

More than 200 participants from al over the world will attend the Conference. In the Abstract Book, almost 300 abstracts were included. There are 50 invited, 90 oral and 160 poster contributions. The title of “Honorary Member of the Romanian Materials Science - Crystal Growth Society” and the diploma, will be awarded to some distinguished participants. Full paper contributions presented in the conference shell be published in some international journals (Journal of Crystal Growth, Thin Solids Films, Applied Surface Science, Computational Materials Science, Journal of Optoelectronics and Advanced Materials, Optoelectronics and Advanced Materials - Rapid Communications).

The organizers of ROCAM 2012 Conference are indebted to the leadership of “Transilvania” University of Brasov and particularly to the vice-Rector Daniel MUNTEANU for collaboration and for special logistic facilities.

We are indebted to our colleagues from the University of Bucharest, Faculty of Physics, from the National Institute of Materials Physics (NIMP) Bucharest, from the Transilvania University of Brasov and from the National Institute for R&D in Electrical Engineering ICPE-CA, members of the organizing team of the conference, who voluntarily accomplished difficult technical, logistic and administrative tasks.

Bucharest, August. 2012,

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Plenary Lectures

NOVEL TECHNOLOGIES, NUMERICAL MODELING AND THERMODYNAMIC COMPUTATION FOR THE CRYSTAL GROWTH OF COMPLEX MATERIALS

Roberto Fornari
Leibniz Institute for Crystal Growth and Institute of Physics, Humboldt University, 12489 Berlin (Germany)

The development of cost-effective methods for the production of high-quality single crystals of always larger size and the growth of novel materials with complex thermodynamic properties, such as very high melting temperatures, high dissociation pressures at the melting point, incongruent melting, post-growth phase transition are the most prominent challenges of modern bulk crystal growth.

In order to meet the requirements posed by more advanced device technology, crystal growers are continuously engaged in modifying well established growth systems and technologies, independently of the involved phase transition (melt-solid, solution-solid, vapour-solid). The search for bigger and more perfect “traditional” crystals essentially led to upgraded furnaces with better control over temperature gradients, application of ultrasonic signals or low-frequency vibrations to the melt, use of external magnetic fields, etc., whereas the search for novel crystalline materials triggered a number of advancements, like new models of autoclaves, laser-heated floating-zone systems, high-temperature PVT systems, use of furnaces with special atmosphere, etc.

Numerical simulation of crystal growth processes has become an indispensable tool for the optimization and up scaling of traditional growth equipments. Indeed it would be impossible to develop Czochralski pullers for the new generation of silicon ingots by just an empirical approach, since the cost of materials involved in each pulling experiment is extremely high, which makes a trial-and-error approach unfeasible. The same applies to many other standard materials such as semiconducting compounds, sapphire, scintillator and laser crystals.

When considering novel materials there is an additional complication as often the phase diagram is unknown. For this reason also thermodynamic studies (for example by DTA and TG), computations and analysis in situ are very important for the development of suitable crystal growth processes. This presentation will highlight the recent trends of modern bulk growth and provide few examples of successful combinations of advanced growth technology, computer modelling and thermo-dynamic analysis.

FUNDAMENTAL PROPERTIES OF TM NITRIDES AND DESIGN STRATEGIES FOR GROWTH OF SELF-ORGANIZED NITRIDE NANOSTRUCTURES

Joe Greene
Departments of Materials Science, Physics, and the Materials Research Laboratory, University of Illinois, Urbana, Illinois, USA, Physics Department, Linköping University, Linköping, Sweden, Mat. Sci. Dept, National Taiwan University of Science & Technology, Taipei, Taiwan

Cubic TM nitrides have wide single phase compound fields which can be exploited. We show results for vacancy hardening (not associated with film strain) in 3d group-IV $TiN_x(001)$: the hardness H of epitaxial layers increases dramatically, while the elastic modulus E and the relaxed lattice constant decrease linearly, as x is decreased from 1.0 to 0.67. Over the same x range, the resistivity $\rho(x)$ increases from 13 to 192 $\mu\Omega$-cm due to electron scattering from N vacancies. In contrast, H(x), E(x), and $\rho(x)$ for 5d group-V $TaN_x(001)$ remain constant due primarily to the presence of isoelectronic antisites.

While TiN and TaN are metallic, 3d group-III ScN(001) is a transparent semiconductor with an indirect Γ-X gap of 1.3 eV and a direct X-point gap of 2.4 eV. Reflectivity measurements from $Sc_{1-x}Ti_xN(001)$ layers show TiN is strongly reflecting up to the reflectance edge at $\hbar\omega_e = 2.3$ eV, ScN is transparent, and $\omega_e \propto x^{0.5}$. Thus, hard decorative coatings with a wide palette of colors can be obtained.

The extreme range of materials properties available in TM nitrides and related systems can be enhanced through the formation of self-organized superhard nanostructures consisting of commensurate nanolamellae, nanocolumns, nanospheres, and nanopipes. Self-organization strategies include controlled phase separation, surface-induced spinodal decomposition, surface segregation-induced renucleation, strain-induced roughening, surface anisotropy, and dynamic resputter yield amplification.

As a final example, the Ti-B-N system, exhibiting a deep miscibility gap in the ternary phase diagram, is used as an archetype to probe reaction paths leading to the directed self-organized growth of "intelligent" high-temperature nanostructures (e.g., the hardness actually increases with temperature!).

VAPOR PHASE GROWTH OF COMPLEX SEMICONDUCTOR ALLOYS

Thomas F. Kuech[1*], Luke J. Mawst[2], Dane Morgan[3] and Susan E. Babcock[3]
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The metalorganic vapor phase epitaxy of semiconductor materials has been developed over the past three decades and has focused on the formation of semiconductors and alloys useful in the formation of optoelectronic and high-speed electronic devices. These materials are generally binary semiconductors or alloys that have broad miscibility. The extension to alloy systems that are immiscible in bulk form requires modification to conventional growth systems preventing the nucleation and growth of multiple compositional phases. The formation of these multi-component alloys is discussed in terms of their known thermodynamic behavior which can be altered by the presence of strain in the material and the growth temperature. Additionally, the stoichiometry and reaction kinetics of the alloy constituents can impose kinetic constraints allowing for the formation of alloys within the miscibility gap. This talk will review and discuss those influences in determining the film composition and microstructure of the model alloys GaAsSb and GaAsN, which have both thermodynamic and chemical kinetic influences on the formation of the alloy. Additionally, there are new materials such as GaAsBi and Sb-N alloys that extend the range of device opportunities provided they can be synthesized in stable and homogeneous forms. The extension of this work to more complex alloys, such as 5 element or quinternaries, which open the palette of device materials will be discussed along with some recent applications of these materials to optical devices.

FUTURE OF Si WAFER TECHNOLOGY

Martin Green
ARC Photovoltaics Centre of Excellence, School of Photovoltaic and Renewable Energy Engineering,
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The vast majority of photovoltaic solar cells that have been produced to date have been based on silicon wafers, with this dominance likely to continue well into the future. The surge in manufacturing volume over the last decade has resulted in greatly decreased costs. Multiple companies are now manufacturing at costs below the US$1/Watt module manufacturing cost benchmark that was once regarded as the lowest possible with this technology. Despite these huge cost reductions, there is obvious scope for much more of the same, particularly as the market for the polysilicon source material becomes more competitive, the new "quasi-mono" directional solidification processes are brought on-line, wafer slicing switches to much quicker diamond impregnated approaches and cell conversion efficiencies increase towards the 25% level. Evolutionary paths to lower cost beyond this point are also explored.

S1

Crystal Growth Fundamentals and Applications

Invited Papers

CHALENGES WITH CRYSTAL GROWTH AND CHARACTERIZATION OF FRUSTRATED AND TITANIUM PYROCHLORES

H.A. Dabkowska, A. Dabkowski, C. Marjerrison
Brockhouse Institute of Materials Research, 1280 Main St. West, Hamilton, ON, Canada, L8S 4M1

Rare earth pyrochlores are fascinating oxides with interesting physical properties. They usually disclose frustrated magnetic properties (spin ice, spin liquid etc) and are the favourite playground for solid state physicists. With few exceptions RE pyrochlores melt congruently, at temperatures often well above 2000°C. To reliably assess their physical, crystallographic and optical properties high quality single crystals are essential. The Optical Floating Zone [OFZ] technique and related Travelling Solvent Floating Zone [TSFZ] technique are among the newest, most sophisticated crucible-free methods for the growth of various single crystals of both titanates and vanadates.

This presentation discusses the application of the FZ and the TSFZ methods in growth of high quality single crystals of RE2Ti2O7 and RE2V2O7. The materials grown require relatively slow (slower than 5 mm/h) or very fast (faster than 25 mm/h) growth speed each presenting its own challenges. Oxygen pressure was controlled by using H2/Ar, CO/CO2 or O2/Ar mixtures. The relevant processes and reactions are explained on the basis of known phase diagrams.

In the 21st century the research on frustration entered a new era. Frustration is now recognised as the fundamental concept important not only for oxides but also for metals, multiferroics and dielectrics. Thanks to this fact, the well characterised pure and doped single crystals are in constant demand.

Keywords: crystal growth, optical floating zone, pyrochlores, frustrated magnets

INSTABILITIES DURING MENISCUS-DEFINED CRYSTAL GROWTH: A TALE OF SEVERAL SYSTEMS

Jeffrey J. Derby, Parthiv Daggolu, and Andrew Yeckel
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Meniscus-defined melt crystal growth processes offer the possibility of growth of a crystal directly from a liquid surface, thus avoiding deleterious wall contact. Several processes have been developed, including detached Bridgman growth (DVB), edge-defined film fed growth (EFG), horizontal ribbon growth (HRG), and micro-pulling-down crystal growth (MPD). From a theoretical point of view, these are low to intermediate Bond number systems in which there often exists several meniscus shapes that can satisfy the local force balance. Consequently, these systems exhibit a variety of dynamical bifurcations, signaling existence of multiple steady solutions as well as time-periodic behavior. Bifurcations of the saddle node, pitchfork, transcritical, and Hopf types can occur depending on the growth configuration, particularly the manner in which the meniscus is confined at its ends. In addition, thermal effects often play an important role, either instigating or modulating the bifurcation. Commonly, solutions are found to exist only within a narrow range of operating parameters, posing problems to both physical operation and computer simulation of these systems.

We investigate these systems using thermal-capillary models which rigorously conserve mass, energy, and momentum within the meniscus and solidification regions. Steady state model equations are solved on deforming grids by the Galerkin finite element method. Locations of the growth front, triple-phase line, and meniscuses are all computed to satisfy appropriate physics at these interfaces. Solution stability is assessed by nonlinear transient analysis.

We present cases of instabilities for several crystal growth systems with technological relevance, including the detached Bridgman growth of cadmium zinc telluride for radiation detectors, the edge-defined film fed growth of scintillator crystals, the micro-pulling-down growth of sapphire fibers, and the horizontal ribbon growth of photovoltaic silicon. Specific failure mechanisms are discussed, as well as generic behavior common to all meniscus-defined growth systems.

The nature of the end condition imposed on the meniscus, namely whether it meets a smooth surface at the wetting angle, or is pinned at a sharp corner, has a profound influence on system behavior. Saddle node limit points are commonly observed in pinned systems such as MPD and HRG. Symmetry-breaking pitchfork bifurcations are observed in systems of a planar geometry having two pinned menisci, such as planar EFG. Perhaps most unusual, however, is the appearance of a transcritical bifurcation in the detached vertical Bridgman system which marks a loss of solution stability above a critical wetting angle of melt on crucible. This bifurcation presents a limit on stability but none on existence, making it possible to employ an active control strategy based on controlling the pressure below the meniscus. In particular, this feature is discussed with respect to making the growth of cadmium zinc telluride (CZT), an important material for radiation detection, practical in a detached Bridgman system.

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VIS AND UV NONLINEAR CRYSTALS FOR BIREFRINGENT PHASE MATCHING

Pascal Loiseau[1], Simon Ilas[1], Gérard Aka[1], Takunori Taira[2]
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The use of non linear optical crystals for the design of compact all solid state lasers emitting in the visible down to the UV range is now widely practised. According to the set-up, the target wavelength and power, the non linear optical (NLO) crystal is the key-point of many laser systems since the fundamental laser to be used is mastered.

The nonlinear optical properties of rare earth calcium oxyborates were first reported in the literature in 1996 and 1997 for $GdCa_4O(BO_3)_3$ (GdCOB) and $YCa_4O(BO_3)_3$ (YCOB) respectively. Their practical properties, especially their congruent melting and their non hygroscopic character, were immediately stressed as great advantages. As a result, oxyborate crystals are now envisaged for more and more practical applications.

On the basis of the data which have been compiled in the literature, a point was made about the optical properties which can be calculated for YCOB and GdCOB. As main competitors of LBO (LiB_3O_5), the SHG conversion efficiencies of RECOB crystals were compared to LBO by using a high-brightness passively Q-switched Nd:YAG microchip laser.

To extend towards UV the NLO applications of oxyborates, deeper structural modification is considered by substituting RE^{3+} and O^{2-} by Ca^{2+} and F^- respectively: this leads to the calcium fluoroborate $Ca_5(BO_3)F$ (CBF), presenting a transparency UV cutoff at 190 nm. Although CBF shows a non congruent melting, flux growth method allows obtaining CBF crystals, chemically stable and not hygroscopic. The SHG efficiency of CBF is similar to that of GdCOB, about twice as high as KDP one, and CBF has enhanced potential applications as nonlinear optical material in the UV range.

Furthermore, other NLO crystals can also be proposed for UV applications, especially those belonging to the huntite group like YAB ($YAl_3(BO_3)_4$). If they have potentially great properties for UV frequency conversion, their crystal growth needs nevertheless to be improved.

CHALLENGE OF BLUE-GREEN LASER FROM ND$^4F_{3/2}$ →$^4I_{9/2}$ LASER TRANSITION OR YTTERBIUM DOPED CRYSTALLINE HOSTS: REVIEW OF CRYSTAL GROWTH AND LASER PERFORMANCES

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Diode-pumped solid-state lasers operating in the blue-green spectral range have a large number of applications ranging from high-density optical data storage to photo therapy and medical diagnostic. Such a device can be designed using a neodymium-doped laser material, operating on the $^4F_{3/2} \rightarrow ^4I_{9/2}$ laser transition and frequency doubling in a quadratic non-linear optical crystal. Such laser materials should obey to the following conditions: high energy for the emitting level $^4F_{3/2}$, moderate crystal field splitting of the ground manifold $^4I_{9/2}$ and high branching ratio for the $^4F_{3/2} \rightarrow ^4I_{9/2}$ emission. Nevertheless, the energy of the terminal laser level must not be too low, to avoid strong reabsorption of laser emission owing to the thermal population of this level. The above conditions are found in compounds with low covalency (such as oxides or fluorides) and with high anionic coordination for the Nd^{3+} site. Restriction to only congruent melting composition allows crystal growth of large boules by the Czochralski pulling technique. Several series of oxides fulfilling the above requirements were investigated: $ABGa_3O_7$ (A=Ca, Sr and B = Gd or La), $ABAlO_4$ (A=Ca, Sr, Ba and B= Y, La or Gd) and AMO_4 (A=Ca, Sr, Ba and M=Mo,W). The Nd-doped strontium and lanthanum aluminate (Nd:ASL), with formula $(Sr_{1-x}Nd_yLa_{x-y})(Mg_xAl_{12-}x)O_{19}$, particularly fulfils the above requirements. We will make a review of the recent works regarding these Nd or Yb doped crystalline hosts for blue-green laser generation. The potential laser wavelengths of all these compounds will be discussed.

Another possibility is the use of neodynium or ytterbium doped non linear laser host in order to generate green laser emission by self-frequency doubling process. We will discuss the self-frequency doubling performances of Yb or Nd doped $ReCa_4O(BO_3)_3$ (Re = rare earth, YCOB and GdCOB) regarding the state of art.

ADVANCED NLO CRYSTALS FOR UV LIGHT GENERATION

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Up to now, many efforts have been carried out in order to develop new NLO materials and/or to improve their specific properties or workability. The experimental results demonstrated that borate-type compounds constitute a veritable source of NLO crystals with good nonlinear properties. Nowadays, two families of borate crystals are known that melt congruently or can include congruently melting compounds which can be grown with high quality and large size by Czochralski method: rare-earth calcium oxyborates RECOB ($\mathrm{RECa_4O(BO_3)_3}$, RE = Gd, Y, La) and binary borates $\mathrm{LnMe_3(BO_3)_4}$ (Ln = lanthanide, Me = Al, Ga, Sc) with trigonal huntite-type structure. Two types of new NLO borate crystals with congruent melting behaviour belonging to these two families are presented in this work:

(i) $\mathrm{Y_{1-x}R_xCa_4O(BO_3)_3}$ ($\mathrm{R^{3+} = Sc^{3+}, Lu^{3+}}$) crystals where in function of the substitution degree with $\mathrm{R^{3+}}$ ions it is possible to achieve second harmonic generation (SHG) in non-critical phase matching (NCPM) conditions of some important laser emissions at specific wavelengths in the near-IR range, conditions which ensure obtaining of maximum conversion efficiency into near-UV spectral range;

(ii) $\mathrm{La_xGd_ySc_z(BO_3)_4}$ ($x + y + z = 4$) crystals with wide transparency range from the UV to the IR domain and excellent NLO properties characteristics to huntite-type borate crystals.

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TECHNIQUES FOR IMPROVE ELECTRON DIFFRACTION DATA

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Electron diffraction studies of materials are less used for accurate measurements in lattice parameters determination. Contributions of unwanted effects, such dynamical effects, influence of beam-sample geometry and presence of amorphous sample/substrate, are large compared with X-Ray diffraction and results are compromised. For these reasons, we expect larger errors for electron diffraction results compared with X-Ray diffraction. Another infrigment are magnitude of diffraction angle, very small in case of electron diffraction with maximum magnitude about 3-5 degree.

Data mining in electron diffraction study can be improved by means of software techniques. The Hough or Cos transform are example of such techniques. We use a Radon transform to evaluate more precisell peak position, and check misalignement of diffraction pattern. The results are indexed and used to evaluate lattice parameters from Cohen method with modified Nelson-Riley function for small angles that includes approximation of $\cos\theta = 1$.

We use this technique to evaluate data from cubic(Au) and hexagonal(ZnO) structures. Diffraction patterns was acquired on CM120ST microscope at 100KeV. Gold samples are nanocrystalline powder on amorphous carbon film obtained by evaporation, and used for calibration of electron diffraction patterns. ZnO samples are synthesized by three methods: doctor blade, spin coating and electrochemical deposition.

Keywords: electron diffraction, Radon transform, Cohen, Nelson-Riley

GROWTH INDUCED IMPURITY NON-UNIFORMITY ASSOCIATED WITH GROWTH MORPHOLOGY

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An example of impurity non-uniformity associated with morphology has been known as the different impurity incorporation in facet and off-facet regions of CZ Si. In this talk, impurity or composition non-uniformity associated with largely bunched steps (macrostep) is described in detail taking solution growth of compound semiconductor as a model. At first, the shape and the behavior of macrostep during the solution growth are explained. Then, the theory for giving the surface supersaturation around macrostep in solution growth is developed. It is shown that the calculated surface supersaturation distribution between macrosteps agrees very well with those obtained from the shape of atomistic steps.

Impurity concentration and composition of alloy around macrostep is measured by spatially resolved photoluminescence and the non-uniformity is explained in terms of the facet/off-facet effect.

GROWTH METHODS FOR BULK ZINC OXIDE

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Even if zinc oxide is used worldwide in scales of ca. 10^6 tons annually e.g. for the production of rubber, in medicine, and for animal feed, single crystals play only a minor role so far. ZnO based electronic devices such as varistors are produced from polycrystalline material. Nevertheless, single crystals are required for basic studies: It should be underlined that ZnO is a transparent semiconductor (TSO) with wide bandgap of $\approx$3.4 eV, comparable to GaN.

For zinc oxide a wide choice of completely different bulk crystal growth techniques is reported in the literature:

1. from solutions (hydrothermal alkaline [= most relevant], normal pressure alkaline aqueous, molten salts).
2. from the gas phase (physical vapour transport, chemical vapour transport)
3. from melts (cold crucible = skull melting, Bridgman)

The benefits and drawbacks of these techniques will be discussed, with special emphasis on the Bridgman method. Here a crucible material that is chemically and thermally stable against the melt is required. This means especially that the oxygen partial pressure must be within a useful corridor for a wide temperature range. Reactive growth atmospheres with the active component carbon dioxide can guarantee such stable growth conditions.

The physico-chemical properties of ZnO are similar to that of some other TSO's, such as β-Ga_2O_3, In_2O_3, SnO_2: All of them combine a high melting point where platinum devices cannot be used (> 1600°C) with high volatility and high dissociation to metal and oxygen at the melting point. It will be shown why the concept of reactive atmosphere enables one to stabilize these oxides under growth conditions, and to grow bulk crystals.

ALN SINGLE CRYSTALS AS SUBSTRATES FOR DEEP-UV OPTOELECTRONICS

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In recent years, single-crystalline aluminum nitride (AlN) became a candidate as substrate material for AlGaN epilayers with high Al content. AlN substrates have recently been successfully employed for solid-state deep-UV optoelectronic emission, lasing, and sensor devices. The preferred method to grow AlN bulk single crystals is the sublimation–recondensation method at temperatures well above 2000°C. As AlN does not occur in nature, three strategies for obtaining seeds for bulk crystal growth have been investigated: Grain selection, spontaneous nucleation, and growth on SiC as a foreign substrate. While spontaneous nucleation yields crystals of highest structural perfection but limited size, seeding on SiC seemed especially promising to quickly reach AlN crystals of industrial relevant size and diameter. However, lattice and thermal expansion mismatch as well as defects present in the SiC seed or generated at the SiC/AlN interface lead to formation of grain boundaries and tilted domains, which persists when using such crystals as seeds for subsequent homoepitaxial growth. While as AlN is still commercially available only in small quantities and sizes, both routes discussed above are actively researched.

In this presentation, the two routes to AlN bulk growth will be contrasted and evaluated in respect to their potentials and consequences for AlN substrate preparation and application. Furthermore, we will show in detail how the choice of growth strategy not only influences the available size and quality of the AlN crystals, but also their optical properties. Due to materials compatibility issues, the two routes are followed using different crucible materials and growth parameters. The resulting differences in impurity and intrinsic defect concentrations have a decisive impact on deep-UV absorption in bulk AlN. Thus, controlling contamination as well as faceting and orientation-dependent segregation effects will be a key prerequisite for further commercialisation of AlN substrates.

REVISITING BPS, AND A NEW MODEL FOR THE EFFECTIVE SEGREGATION COEFFICIENT

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The Burton, Prim and Slichter's (BPS) model for the effective segregation coefficient k_{eff} has been used by crystal growers for a half of a century. The accuracy of the BPS model is hard to establish, because k_{eff} is sensitive to the solute diffusion coefficient, for which consistent data are not available. The model is based on the Levich's equation for solute layer thickness, which was derived assuming that: the rotating disk is impermeable, the flow is laminar, natural convection is negligible, and the Schmidt number is infinite.

A new model for k_{eff} will be presented. It accounts for both natural and forced convection, laminar or turbulent flow, finite Schmidt numbers, and permeable interface. Furthermore, simpler expressions are developed for restricted range of conditions, e.g., Czochralski growth under forced laminar convection (no natural convection or turbulence)

Oral Presentations

CHARACTERIZATION OF KDP SINGLE CRYSTALS WITH EMBEDDED ALUMINIUM OXYHYDROXIDE NANOPARTICLES

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Investigations aimed at the creation of composite materials based on dielectric matrices with incorporated organic impurities and nanoparticles are being actively carried out nowadays. Composite systems of “dielectric matrix/nanoparticles” type are promising for the development of nonlinear optical elements meant for laser radiation control. Our preliminary investigations showed that the energy resonances due to the nanostructured internal interface may considerably enhance the material's nonlinear susceptibility, increasing the conversion efficiency.

The given work presents the experimental results concerning structure and physical properties of a new composite functional material based on nanostructured aluminium oxyhydroxide (NAO) embedded into KDP matrix (KDP:NAO). For obtaining of the mentioned composite system the temperature lowering method of the growth solution was used. The characterization of as-grown crystals was performed using UV-vis-NIR spectroscopy, transmission and scanning electron microscopy, optical microscopy, etching method, the spatial beam profile distortion analysis in the far field and the laser damage threshold measuring method.

The quasiperiodical zonal structure was revealed in KDP:NAO crystals caused by the capture of nanoparticles into the boundaries between the layer stacks. The mechanism of nanoparticles incorporation into the crystal matrix is found. Under the action of picosecond laser pulses the photoinduced changes of nonlinear optical refractive response was observed which is probably due to the excitation of defect states on the nanoparticles surface and their interaction with the hydrogen subsystem of KDP matrix. These defect states also cause the appearance of the absorption band at 270 nm which intensity depends on NAO concentration (C). At C<1 ppm KDP:NAO crystals show high structure perfection and high resistance to the action of impulse laser pulses.

THE FEATURES OF MORPHOLOGY AND CRYSTALLINE STRUCTURE OF THE MONODISPERSE Lu_2O_3:Eu^{3+} SUBMICROSPHERES

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The preparation of the new luminescent crystalline nanomaterials is perspective research area. The design conception of such particular low-dimensional materials directs on providing required chemical and phase composition, perfect low-defects crystalline structure, optimal morphology and size distribution, avoidance of the uncontrolled agglomeration. For this purpose, ideal sphericity, well-defined diameters with a narrow size distribution of the structural units are strongly desired. Re_2O_3:Eu^{3+} (Re – Lu, Y) is one of the most perspective materials for X-ray detection and imaging as well as laser materials creation.

Uniform-sized spherical crystalline Lu_2O_3:Eu^{3+} phosphors with the diameters ranging from 40 to 500 nm have been obtained by the simple and economic urea-based homogeneous precipitation technique. The evolution of the structure and morphology of the crystalline Lu_2O_3:Eu^{3+} spheres during annealing at temperatures ranged from 600 to 1200ºC was studied. It was established that the crystalline spheres had high-developed surface area due to the porosity with the size of the voids was about 10-15 nm. The rise of the crystallite Scherrer sizes with the increase of the annealing temperature was observed. The spherical shape of the particles was destroyed at the temperature above 1000ºC because of the grain recovery with subsequent sintering of the particles. It was shown that X-ray luminescence intensity of spherical particles rose with the increase of annealing temperature which was concerned with the improvement of the crystallinity. The crystallites Scherrer sizes growth was observed with the incensement of particles diameter which defined the improvement of the luminescence efficiency for the Lu_2O_3:Eu^{3+} with the major diameter. The results obtained allow one to consider the uniform-sized Lu_2O_3:Eu^{3+} submicrospheres as perspective material for display technologies.

CZOCHRALSKI GROWTH OF INDIUM IODIDE

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The Czochralski pulling process is the most valuable and cost efficient method for producing large oriented single crystals of the group IV and III-V semiconductors. However, there have been only a small number of reported attempts to use the Czochralski process for growing the wide bandgap compound semiconductors, needed for the room temperature operated detectors.

The main difficulty is in the low chemical stability and high vapor pressure of the group II, V, VI and VII elements, leading to the off-stoichiometric composition, and various related defects. Among the heavy metal halides, indium iodide and indium bromide present an exception. InI has a high disassociation energy and low relatively low vapor pressure, allowing for Czochralski pulling. We have assembled an easy to clean Czochralski (CZ) setup, which provides a good visibility of the process. Yet, because of the low temperature gradients, CZ pulling difficult.

We will describe the procedures used for synthesis, zone refining, Czochralski growth and characterization of indium iodide. A numerical model of the CZ process, based on the code CrysMass was developed and used to study convection during InI pulling.

PROPERTIES OF BGO CRYSTALS GROWN BY THE EFG METHOD

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To reduce the cost of production of scintillation elements shaped crystals bismuth germanate were grown by EFG method. Growth of shaped crystals for the production of elements in the form of plates, pixels and rods in diameter to 20 mm reduces the losses of material on 50% in compare with production from the bules of large diameter. Shaped crystals of bismuth geramanate have energy resolution of 9.5% (irradiation of ^{137}Cs 662 keV), the light output is equal to the crystals grown by Czochralski method. Optical absorption of shaped crystals at the maximum of the luminescence 480 nm is the same for crystals by Czochralski technique. Also found a significant reduction of the photochromic effect in shaped crystals with respect to the Czochralski crystal, which indicates a more efficient displacement of the impurities during crystal growth.

ON GROWTH RATE HYSTERESIS AND CATASTROPHIC CRYSTAL GROWTH

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Different crystal growth rates as supersaturation is increasing or decreasing in impure media is a phenomenon called growth rate hysteresis (GRH) that has been observed in diverse systems and applications, such as snow crystals formation or during biomineralization. We have recently shown that the transient adsorption of impurities onto newly formed active sites for growth (or kinks) is sensitive to the direction and rate of supersaturation variation, thus providing a possible explanation for GRH (Ferreira et al., 2012). In the present contribution, we expand on this concept by deriving the analytical expressions for transient crystal growth based on the energetics of growth hillocks formation and kink occupation by impurities. Two types of GRH results are described according to the variation of kink density with supersaturation: for nearly constant density, decreasing or increasing supersaturations induce, respectively, growth promoting or inhibiting effects relatively to equilibrium conditions. This is the type of GRH measured by us during the crystallization of egg-white lysozyme. For variable kink density, slight changes on the supersaturation level may induce abrupt variations on the crystal growth rate. Several literature examples of this so-called 'catastrophic' crystal growth are discussed in terms of the fundamental consequences involved in the process.

GROWTH AND CHARACTERIZATION BY STM OF $BI_2SR_{0.8}CA_{1.8}CO_2O_Y$ SINGLE CRYSTAL WHISKERS

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For energy conversion, of much interest are layered Co-based thermoelectric oxides such as Bi-Sr-Ca-Co-O phases. According to literature, single crystals of these materials have a power factor (= S/ρ, with S = Seebeck coefficient and ρ = resistivity) 5-7 times higher at 700°C than for bulks. Few articles present growth and characterization of single crystals.

We present growth of single crystal whiskers of the phase $Bi_2Sr_2Co_2O_y$ (Bi222Co). We used the so-called glass route method. The starting composition of the mixture was $Bi_3Sr_2Ca_2Co_{1.2}O_x$ and the substrates were obtained by rapid cooling of the melt from a temperature of 1310°C down to room temperature between metal plates of Fe. As revealed by XRD, substrates were crystallized. Subsequently, substrates were annealed in air. We applied a heating regime with a maximum temperature of 890°C followed by a slow cooling to 870°C with a cooling rate of 0.01°/min. From the substrates, single crystal belt-like whiskers with EDS composition of $Bi_2Sr_{0.8}Ca_{1.8}Co_2O_y$, straight and curved, were obtained. Whiskers of other phases were not observed as usually found in the literature. Surface of the whiskers was clean and with low roughness (<1 nm). This allowed measurements of STM/STS spectra. Whiskers show a semiconducting gap (80-160 K) which is decreasing with temperature increase.

Poster Presentations

NANOSTRUCTURES FORMED IN PURE QUARTZ GLASS AT REACTOR IRRADIATION

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It is known that exposure of SiO_2 glass to fast neutron fluencies > 10^{19} cm^{-2} results in formation of a metamict phase with a higher density and decrease of the mesoscopic order scale < 2 nm. The irradiated glass structure model assumes clusters ordered like cristobalite and tridymite and separated by amorphous layers. While at much less fluencies radiolysis causes atomic rearrangements (Si-Si and O-O defects) driven by non-radiative electron+hole recombination. Our research was aimed at studying the defect pair accumulation and nano-particle growth in pure (< 10^{-4} %) SiO_2 glass under irradiation with fast neutron fluencies $6{\cdot}10^{16} - 5{\cdot}10^{19}\,cm^{-2}$ and γ-quanta up to ~$1.8{\cdot}10^{20}$ cm^{-2} in the reactor core in water filled cell. Using the improved X-ray diffraction techniques, it was shown that the neutron irradiation results in destroying of the initial α- β-quartz mesostructure of 1.7 and 1.2 nm sizes and growing of tridymite and cristobalite nanocrystals to 8 and 16 nm sizes, which are covered with amorphous shell of 0.65–0.85 nm thickness. The total volume of the thermal peaks of atom displacements, where the nanocrystals grow to fluency $5{\cdot}10^{19}\,cm^{-2}$, reaches 4 %. Optical absorption spectra revealed point defects (E'_s , E'_1 , E'_2 and non-bridging oxygen O-O). Splitting of E'_1-center level by 0.13 eV appears at fluency $3{\cdot}10^{17}$ cm^{-2} and that of Si-Si is 0.19 eV at 10^{19} cm^{-2} due to strong exchange interaction between close centers when they are accumulated in the defective shell around the nanocrystals. The E'_1-center (5.75 eV) concentration determined by Smakula formulae ~$0.55{\cdot}10^{17}cm^{-3}$ poorly changes up to $5{\cdot}10^{19}\,cm^{-2}$, while the saturated O-O-center concentration is ~$1.2{\cdot}10^{17}$ cm^{-3}. The intensive (D~2-4) resonances are characteristic for interband electron transitions, with the width of 10-15 nm being close to the nanocrystals' sizes and the level energy depending on their structure. Fast recombination luminescence, excited either with laser 3.67 eV or γ-irradiation, is due to electron transitions in O-O centers: $^1S_0 \rightarrow {}^1D_2$ (2.23 eV) and $^1D_2 \rightarrow {}^3P_2$ (1.96 eV). The work was carried out on the contract FA-F2-F120 from Uzbekistan Committee on Coordination and Development of Science and Technology.

CALCIUM CARBONATE MICROPARTICLES GROWTH TEMPLATED BY AN OXADIAZOLE-FUNCTIONALIZED MALEIC ANHYDRIDE-N-VINYL-PYRROLIDONE COPOLYMER, WITH ENHANCED PH STABILITY AND VARIABLE LOADING CAPABILITIES

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The mineralization characteristics of $CaCO_3$ microparticles from saturate aqueous solutions in the presence of a conjugate drug-copolymer has been investigated, comparative with particles prepared in similar conditions but without polymer. The polymer conjugate, P(NVP-MA-Ox), is based on poly(N-vinylpyrrolidone-co-maleic anhydride) as support and 2-amino-5-(4-methoxy-phenyl)-1,3,4-oxadiazole, having antimicrobial and antifungal activity. The influence of polymer/$CaCO_3$ ratio on the microparticles characteristics and the particles pH stability was deeply investigated by scanning electron microscopy, X-ray diffraction, flow particle image analysis, particles charge density, and electrophoresis. The presence of P(NVP-MA-Ox) as template in $CaCO_3$ mineralization process induced the particles stability increase up to the polymer isoelectric point located at pH = 3.4, irrespective of carbonate content in composite particles. The adsorption capacity of the microparticles as a function of their characteristics was tested using methylene blue. The sorption capacity of composite materials increased with the increase of polymer content in the composites, suggesting that the sorption process takes place mainly by electrostatic interactions.

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GROWTH OF PARATELLURITE CRYSTALS BY THE LOW TEMPERATURE GRADIENT CZOCHRALSKI METHOD WITH A NONUNIFORM HEATING

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Single crystals of partellurite (TeO_2) are widely used in acousto-optics in the range of 0.5-5 μm. Czochralski growth of TeO_2 crystals under the low gradient thermal conditions (1-2ºC/ cm) allows to obtain faceted crystals with small residual thermoelastic stress and high optical homogeneity. However, such crystals often contain characteristic inclusions of bubles and so-called “hair-like” light scattering. Both these defects are not corresponded with variation of impurity concentration in the crystals.

It is known that under low gradient conditions a considerable part of the melt exists in presolidified (metastable) state. In that case crystal growth is strongly affected by association processes of building units in the melt, which in turn may be the source of described defects in TeO_2 crystals. Embedding in the crystal structure of such "bulk" modules favors the capture of bubbles and can lead to low-angle mismatches at the nanoscale, which are likely to provide local variations in refractive index and, consequently, the appearance of “hair-like” light scattering in paratellurite crystals.

We have constructed a setup for growth of paratellurite crystals that has a nonuniform heating of the crucible wall. While temperature gradients near the crystal interface are maintained at low values, the melt region of some sector in the bottom part of the crucible is superheated. We assume that in this case the associates in the melt are destructed when they convectively flowing through the superheated domain, so the crystal is grown by means of smaller "building" particles without the low-angle mismatch.

The setup is equipped with the weight sensor, distributed control system, and four-zone furnace with 1+3+3+1 heaters in each zone. Distribution and nonuniformity of temperature in the melt are organized by controlling the delays between switching of heaters. Helping this setup paratellurite crystals weighing about 1.2 kg completely free of inclusions, bubbles, with a low level of the light scattering were grown from the 100 mm in diameter crucible.

NEW CACO$_3$ / POLY(2-ACRYLAMIDO-2-METHYLPROPANESULFONIC ACID–CO-ACRYLIC ACID) POLYMORPHS, AS TEMPLATES FOR CORE/SHELL PARTICLES

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The special mineralization process in the biological system has attracted many scientists to biomimetically synthesize inorganic/organic materials. An important method of biomimetic synthesis is to use a soft organic template and to control the morphogenesis of inorganic materials with complex forms. Therefore, the crystallization of $CaCO_3$ particles from aqueous solutions in the absence and presence a strong/weak anionic copolymer, poly(2-acrylamido-2-methylpropanesulfonic acid–co-acrylic acid) (PAMPSAA) is followed in this study. The influence of polymer chemical structure and concentration on the nucleation and growth of calcium carbonate has been deeply investigated, showing a strong effect on the morphology of the formed composite materials. Vaterite/calcite polymorphs with different sizes and spherical shapes were obtained. The new materials were characterized by infrared spectroscopy, X-ray powder diffraction, X-ray photoelectron spectroscopy, flow particle image analysis, laser diffraction technology, and SEM and optical microscopy. The encapsulation of porous $CaCO_3$ microparticles can be realized by nanoengineered layer-by-layer self-assembly, which is a self-assembly technique where multiple nano-sized layers are built up on the template using oppositely charged polyelectrolytes. Thus, oppositely charged polyions were assembled onto new prepared carbonate colloidal particles in a layer-by-layer manner to yield core-shell particles.

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SECOND HARMONIC GENERATION IN NEW NONLINEAR OPTICAL CRYSTALS OF $Y_{1-x}R_xCa_4O(BO_3)_3$ (R = Lu, Sc) THROUGH NON-CRITICAL PHASE MATCHING

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Frequency conversion of solid-state lasers operating in the near infrared range by nonlinear optical (NLO) crystals has become the most available method to obtain shorter wavelength lasers with high beam stability, low cost and compactness. $YCa_4O(BO_3)_3$ (YCOB) crystal has attracted great attention as a new NLO material for frequency generation. Our previous researches showed that in YCOB crystal, the Y^{3+} ions can be partially substituted by smaller radius ions Sc^{3+} or Lu^{3+} (r_{Lu} = 0.861 Å, r_{Sc} = 0.745 Å, r_Y = 0.9 Å) in order to tune the chemical composition of the crystal. By changing the compositional parameter x of $Y_{1-x}R_xCa_4O(BO_3)_3$ (R = Lu, Sc) crystals, their optical birefringence can be controlled in order to perform non-critical phase matching (NCPM) second harmonic generation (SHG) of specific near infrared laser emission wavelengths shorter than phase matching cutoff wavelengths of YCOB crystal (724 nm along Y axis and 832 nm along Z axis at room temperature). Five new nonlinear crystals of $Y_{1-x}Lu_xCa_4O(BO_3)_3$ and $Y_{1-x}Sc_xCa_4O(BO_3)_3$ with x = 0.19, 0.29, 0.39 and x = 0.07, 0.11, respectively, of good quality with no cracks and bubbles, have been grown by Czochralski method and their NCPM properties were investigated. We have demonstrated that efficient room temperature type-I NCMP SHG of any wavelength from 692.6 - 724 nm and 791.4 - 832 nm spectral ranges, can be achieved in $Y_{1-x}R_xCa_4O(BO_3)_3$ crystals by tuning the composition.

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STRUCTURAL ANALYSIS OF LONGITUDINAL Si-C-N PRECIPITATES IN MULTI-CRYSTALLINE SILICON

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During crystallization of multi-crystalline silicon, carbon-rich liquid-solid phase-boundary layers appear, resulting in precipitation of n-type conductive Si-C-N filaments. We present an in-depth structural analysis of distinct types of filaments to support modeling of their growth. Phase-contrast micro-tomography down to the sub-micron level is used to study morphology and seeding of precipitates while still embedded in fully functional solar cell samples. A detailed TEM analysis and quantitative EELS analysis is presented, based on tomography-assisted target preparation.

Index Terms — photovoltaic cells, silicon, precipitation.

HgI_2 NANOPARTICLES OBTAINED BY ELECTRON BEAM IRRADIATION

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Mercuric iodide is a vast studied semiconductor because of its properties for ionizing radiation detection. For imaging applications, a uniform and oriented film must be grown onto amorphous substrates. This growth is mainly guided by the first nucleation. In order to obtain adequate HgI_2 nuclei for nucleation, nanotechnology was employed.

HgI_2 particles were synthesized by the hydrothermal method from $Hg(NO_3)_2H_2O$ and NaI or KI as iodine sources. The dependence of the obtained product with iodine source and time of synthesis was studied. A synthesis time varying from 0 to 24 hours was studied, always at a furnace temperature of 120 ºC. The products obtained were then washed with distilled water and left to dry. The initial obtained clusters were characterized by X-ray diffraction (XRD) confirming alpha-HgI_2 phase identity by the Rietveld method. Clusters were also characterized by scanning electron microscopy (SEM) observing that no size uniformity was reached. In order to modify size and morphology of these clusters, they were subjected to the electron beam of a transmission electron microscope. When HgI_2 nanoparticles synthesized from NaI clusters were irradiated with a 100kV electron beam, we obtained plate nanoparticles 20-200 nm in size, and when clusters synthesized from KI were irradiated with the same electron beam we obtained nanoparticles 20-70 nm in size. For all samples, coalescence was observed after nanoparticle formation, giving nanoparticles of larger size. Crystallinity of mercuric iodide nanoparticles formed after irradiation was not yet confirmed. The synthesis method followed by electron beam irradiation promises accurate control of size and morphology of heavy metal iodide nanostructures, which is very propitious for improving HgI_2 film growth orientation and, thought it, the current applications of this material, as far as for the opening of new ones.

EFFECT OF CRYSTAL PULLING RATE FLUCTUATION ON THERMO-ELASTIC STRESSES DURING CZOCHRALSKI SILICON CRYSTAL GROWTH

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Fluctuations in diameter control system, and melt flow perturbs the stability of the Czochralski silicon crystal growth process. In order to keep the desired crystal diameter fixed, two tuning techniques are available: 1. Fine tuning by varying the pulling speed. 2. Rough tuning by changing the heater power. Considering the former method, for a certain average crystal growth rate, the instantaneous pulling rate can fluctuate around its nominal value. Hence, the melt/crystal interface shape deflects in order to balance the latent heat which is generated by new pulling rate. This can affect the thermal field and thermo-elastic stresses significantly. Controlling the formation of crystal defects like dislocations is crucial for the Silicon Cz crystal growth process. Dislocation formation and multiplication is attributed to an excessive stress induced inside the crystal larger than critical value of Si resolved shear stress (σ_{Crss}). Dislocations have unfavourable impacts on the PV production efficiency. High thermal stresses and melt/crystal interface shape deflection are known as possible reasons of dislocation generation.

In the present work, a set of 3D simulations are performed for a crystal in the <100> orientation. The pulling rate is imposed as an intermittent function, and the transient effects on the thermal field, the interface shape and resulting thermo-elastic stresses are computed. The excess stress from critical resolved shear stress value is calculated for all 12 dislocation slip systems. Simulation results indicate that sharp variation in pulling rate results in higher risk of dislocation generation. The model is used to assess the relation between the fluctuation magnitude and frequency on the resulting thermo-elastic stresses.

THERMAL STRESS ANALYSIS OF CZOCHRALSKI GROWN SILICON CRYSTALS ACCOUNTING FOR DISLOCATION PINNING BY OXYGEN

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Controlling the formation of crystal defects like dislocations is crucial for the Czochralski Silicon crystal growth process. Dislocation formation and multiplication is attributed to stresses induced inside the crystal larger than the critical value of resolved shear stress for Si (σ_{Crss}). The presence of oxygen (the main impurity in solar grades silicon) in Si crystal influences the multiplication and motion of dislocations due to locking effects. Consequently, an accurate map of the excess stress field can be only obtained by considering the dislocation pinning effect of oxygen.

In the present work, the effect of dislocation pinning by oxygen on the excess thermo-elastic stresses inside the crystal has been studied using 3D thermal and mechanical simulations accounting for crystal orientation and elastic anisotropy. To compute the unlocking stress for a given bulk oxygen concentration, a model for oxygen transport by diffusion into the dislocation core is employed taking into account the thermal history. The core concentration is used to compute the unlocking stress and, thereafter, the excess shear stresses for all 12 slip systems. A case is carried out to analyse the effects of bulk oxygen concentration and its (radial and axial) distribution as well as cooling conditions on the computed excess stresses. It is shown that neglecting the dislocation pinning effect by oxygen can lead to significant error in prediction of dislocation generation and multiplication inside the crystal.

A NEW APPROACH TO FREE-STANDING GaN USING Β-Ga2O3 AS A SUBSTRATE

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Hexagonal GaN (0001) has been grown on β-Ga2O3 (100) making use of an in-situ intermediate (low-temperature) buffer layer. A simple method for self-separation of bulk GaN from the Ga-oxide substrate is reported. The structural properties of the GaN and GaN/β-Ga2O3 interface were investigated by high-resolution X-ray diffraction and electron microscopy techniques. The properties of GaN deposited on β-Ga2O3 are compared with those of samples grown on sapphire.

A CRYSTALLIZATION-LIKE MODEL THAT DESCRIBES THE KINETICS OF AMYLOID FIBRIL FORMATION

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Associated to neurodegenerative disorders such as Alzheimer's, Parkinson's, or Prion diseases, the conversion of soluble proteins into amyloid fibrils remains poorly understood. Extensive "in vitro" measurements of protein aggregation kinetics have been reported but no consensus mechanism has emerged until now. This contribution aims at overcoming this gap by proposing a theoretically consistent Crystallization-Like Model (CLM) that is able to describe the classic types of amyloid fibrillization kinetics identified in our literature survey. Amyloid conversion represented as a function of time is shown to follow different curve shapes, ranging from sigmoidal to hyperbolic, according to the relative importance of the nucleation and growth steps. Using the CLM, apparently unrelated data is deconvoluted into generic mechanistic information integrating the combined influence of seeding, nucleation, growth and fibril breakage events. It is notable that this complex assembly of interdependent events is ultimately reduced to a mathematically simple model, whose two parameters can be determined by little more than visual inspection. The good fitting results obtained for all cases confirm the CLM as a good approximation to the generalized underlying principle governing amyloid fibrillization. A perspective is presented on possible applications of the CLM during the development of new targets for amyloid disease therapeutics.

NANOMECHANICAL STUDIES OF MOVPE GROWN Ge/GaAs EPILAYERS

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The study of mechanical properties has gained much attention in recent days as the accurate measurement of the mechanical properties of the germanium thin films is required to use them as functional elements in device fabrication. Germanium is an interesting material for high efficiency solar cell applications.

In this paper we report on the heteroepitaxial growth of germanium film on gallium arsenide substrate at different growth temperatures and were characterized for morphology, structural and mechanical properties. Germanium epilayers were deposited by horizontal home-made metal organic chemical vapor phase epitaxy equipment, using isobutyl-germane as Ge source and hydrogen as carrier gas. The samples were grown at different temperatures of 650, 600 and 550 ˚C for 15 minutes.

Atomic force microscopy measurements to study the surface morphology of the grown samples were carried out and the numerical analysis was performed using Gwyddion software to estimate surface roughness in the form of average root mean square roughness values found to be in the range of ~ 20 to 35 nm. High resolution X-ray diffraction measurements were performed for structural analysis. The mechanical properties of the grown samples were studied with nanoindentation using Berkovich and Vickers indenters, hardness and elastic modulus values have been determined. The defects induced phenomenon due to change in load on Ge/GaAs epilayers have been elucidated.

GROWTH AND MAGNETIC BEHAVIOR OF SOL-GEL PREPARED TM DOPED NANOCRYSTALLINE TIO$_2$

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We report the growth of [Fe, Co & Mn]$_x$TiO$_2$ (x=0.01,0.02 &0.04) nanocrystals prepared by Sol-Gel technique, followed by freeze-drying treatment at -30°C temperature for 12hrs. The obtained Gel was thermally treated at different temperature like 200,400,600, 800°C. X-ray Powder Diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDX), was used to study its structural properties. The XRD pattern shows the coexistence of anatase phase and rutile phase. Thermal gravimetric analysis (TGA) shows that dopant concentration affects thermal decomposition. Photo luminescence (PL), was used to study its optical properties where all TM doped TiO$_2$ nanostructures show an appearance of Red shift relative to the bulk TiO$_2$. Vibrating Sample Magnetometer (VSM) was used to determine room temperature FM behavior of TM doped TiO$_2$ materials. The effects of [Fe,Co & Mn] doped TiO$_2$ on the formaldehyde photo catalytic degradation have been studied and discussed.

THERMAL AND STRUCTURAL ANALYSIS FOR A REACTOR USED IN HYDROTHERMAL SYNTHESIS UNDER SUPERCRITICAL CONDITION

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This paper presents a thermal and structural analysis for hydrothermal reactor under high pressure and high temperature operating condition. The study was performed using ANSYS software to obtain the stress and strain for equipment used in hydrothermal synthesis in crystal growth process. This study was performed for different temperatures in variation domain from 300°C to 500°C and different pressure from 1000 bar to 2500 bar. The stress results obtained from numerical simulation show the maximum loadings and equipment behaviour in high pressures and temperatures condition. The thermal analysis obtained show the heat flux in reactor body and also this thermal analysis was coupled with structural analysis to obtain the very good agreement with the normal operating condition from hydrothermal synthesis. In conclusions, this paper presents a numerical analysis regarding the reactor behaviour in hydrothermal process.

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S2

Organic Materials: Advanced Methods, Advanced Applications

Invited Papers

MODULATION OF SURFACE CHARGE TRANSFER THROUGH COMPETING INTERACTIONS

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The charge transfer of the π-donor molecule tetrathiafulvalene (TTF) on Au(111) as a function of coverage (< 1 ML) has been investigated both experimentally, by means of LTSTM and high-resolution XPS using synchrotron radiation and theoretically with the help of DFT calculations using the SIESTA code [1,2]. The charge transfer is modulated by two competing interactions, long-range repulsive (Coulombic) and short-range attractive (H-bonding and van der Waals), leading to characteristic 1D, 2D, and labyrinth-like patterns. In the low coverage regime (< 0.2 ML) TTF monomers build 1D quasiperiodic arrays with intermonomer separations of 2-3 nm, due to Coulomb repulsion, which has been assigned to a 1D Wigner crystal [3]. The work function exhibits a quadratic dependence on coverage, which is essentially due to the decrease of the mean monomer surface dipole moment, following the classical Langmuir's depolarization model (Pauli's push-back effect cannot be excluded). The XPS S2p lines can be decomposed in two components, separated by 0.4 eV. This is attributed to the coexistence of TTF molecules in different charge states, i.e., neutral and charged, which responds to the short timescale associated to the photoemission process.

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MOLECULAR NANOCRYSTALS CONFINED IN SILICATE NANOPARTICLES: A NEW FAMILY OF ULTRABRIGHT FLUORESCENT TRACERS FOR MEDICAL IMAGING

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We have developed the confined nucleation and growth of organic nanocrystals into the pores of silicate matrices. This spatial confinement of nanocrystals, accurately modulated through the sol-gel chemistry, provides narrow size distributions, prevents the coalescence of nanocrystals, and stabilizes them through one-step processes. Thus, nanocrystal diameters ranging between 20 and 800 nm can be obtained with size distributions of 10%. These hybrid organic-inorganic nanocomposites combine high fluorescence efficiency and photostability of nanocrystals with the easy shaping and transparency of the inorganic amorphous matrix. We have applied this generic confined nucleation and growth process to the preparation of nanocrystals embedded in bulk or sol-gel thin films. The organic nanocrystals, being constituted of high numbers of molecules, exhibit high cross section absorptions and high fluorescence intensities while having single spectroscopic signatures. We have demonstrated the feasibility of using molecular nanocrystals in bulk silicate matrices for the development of nonlinear filters used in optical limiting applications at visible wavelengths. Then we have optimized the preparation of fluorescent nanocrystals confined in silicate thin films for the development of a new type of signalization function for chemical and biological sensors based of fluorescence quenching and turn-on emission. Recently, we have also undertaken a new shaping of organic nanocrystals grown in silicate spheres by using an original spray-drying process. These new 0D hybrid nanocomposites, constituted by crystalline organic cores surrounded by amorphous silicate shells, are promising for bio-imaging: very bright fluorescence, easy to adjust the particle size between 20 and 500nm, good stability, low production cost of large amounts of tracers while the silicate shell is transparent in the visible and biocompatible with hydrophilic character that is crucial for the dispersion of labels and their furtivity *in vivo*.

IN-PROCESS ANALYTICAL TECHNIQUES FOR MONITORING, CONTROLLING & SCALING-UP SOLUTION CRYSTALLISATION PROCESSES

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Crystallisation from the solution phase represents a key unit process for the pharmaceutical and fine chemical industries conferring the advantage of facilitating through processing both the target products' isolation/separation and purification. Despite this, and the fundamental-scale understanding, and hence control, associated with the crystallisation of such high added-value materials, is surprisingly rather weak. Reflecting this, the ever stringent quality enhancement demands that the regulatory bodies are providing a research driver towards significant activity in the area of crystal growth fundamentals, in particular in-process monitoring and control. In this respect the chemical and life sciences industries are very much following the revolution undertaken by the semiconductor and IT industries in the development of integrated circuit more than 50 years ago.

Specifically, the paper will overview recent research which has been aimed to improve fundamental understanding associated with crystallisation processes, notably through the use of in-situ techniques to yield capabilities for rapid process and product development together with their scale-up to provide highly robust manufacturing roots. Through this, in the modern pharmaceutical industry, the development and use of process analytical technology (PAT) for monitoring and controlling crystallisation has now become a pre-requisite need for preparing target drug compounds in a consistent and reliable manner.

Against this perspective the lecture will highlight both the use of PAT techniques and their synergy with molecular scale modelling for crystallisation monitoring and control, notably

- Optical turbidity, meta-stable zone widths and nucleation kinetics;
- ATR FTIR spectroscopy: species identification, solution concentration and supersaturation;
- Acoustic attenuation spectroscopy: crystal size, size distribution and solids concentration;
- X-ray diffraction, phase composition and identification of polymorphic form;
- Digital video microscopy, particle shape and crystal habit modification.

presenting an integrated review of recent developments in the field.

AROMATIC DERIVATIVES ORGANIC SEMICONDUCTORS

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Organic materials are considered an important alternative to inorganic semiconductors in the manufacturing of electronic and opto-electronic devices because of the great diversity of the organic compounds, their remarkable properties, low production cost, compatibility and complementarity with the silicon technology and large area of potential utilization from Organic Light Emitting Diodes (OLEDs) to solar cells and from lighting source to sensors.

We present some comparative investigations on the electrical and optical properties of the interfaces inorganic/organic, organic/organic in different single and multi organic layer based structures.

Some particular organic semiconductor/metal contacts (examples: m-DNB/Cu; perylene/In, Al, Ga, Ag, Cu and PTCDA/Cu, Al, Ga, Ag) will be presented and the transport properties in ITO, Si/organic layer(s)/metal heterostructures (examples: ITO/PTCDA/Cu, Al; Si/m-DNB, ZnPc/Cu; ITO/PTCDA/Alq3/Al; Si/perylene, PTCDA, ZnPc/Cu, Al) and metal/organic layer/metal (examples: Al/ZnPc, TPyP, PTCDA, α-NPD/Al) will be investigated.

Concerning the bulk organic crystals, our interest will be focalised on aromatic derivatives such as meta-dinitrobenzene (m-DNB) and benzil characterised by large transparency domain and good fluorescence emission.

New results will be bought in the field of composite films prepared from polymeric matrix and active monomeric inclusions based on π conjugated systems containing functional groups with special properties to improve the emission properties of the matrix and the charge carrier mobility.

METASTABLE STATE AND RELAXATION IN FERROELECTRIC TRANSITION OF PURE TGS CRYSTAL

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Triglycine sulphate crystal (TGS), which has an extended hydrogen bridge bonding system, undergoes a typical second order phase transition. A prominent instability of permittivity components was found walking temperature up and down, several time, through the Curie point (C.P. ≈ 49 °C). TGS samples (1 mm thick / ~0.5 cm^2 surface) where cleaved from TGS crystals grown in the paraelectric phase, by slow solvent evaporation. Samples were polished and silver electrodes were painted. Dielectric dispersion of pure TGS samples was studied on a large frequency (1 Hz ÷ 10 MHz) and temperature (-120 to +65 °C) range, crossing up and down the C.P. several times.

After the first up/down run through the C.P. both real and imaginary components of permittivity increases unexpectedly more than one order of magnitude in ferro phase.

It followed a long period (many weeks) of metastable state, due to ferroelectric domain relaxation. In the second, up/down run through the C.P., permittivity gets the same values in the paraelectric phase, but not in the ferro phase.

In Cole-Cole, representation two types of relaxations were found. The higher frequency relaxation (typical relaxation time of $\tau_H = (3\text{-}4)\cdot 10^{-7}$ sec.) is almost constant on -40 /+45 °C temperature range (a slight non-Arrhenius dependence). This is the “critical slowing down” mechanism related with long distance order in the lattice.

The lower frequency relaxation ($\tau_L \sim 10^{-3}$ sec) is related with ferroelectric domain cropping and has a thermal evolution, with the activation energy of 0.7 eV (~28 k_BT).

An unusual, mid frequency relaxation ($\tau_M \sim 10^{-5}$ sec.), was clearly discerned in some temperature range of the ferroelectric phase. Apparently, it does not have a physical support, being rather an interaction of the previously two related mechanisms.

Oral Presentations

SURFACE AND MORPHOLOGICAL PROPERTIES OF INTERPOLYMER COMPLEXES AND BLENDS BASED ON CELLULOSE ACETATE PHTHALATE/HYDROXYPROPYL CELLULOSE

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The surface properties of cellulose acetates phthalate(CAP)/hydroxypropyl cellulose(HPC) films obtained from solution in 2 methoxyethanol at different mixing ratio (100/0, 25/75, 50/50, 75/25 and 0/100% (v/v) CAP/HPC) were discussed. The films were prepared by a dry-cast process. Chain associations determined by presence of hydrogen bonding and implicitly of HPC content represent important variables, governing the morphology of the membranes.

The results concerning the surface tension properties show that the polar components obtained for each cellulose acetate phthalate(CAP)/hydroxypropyl cellulose(HPC) films have a lower electron donor parameter, comparatively with the electron acceptor one, and also that the apolar components of surface free energy are higher than the polar components. Moreover for 50/50% (v/v) CAP/HPC films apolar component attaining a maximum values while polar components, electron donor, electron acceptor parameters and total surface tensions have a minimum values. Generally, increase of hydroxypropyl cellulose content determines a minimum value of hydrophilicity for 50/50% (v/v) CAP/HPC films. The study put into evidence the competition between the effect of hydroxypropyl cellulose content, solution properties and even their possible association, a phenomenon which should be considered in any further study on surface morphology.

Surface properties were correlated with data concerning the atomic force microscopy images. Each micrograph shows that the films surfaces are characterized by pores and nodules of different size and intensity. Values of average roughness and pore volumes and their diameters are correlated with hydrophilicity properties.

Surface wettability trends were also studied by means of the free energy of hydration between compounds and water. These informations may contribute to extending the possible CAP/HPC blends applications.

STRUCTURE-RHEOLOGY PROPERTIES RELATIONSHIP IN COMPLEX ALICYCLIC POLYIMIDES

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Modern polyimide materials are the subject of great interest all over the world, due to their specific properties. In this context, new alicyclic polyimides, poly(BOCA-ODA) and poly(BOCA-p-BAPS), having a common monomer based on bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride (BOCA), and different aromatic diamines, 4,4’-oxydianiline (ODA) and 4-(4-((4-(4 aminophenoxy)phenyl)sulfonyl)phenoxy) (p-BAPS), respectively, were synthesized and investigated by rheological method in N-methyl-2-pyrrolidone, at different concentration and temperatures. These investigations indicate that both polyimides exhibit a Newtonian behaviour. The interactions among chain segments induce modifications of dynamic viscosity, generated by chemical structures and polyimides affinity for the used solvent. These modifications are reflected in the activation energy, evaluated from Arrhenius expression. Increasing of the rigid component amount of poly(BOCA-p-BAPS) induces stronger polymer entanglements and interactions among the chain segments, as revealed by the higher flow activation energy values, compared to poly(BOCA-ODA). The effect of the chemical structure of polyimides on the viscoelastic properties is reflected in the mobility of segments from the shear field. It is found out that the frequencies corresponding to the crossover point, which delimit the viscous flow from the elastic one, exhibit lower values for the poly(BOCA-p-BAPS) with lower flexibility. Also, the storage and loss moduli for these partial aliphatic polyimides exhibit the power law dependence on frequency, where exponents around 1 and 2, respectively, are characteristic to viscoelastic fluids. In conclusions, depending on the molecular chains characteristics of individual polyimides, the factors that contribute to the interactions in the polymer system can be optimized for subsequent microelectronic applications, in which relatively low permittivity and high thermal stability are required.

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COPOLYMER ARCHITECTURES CONTAINING PERYLENEDIIMIDE AND OXADIAZOLE DONOR AND FLUORENE ACCEPTOR UNITS FOR OPTOELECTRONIC APPLICATIONS

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In an expanding field of organic optoelectronic devices which utilize polymers as active materials, it is worthwhile to design soluble and film-forming polymers with enhanced electron transport property and fluorescence behaviour. Therefore, we considered interesting to synthesize a series of copolyimides containing oxadiazole and perylene units as electron-donor groups and fluorene moieties as electron-acceptor groups in the main chain. The combination of donor-acceptor groups in the same chain in attempts to achieve balanced electron–hole injection and transport avoids the use of intermediate transport layers that induce some limitations. In this work we report on the synthesis and characterization of new polyimides containing the above mentioned chromophores with emphasis on their thermostability, film forming ability, morphology, and fluorescence behaviour. The synthesized polymers are highly thermostable, the initial decomposition temperatures being above 400°C and display glass transition temperatures in the range of 245-304°C. They are soluble in polar amidic solvents, such as N-methylpyrrolidinone (NMP), and can be cast into thin films. Their photo-optical properties were assessed on the basis of UV-absorption and photoluminescence (PL) spectra. It was found that those polymers show absorption peaks in the UV region due to fluorene and oxadiazole fluorophores and in the visible spectral range due to perylenediimide moieties. Upon excitation with UV and visible light corresponding to each chromophore, PL emissions in the blue and green-yellow spectral range were obtained. The FRET phenomenon between oxadiazole to perylenediimide chromophores was observed to occur for which the oxygen bridge appear to be responsible.

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TECHNOLOGICAL SOLUTION FOR MANUFACTURING HIGH ADHERENT CONDUCTIVE THIN FILMS ON FLEXIBLE ORGANIC SUBSTRATES

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A major problem in using flexible substrates is the poor adherence of the deposited conductive thin films.

The paper presents some technological experiments used for strengthening the adherence of conductive thin films on flexible organic substrate. The substrate was an organosilicon compound, polydimethylsiloxane (PDMS).The interconnection conductive thin films are Ti /Au sputter deposed and photolithographic configured and Ag conductive ink for ink jet technique.

The PDMS substrate is processed in RIE plasma in order to functionalize the surface and to have a better adherence of the conductive films.

For the Ti/Au films we experiment 2 films: the Ti 50nm/Au 400nm and Ti 50nm/Au 800nm.

For RIE we used SF6 with O2 or Ar plasmas. The RF power was 250W and 50W and the pressure 30Pa. The report of SF6 and O2 or Ar flows were varied. The process time was 10 minutes.

We also study the modification of treatments in photolithography technological process flow request by the PDMS substrate.

For ink jet technological flow, a new post ink deposition treatments were proposed.

The characterization techniques used were: optical microscopy; SEM microscopy; contact angle measurements; AFM; pull test for adherence

The result of experiments was a new technological sequence in the technological flow of the device. We obtain improved adherence of films both in the case of metal deposition and also in the ink jet imprinting.

The final application was a flexible white LED matrix.

We also put in evidence a problem in using PDMS as flexible substrate- cracking apparition after deposition process. The problem was solved using a change in recipe of PDMS sample manufacturing.

COMPOSITE POLY(ETHER IMIDE)/ZnO WITH APPLICATION IN AIR RELATIVE HUMIDITY SENSING

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The polymer was prepared by thermal treatment of the corresponding poly(amic acid), which was synthesized by polycondensation reaction of 2,2-bis[(3,4-dicarboxyphenoxy)phenyl]propane dianhydride (**6HDA**) with 4,4'-(1,3-phenylenedioxy)dianiline (**PODA**), in N-methyl-2-pyrrolidone as solvent, at a concentration of 10-15%. ZnO particles calculated to be 5.5% in the composite material were mixed in poly(amic acid) solution and vigorously stirred, for 4 h, at room temperature. The resulting homogeneous suspension was cast onto glass plates to obtain films. Finally, the composite films were heated at 80, 120, 160, 200 and 220°C, each for 30 minutes, in order to remove the solvent and to perform the cyclodehydration reaction of the poly(amic acid) to the corresponding imide structure. Such films having a thickness in the range of 40-50 μm were dried under vacuum at 100°C. For 1000 KHz frequency, the dielectric constant was 2.57 and the loss factor (tgδ) was $1.9 \mathrm{x} 10^{-1}$. The sensor structure comprise a substrate printed circuit board PCB type FR-4, a Cu interdigitated array electrode and a thin film sensitive layer obtained by dissolving composite polymer/ZnO in chloroform and deposed by dipping method. For the measure range of air relative humidity 40-95% RH and 30^{o}C temperature testing, the resistance variation of sensor was between 5.4 MΩ and 30 KΩ. The sensor hysteresis was 4% and the temperature coefficient was 0.35%RH/^{o}C for 30-50^{o}C temperature range.

LASER-STRUCTURING OF AN AROMATIC PHOTOSENSITIVE POLYIMIDE

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The structuring by direct laser irradiation of polyimides has received a great attention since it represents a better alternative to rubbing method of processing surface morphology. The effect of laser exposure consists in clean etching of the material with micron size precision. In this work, the preparation of an aromatic photosensitive polyimide from 3,3',4,4'-benzophenone tetracarboxylic dianhydride and 4,4'-diaminodiphenyl methane is reported. Also, a study of the polyimide films structuring by laser beam irradiation at different laser fluencies is performed. The changes induced by the employed patterning technique in the chemical structure are reflected in the surface morphology. According to atomic force microscopy data a cone-like structure is formed and the corresponding mechanism is analyzed taking into account the irradiation conditions. The obtained results indicate that the investigated photosensitive polyimide is a good candidate for microelectronic packaging, liquid crystal alignment layer and cell culture substrates with tunable morphology.

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Poster Presentations

ZnO STRUCTURED FILMS SYNTHESIZED BY ELECTROLESS DEPOSITION ON POLYMER NANOSPHERE ARRAYS

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The electroless technique is a simple, efficient and suitable method for industrial preparation. In the present study ZnO nanostructures were obtained by electroless deposition using polymer nanosphere arrays. The polymer spheres were assembled on glass substrates using dip-coating technique. The ZnO nanostructures were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), optical and electrical measurements. The SEM images prove that aligned rod-shape ZnO particles with a fairly uniform diameter of around 500 nm and length of 1 μm are synthesized.The XRD studies demonstrate that the ZnO nanoparticles have a hexagonal wurtzite structure. In the photoluminescence spectra of ZnO films deposited on polymer array an ultraviolet emission band peaked at about 2.9 eV is observed. The electrical measurements were carried out in the dark and under illumination. The ZnO films deposited on glass substrate have a resistivity of ~5.4x10^3 Ωcm, measured in the dark. On polymer array ZnO nanostructured films present a non-linear current-voltage characteristic with decreasing resistivity from 1.5x10^4 Ωcm at low voltages to 3x10^2 Ωcm at 80V, which could be explained by a transition from ohmic to space charge limited conduction due to traps. Strong carrier photogeneration was evidenced for these films, the resistivity decreasing 2-3 orders of magnitude under illumination. In order to check the possibility of using such ZnO nanostructured films in solar cells an organic absorbent (copper phthalocyanine) has been thermally evaporated. Current-voltage characteristics of the ZnO - copper phtalocyanine structures revealed diode-like curves with generated photoelectric voltages of about 0.1-0.7V having current densities of 1x10^{-8} A/cm^2 for ZnO films on glass substrate and 1.4x10^{-6} A/cm^2 for ZnO nanostructured films on polymer nanosphere array.

MOLECULAR DESIGN OF SOME SEMI-ALICYCLIC POLYIMIDES FOR LIQUID CRYSTAL ALIGNMENT LAYERS

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Thin polyimide (PI) films are the most commonly employed liquid crystal alignment layers (LC ALs) because of their good thermal and chemical stability, excellent mechanical strength and insulation ability. For these applications the PI film surfaces need to be adapted. In this work, two semi-alicyclic PI were prepared by solution polycondensation of bicyclo[2.2.2.]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride (BOCA) with two different aromatic diamines: 4,4-oxydianiline (ODA) and 4-(4-{[4-(4-aminophenoxy)phenyl]sulfonyl}phenoxy)aniline (*p*-BAPS).

The morphology of these PIs was processed by unidirectionally rubbing with a cloth to produce a uniform alignment of the LC molecules and to generate an appropriate LC pretilt angle, preventing the creation of disclinations in the LC cells. The dimensions of the features created on the PI film surface are influenced by the rubbing conditions. The distribution of the LC molecules on such surface, depends on the shape of the pattern evidenced by angular spectrum evaluated by atomic force microscopy technique. The resulted stability of LC molecules recommends these materials for LC display devices applications.

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EFFECT OF RUBBING MATERIAL TEXTURE ON POLY(OXADIAZOLE-IMIDE) FILMS MORPHOLOGY

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Some poly(oxadiazole-imide)s containing naphthalene rings were synthesized and their suitability as liquid crystal alignment layers was evaluated. The polymer surface was patterned by subjecting it to a rubbing process with two different textile materials: cotton velvet and synthetic velvet. Effect of textile fiber on polyimide morphology developed after rubbing process was investigated through atomic force microscopy (AFM) measurements. The different sizes of the nanochannels are dependent on both fiber dimension and flexibility. Therefore, the stability and pretilt angle of liquid crystal molecules can be tuned depending on the rubbing cloth characteristics. The alignment behavior of a nematic liquid crystal casted on the processed poly(oxadiazole-imide)s was analyzed through polarized optical microscopy and discussed according to the obtained differences in the sample surface topography.

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THERMAL AND MORPHOLOGICAL PROPERTIES OF P4VP/MWCNT NANOCOMPOSITES PREPARED BY ULTRASONICATION AND SHEAR CASTING METHOD

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Blending polymers with inorganic filler offers new perspectives in obtaining hybrid materials with various utilities ranging from aeronautics to microelectronic industry. The aim of this work is to obtain thermally conductive composites from poly(4-vinylpyridine) (P4VP), which is a high-performance polymer, which exhibits high electrical and thermal conductivity, and multiwall carbon nanotubes (MWNT), which present excellent mechanical, thermal and electrical properties. The dispersion and orientation of MWNT in the polymer matrix was performed by ultrasonication and shear casting method. The influence of the nanofiller content on composite morphological features of the P4VP/MWCNT was analyzed by atomic force microscopy and scanning electron microscopy. Thermal resistance of investigated composites were evaluated, revealing a good thermal stability over 100°C. The thermal conductivity of these materials was evaluated by considering it as an additive function of the P4VP and MWNT content. The obtained results indicate that the studied nanocomposites are good candidates as protecting materials of chips overheating in power electronics.

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ORGANIC HETEROSTRUCTURES BASED ON ARYLENEVINYLENE OLIGOMERS OBTAINED BY MAPLE

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The molecular structure of the oligomers based on aromatic amines associated with the donating groups situated at the ends linked by conjugated bridges determining good optical properties and reduced steric effect and improving the conduction recomand these materials to optoelectronic applications. The method used to prepare p-n heterostructures with arylenevinylene oligomers was Matrix Matrix Assisted Pulsed Laser Evaporation (MAPLE). Arylenevinylene oligomers containing triphenylamine (P78) or N-alkylcarbazole (P13) donating groups were used as p type conduction layer and tris-8-hydroxyquinoline aluminium (Alq3) as n type conduction layer. Single or double organic layers deposited by MAPLE on ITO/glass and silicon substrates were characterized by spectroscopic methods (UV-VIS, PL and FTIR), by Atomic Force Microscopy (AFM) and with four point probe technique. The I-V characteristics of the p-n heterostructures were recorded in dark. The correlation between the morphology and thickness of these films and the electrical properties of the heterostructures was emphasized. Generally the I-V characteriscs plotted are symmetricaly and were not observed rectifing properties for thsese structures. The best current obtained (10^{-5} A for 1V applied voltage) was for the ITO/P78/Alq3/Al heterostructure.

DYNAMICAL LINE SHIFTS IN HIGH-FIELD ELECTRON PARAMAGNETIC RESONANCE - APPLICATIONS TO POLYMERIC SYSTEMS

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High-field electron paramagnetic resonance spectroscopy is a powerful tool to investigate, with ultra-high angular resolution, the rotational dynamics of complex systems like polymers, viscous fluids and glasses. Here, we present a simplified analytical expression of the shifts from the rigid limit positions of the turning and saddle points. They are used to analyze the experimental spectra of the spin probe TEMPO dissolved in polystyrene in the range 50-270 K. We found that the best-fit parameters yield equally acceptable best-fits of the overall spectrum by numerical simulations and independent analytic expressions of dynamical line shifts.

THE SIGNIFICANCE OF ANTIMICROBIAL FINISHING TREATMENT DESIGNED TO SPECIAL CLOTHING

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Textiles for clothing with special use are treated to improve quality of users life . Besides the discomfort produced by mechanical hardness of the material and property to prevent the repellence of moisture from sweat, a particularly important role is its biological effect. The antimicrobial activity of treated textile material may be beneficial for carriers, reducing the microbial load of the skin, under sustained effort.

The present study presents the antimicrobial efficiency, and roughness analysis of polyester material treated with ZnO powder, having different concentrations of suspensions, using different dispersion methods of active agent in methanol.

This treatment has been performed by padding different concentrations (1%, 3%, 5%, 7%) of zinc oxide suspensions onto textile materials, aiming at obtaining a product with antimicrobial properties, as well as the achievement of optimal values in terms of comfort. These results can briefly demonstrate the antimicrobial effect of ZnO, through a direct contact with carrier human skin, in case of treatment of textiles utilized in achievement of some work equipments.

SIMULATIONS OF GROWTH AND EQUILIBRIUM FORM OF UREA CRYSTALS

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Theoretical growth and equilibrium forms of urea crystals ($CO(NH_2)_2$) were constructed from the calculated attachment energies which are assumed to be directly proportional to the growth rate for F faces. F forms used in growth and equilibrium form of urea crystals ($CO(NH_2)_2$) are {001}, {110},{111}, {-1-1-1}, {200} and {101}. The theoretical growth and equilibrium forms of crystalline urea were constructed from the calculated attachment energies theoretical growth forms and equilibrium forms are simulated for different values of the charges in Model I, II and III.

Urea crystals ($CO(NH_2)_2$) crystallize in a tetragonal system and have a space group : $P\text{-}42_1m$, and the parameters for the unit cell: a=b= 5.565 Å and c= 4.684 Å.

Crystal structure determines the surface structure and related surface energies, e.g. specific surface energy, attachment energy, dispersion because Van der Waals attraction and Born repulsions. Were obtained this energies for urea crystals ($CO(NH_2)_2$) as they control the crystal growth form and the equilibrium form of a crystal.

Attachment energy is at least for F faces directly proportional to their growth rates(Hartman and Bennema,1980).

Model for calculations is an electrostatic point charge model. In the present paper is presented the structure analysis of urea crystals ($CO(NH_2)_2$) using Hartman Perdok Theory (Engkvist et.all., 2000). Were found from simulations that urea crystals ($CO(NH_2)_2$) at a first analysis have the next F faces: {001}.{110},{111}, {-1-1-1}, {200} and {101}. The conclusions from the present study is that were found the next growth forms with the next F faces: {001} and {110}, and the crystals that have a volume of V= 206.51 $Å^3$ and a surface S= 213.99 $Å^2$, and a growth form of a crystal with the next F forms: {101}, {200}, {111} with a volume V= 607.06 $Å^3$ and a surface of S=403.72 $Å^2$.

Equilibrium form of urea crystals contain the next F faces: {001}.{110},{111}, {-1-1-1}, {200} and {101} and have a volume: V = 23.531$Å^3$ and a surface of S= 23.531 $Å^2$.

OPTICAL AND ELECTRICAL PROPERTIES OF HETEROSTRUCTURES BASED ON MALEIC ANHYDRIDE-ANILINE DERIVATIVES MONOMERS THIN FILMS FOR OPTOELECTRONIC AND PHOTOVOLTAIC APPLICATIONS

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Organics are a class of materials offering a great potential for optoelectronic applications as Organic Light Emitting Devices (OLEDs) for displays and general lightning and for photovoltaic applications as Organic Photovoltaic devices (OPVs) such as solar cells. To reduce the electrical conduction limitations associated with organic semiconductors it is necessary to use adequate device structures to overcome the problems associated with the high resistivity of the organic materials and the difficulty to obtain a good charge carrier injection from electrodes in the organic layers.

This paper presents some investigations on the effect of an amidic monomer (synthesised from maleic anhydride and aniline derivatives) coating on the optical and electrical properties of the ITO transparent conducting electrode deposited on glass. The electrical properties of different types of heterostructures based on single/multiple active organic layers [p type: pentacene; rubrene; n type: 2-(4-biphenyl)-5-(4-tert-buthylphenyl)-1,3,4oxadiazole (PBD) and 5,10,15,20-tetra(4-pyridyl)-21H,23H-porphine, synthetic (TPyP)] prepared between an ITO/glass electrode covered with an amidic monomer layer and a Si electrode have also been analysed.

We will emphasise that the electrical conduction in single/multi organic layer heterostructures based on amidic monomers depends on the energetic properties of the organics involved, the morphological particularities of the organic layer in contact with ITO or Si electrode and the presence of dipolar layer at the contact with electrodes (ITO, Si).

NONLINEAR OPTICAL PHENOMENA IN DOPPED AND IRRADIATED AROMATIC DERIVATIVES CRYSTALS

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Organic molecular crystals are interesting for applications in photonics in optical signal processing and integrated optics because of their properties associated with the presence of the delocalized π electrons cloud and, donor and acceptor substituent groups to the aromatic nucleus determining significant inductive and mezomeric effects at the molecular level and significant optical non-linear effect at the macroscopic scale. From theoretical considerations, high non-linear coefficients can be anticipated for aromatic derivatives compounds such as meta-dinitrobenzene (m-DNB) and benzil.

Pure and doped (with iodine) m-DNB crystals and pure and doped (with iodine, silver, sodium, naphthalene, m-DNB) benzil crystals have been grown from the melt in a vertical configuration (thermal gradient at the growth interface: 5-30 °C/cm; moving speed of the growth interface: 0.5-3 mm/h). The thick crystalline wafer (1.5-3 mm) obtained by mechanically polishing the wafers cut from these ingots have been either illuminated with the ultrashort pulse of a pulsed laser Spectra Physics “Tsunami” (maximum emission wavelength of 800 nm, pulse duration of 60 fs and different medium power) or irradiated at room temperature, in a direction normal to the wafer surface at UNILAC Darmstadt, with Ni ions with a kinetic energy of 11.4 MeV/u at different fluences of 1×10^{10} ions/cm2; 5×10^{10} ions/cm^2 and 1×10^{11} ions/cm^2 for a period of ~ 1 h. The effect of doping or irradiation on the optical non-linear phenomena such as Second Harmonic Generation and Two Photon Absorption Fluorescence has been analysed. The correlation between the intensity of the optical non-linear phenomenon and the position on the wafer was also studied by moving the focused beam across the surface and in depth of the wafer.

S3

Biocompatible Materials and Applications

Invited Papers

NOVEL BIOBASED MATERIALS FOR ENVIRONMENTAL AND BIOMEDICAL APPLICATIONS

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Nowadays, the use of renewable biobased carbon feedstock is highly taken into consideration because it offers the intrinsic value proposition of a reduced carbon footprint, in agreement with a sustainable development. Replacing petroleum-based raw materials with renewable resources is now a major concern. However the great majority of these polymers are developed for environmental purposes in order for instance, to improve the LCA (Life cycle analysis); but these systems are also more and more used for biomedical applications [1].

Two main cases will be presented with two different biomedical applications: (i) Plasticized chitosan-based materials to develop new membranes for dressing, in contact with the skin [2-3], (ii) PLA-based materials to develop scaffold elaborated by electrospinning for tissue engineering, for bone regeneration [4-6].

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RESEARCHES ABOUT THE COVALENT IMMOBILIZATION OF ENZYMES ON POLYMERS, FOR VARIOUS APPLICATIONS

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The conference will present some results concerning the bioactive polymer nanocomposites obtained by the covalent immobilization of various enzymes on polymers.

There will be highlighted results about the xylanase immobilization on cellulose acetate membranes targeting the use of the bioactive composites in the xylan depolymerisation in order to obtain new sweetener for soft drinks. Also the immobilization of xylanase on plyvinyl alcohol fibers leads to improvement in the enzymic depolymerization of xylan, with the reuse of the enzyme in other incubations.

The immobilization of acetylcholynesterase and tyrosinase on molecularly imprinted polyvinyl alcohol membranes, proved useful for the obtaining of electroconductive biosensors for pesticide pollutants (atrazine) monitoring in surface waters.

Electro conductive polymer, namely polypyrole, proved a good support for the covalent and entrapment immobilization of horseradish peroxydase, and other enzymes. The biocomposites are useful for the electro conductive biosensors for the nitrates and nitrites monitoring in waters for human consumption.

There are also presented the obtained results concerning the covalent immobilization of xylanase on acrylic or methacrylic hydrogels aiming the increase of the bio catalytic activity of the enzyme in polysaccharides depolymerization.

Oral Presentations

SYNTHESIZED MATERIALS FOR PROTECTING PAPER ARTEFACTS AGAINST BIODETERIORATION

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Most of the artefacts of cultural and / or historical value are exposed to fungal attack (especially fungi of the *Aspergillus sp* and *Penicillium sp.*) often causing irreversible damage [1]. The need for new methods of antifungal protection led to the development of a recipe based on hydroxyapatite and barium hydroxide, with promising results [2]. Lin e al. [3] presented the antibacterial effect of partially and totally strontium substituted hydroxyapatite. Based on these data, our group have synthesized and analytical characterized (trough energy dispersive X-ray fluorescence - EDXRF, X-ay diffraction - XRD, Fourier transform infrared spectroscopy - FTIR, thermal analysis) two series of substituted hydroxyapatite (with strontium and barium). The efficiency of the synthesized materials was evaluated by diluted inoculums technique on culture media, using simulated artefacts.

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IMPROVEMENT OF HYDROXYBUTYRATE BIOPOLYESTER PRODUCTION BY CO-SUBSTRATE AND NITROGEN SOURCE OPTIMIZATION

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Biosynthesis of polyhydroxybutyrate (PHB) from glycerol was enhanced by the addition of glucose as a co-carbon source and/or limitation of nitrogen in cultivation medium. Combination of glycerol and glucose as substrate and ammonium sulphate as nitrogen source in mineral medium resulted the optimal PHB production, in which PHB content of 89.0 ± 0.1 % and productivity of 0.11 g L-1 h-1 were observed after 72 h cultivation in 1-L reactor. Production of PHB in 10-L reactor under optimized condition in 1-L reactor was examined. Results displayed the maximamu PHB productivity and PHB content of 83.0 ± 1.2 % and 0.068 g PHB L-1 h-1, respectively.

BIOCOMPATIBILITY OF SOME NEW PHOSPHORUS-MODIFIED POLYSULFONES

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Polysulfones with bulky phosphorus pendant groups (PS-DOPO) with different substitution degrees were synthetized and studied the effect of the functional group on their hydrophobic character and their correlation with biomedical properties. Thus, one can say that increasing the substitution degrees of functionalized polysulfones leads to a slight increase in hydrophobicity - described by the work of spreading of water parameter.

Both red blood cells and platelets are extremely important in deciding the blood compatibility of a material. Adhesion of the red blood cells and cohesion of platelets to surface films were discussed in correlation with their specific applications in medicine. In this context, to analyze the using possibilities of functionalized polysulfones in biomedical applications and for establishing their compatibility with blood, the work of spreading of red blood cells and platelets, respectively, were considered. The materials which exhibit a lower work of adhesion would lead to a lower extent of cell adhesion than those with a higher work of adhesion. One can observe positive values for the work of spreading of red blood cells, and negative values for the work of spreading of platelets for modified polysulfones. Thus, a higher work of adhesion comparatively with that of cohesion for the red blood cells, but a smaller work of adhesion comparatively with the one of cohesion for platelets appears. In conclusion, the obtained data suggest that a small hydrophobicity can be correlated with a good adhesion of the red blood cells on the surface of the polysulfone films.

These results seem to be applicable for evaluating bacterial adhesion to the surfaces, and could be subsequently employed for studying possible implanted induced infections, or for obtaining semipermeable membranes.

EVALUATION OF STRUCTURAL AND FUNCTIONAL FEATURES OF IMMOBILISED BIOMOLECULES FOR BIOSENSOR DEVELOPMENT

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Using devices, such biosensors, employing biomolecules as analiytical tools offer advantages compared with conventional methods due to of their simplicity, specificity, selectivity and quick response for real-time analysis. Immobilised biomolecules usually show lower activity towards specific biomolecular interactions compared with free ones, thus biosensors stability and analytical performance depends on the matrix and on the immobilisation process used. The nature and bioactivity of electrode material and immobilization matrix present a very important aspect also. In this work we have immobilised glucoseoxidase enzymes using different matrix on mediated electrodes for biosensors development with the main purpose of enzymatic reactions characterization and used materials bioactivity. Electrochemistry and Electrochemical Impedance Spectroscopy (EIS) are used to characterise the structural and functional features of immobilised biomolecules on electrode surface.

NEW OXADIAZOLE-FUNCTIONALIZED N-VINYLPYRROLIDONE - MALEIC ANHYDRIDE COPOLYMER APPLIED IN BIOMIMETIC CRYSTALIZATION OF $CaCO_3$ NANOPARTICLES

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Copolymers of N-vinylpyrrolidone with maleic anhydride P(NVP-MA) are known to be biocompatible, with low toxicity and carriers of biologically active compounds, gradually releasing native drugs from their conjugates in response to changes in pH. 1,3,4-Oxadiazole derivatives exhibit diverse biological activities such as antimicrobial, anti-HIV, antitubercular, analgesic, hypoglycaemic, antitumoral etc. Here we report the synthesis and characterization of a polymer–drug conjugate based on P(NVP-MA) as support and 2-amino-5-(4-methoxy-phenyl)-1,3,4-oxadiazole, having antimicrobial and antifungal activity and of the $CaCO_3$ microparticles growth templated by this polymer. The copolymer was obtained by Yamazaki-Higashi method, involving carboxylic groups of the copolymer and the amine group of the oxadiazole derivative. The oxadiazole derivative contents relative to maleic comonomer units was estimated to be 29%. The obtained particles were characterized by scanning electron microscopy, polarized optical microscopy, X-ray diffraction (XRD), particles charge density, and electrophoresis. XRD showed that vaterite was the main polymorph present in $CaCO_3$ particles, about 95%, whereas the copolymer slightly favoured the formation of the most stable polymorph, calcite. Larger, more circular particles, and less porous were obtained for $CaCO_3$/copolymer sample. Due to the presence of the copolymer as template in $CaCO_3$ mineralization process the particles stability increased up to pH = 3.4. Thanks to biocompatibility properties of $CaCO_3$ and of the polyanion used in this study, these new composite materials may be used in biomedical applications, e.g. drug release systems.

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ULTRASOUND ASSISTED SYNTHESIS OF SEMICONDUCTOR OXIDE PHOTOCATALYSTS ON ORDERED MESOPOROUS CARBON

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Ultrasound is a useful method in the synthesis of a wide range of nanostructured materials. Here we are reporting the ultrasound assisted preparation of TiO_2 and Bi_2O_3 nanoparticles as photoactive species for removal of organic compounds from waste waters. Besides, in order to improve the photocatalytic activity, a modification method has been employed, coupling the semiconductor metal oxides with a non-metal matrix as carbon, due to its photoelectrochemical and electronic properties. It is known that the electronic property of carbon is closely related to its graphitic nature and photocatalysts supported on graphite-structured carbon would possess high carrier mobility and little recombination in the space charge region. The ordered mesoporous carbon with graphite structure and high surface area is used here to avoid the nanoparticles agglomeration and a good processability.

TiO_2 and Bi_2O_3 nanoparticles and their composites with ordered mesoporous carbon were prepared successfully by means of ultrasound assisted hydrolysis process. As the mesoporous carbon shows polar oxygenated groups on its surface, this might stimulate the formation of the composites, and enhance the interfacial combination of metal oxides with carbon nanostructures. The as-growth materials were characterized by using nitrogen sorption measurements, X-ray diffraction, UV-VIS and FTIR spectroscopies. Compared with obtained nanostructured oxides, their composites with mesostructured carbon showed higher surface areas and oxide nanoparticles have been well dispersed on the carbon surface preventing their agglomeration.

The photocatalytic activities of supported and unsupported nanoparticles on ordered mesoporous carbon were evaluated comparatively by quantifying the degradation of aromatic pollutants in aqueous media under UV and visible light. In comparison of pure oxides and their composites with carbon, higher photocatalytic activity of the composites is obtained and mainly attributed to greater band-gap energy and high surface areas.

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FUNCTIONALIZATION OF NANOSTRUCTURED NICKEL FERRITE FOR BIOMEDICAL APPLICATIONS

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Nanoscience is one of the most important research and development frontiers in modern science. Designing of nanostructured materials by choosing the proper synthesis protocol offers several advantages for biomedical applications. Introducing nanostructured oxidic compounds in the polymers with three-dimensional network leads to obtain the materials with pre-established architectures, excellent magnetic properties and high biocompatibility provided by high water content. These materials can act as linkers for active principles (antibiotics, antitumor, antifungal) being used for disease targeting and drug triggering applications.

In this paper are presented results obtained from synthesis, characterization and microbiological testing of nanostructured nickel ferrite functionalized with polyacrylamide based hydrogels. Ferrite is synthesized by sol-gel autocombustion method using citric acid as chelating/fuel agent. Hydrogels are synthesized by original method of polymerization-crosslinking simultaneous using a monofunctional crosslinking agent. Characterization of these materials was performed by IR, XRD and SEM. Onto these materials were immobilized antibiotic particles (ampicilin).

In vitro activity was tested using agar diffusion method. Microbiological tests were showing optimistic results which will be followed by magnetic properties investigations to promote the functionalized nanostructured nickel ferrite as a potential magnetic drug carrier.

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NEW IRON(III) COMPLEXES WITH PENTADENTATE SCHIFF BASE LIGANDS: SYNTHESIS, SPECTRAL, THERMAL AND BIOLOGICAL CHARACTERIZATION

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Iron deficiency anaemia, is a global health problem, mostly in developing countries. The most efficient amelioration or treatment for this affection is the supplementation with iron compounds. Oral therapy with iron is usually proper for most of patients and represents an efficient, cheap and good tolerated method of treatment. The iron(III) species are preferred having in view the minimal side effects produced in comparison with iron(II) compounds.

Three new multidentate ligands of Schiff base type (L^1, L^2 and L^3) were synthesized by condensation between ethylenediamine-N,N-dipropionic acid and different aldehydes such as acetylacetone, benzoylacetone and salicylaldehyde. The direct reaction between these ligands and some iron(III) salts leaded to three new iron(III) complexes of type $FeL\cdot nH_2O$ (L: L^1, L^2 and L^3); the ligands and the iron complexes were characterized by elemental and thermal analysis, UV-Vis and IR spectroscopy.

The *in vitro* screening of the antimicrobial properties was performed against Gram-positive, Gram-negative and fungal strains by using both reference and clinical multidrug resistant ones. The ability of complexes to inhibit the colonization of an inert substratum was also determined.

The thermal behaviour of these derivatives was investigated in synthetic air by thermal analysis (TG, DTA) in order to evidence the modifications at heating and also the thermodynamics effects that accompany them. The thermal analysis confirmed the proposed formulas for complexes and provided information concerning their stability in comparison with that of the ligands. The complexes showed a similar thermal behaviour leading at iron(III) oxide as final product.

DIFFUSIVE CONTROLLED IONIC CROSSLINKING AS METHOD FOR OBTAINING NEW 3D HYDROGELS WITH CONTROLLED ARCHITECHTURE AND PROPERTIES

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Hydrogels based on natural and/or synthetic polymers have several properties that make possible their usage as scaffolds in tissue engineering. These properties are related to high water content which is similar to those of human tissues, their capacity to homogeneously encapsulate cells, the ability to effectively mass transfer and for their minimal invasive effect. For these reasons, hydrogels are ideal environment scaffolds, for cells proliferation and differentiation.

The handling of the physical properties in view of their adequacy to the tissue engineering requirements is a significant way to achieve hydrogels with desired properties. In this respects, the paper proposes a new method for natrium alginate ionically crosslinking which lead to hydrogels with controlled properties, suitable as scaffolds for adipose tissue regeneration. Two methods to control the migration of ions which crosslinked the natrium alginate, a physical one and chemical other, were imaged. **The chemical method** to control the calcium ions migration into the reaction medium was based by the use as calcium ions supplier of calcium gluconate, in situ obtained, after the reaction of $CaCO_3$ with gluconic acid formed following the mixing of glucono delta-lactone (DGL) with water. **The physical method** to control the calcium ions migration into the reaction medium consisted in use of calcium gluconate as a calcium ions provider, which, before reaching the alginate solution, diffused through a paper filter with controlled porosity. The paper reveals that the proposed method leads to non-toxic hydrogels, with large, interconnected pores, good dynamo-mechanical properties and “egg-box” type structures. These properties make the new hydrogels appropriate 3-D scaffolds for growth and proliferation of stem cells from human body.

Poster Presentations

ALTERNATIVE SYNTHESIS OF MAGNETIC SILICA THERAPEUTIC NANOSYSTEMS USING ULTRASONIC IRRADIATION

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Magnetic biomaterials, particularly magnetic silica nanosystems such as superparamagnetic Fe_3O_4 nanoparticles (SPIONs) coated with silica, in biomedical applications provide the ability to be directed and concentrated within the target location by means of an external magnetic field and to be removed when the therapy is completed. Moreover, controlled release of drug enclosed within the mesopores of silica is of interest when new drug delivery systems are involved. In this work we report a facile synthesis of MSNPs (Magnetic silica nanoparticles) enclosing a chemotherapeutic agent, by ultrasonic irradiation. The study focused on developing an alternative to the conventional synthesis method in order to obtain stable magnetic silica nanoparticles with magnetite core and ordered mesoporous silica (OMS) shell for applications as in targeted delivery and controlled release of 5-fluorouracil. Citrate-caped Fe_3O_4 NPs were prepared by a facile one-step sonochemical method. Then, freshly dried Fe_3O_4 NPs were coated with silica through the hydrolysis and condensation of tetraethylorthosilicate (TEOS) under ultrasonic irradiation to prevent the agglomeration of the magnetite NP and accelerate the hydrolysis and condensation of TEOS. By changing the TEOS hydrolysis conditions in the presence of Fe_3O_4 NPs and the sonication parameters, the thickness of the silica shell was controlled. This control leads to further manipulation of the composition, morphology and magnetic properties of the core-shell NPs. The as-prepared MSNPs were then used for immobilization of 5-FU under ultrasonic irradiation. XRD, N_2-sorption, TEM, VSM, FTIR and UV tests were used to characterize the magnetic silica therapeutic nanosystems.

It was found that sonochemical technique is an effective method to synthesize MSNPs as therapeutic agents, offering the advantage of short reaction time and good quality nanoparticles in similar conditions.

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ORDERED MESOPOROUS MCM41-TYPE MATRIX FOR AMIKACIN DELIVERY SYSTEMS

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Ordered mesoporous silica or aluminosilicate nanoparticles possess features like high surface area and pore volume, the possibility of surface functionalization, tunable pore size and acidity by varying aluminum content, that recommend them for the preparation of drug delivery systems. These are obtained by loading through adsorption of a biological active compound in the pores of the mesoporous carrier that leads to slow release rate of the drug depending on the interactions between the biological active compound and support, as well as the pore size.

Five ordered mesoporous MCM41-type materials, two silica and three aluminosilicates with different aluminum content were studied as supports for drug delivery systems. As biological active model molecule, amikacin, that belongs to the aminoglycoside antibiotic class and it is used in the treatment of different severe bacterial infections, was chosen for the obtaining of drug delivery systems. MCM-type supports were prepared by sol-gel method followed by a solvothermal treatment from tetraethylorthosilicate, aluminium sec-butoxide and cetyltrimethyl ammonium bromide or tetradecyl trimethyl ammonium bromide, respectively, as template agent. The MCM41-type materials were characterized by low-angle XRD, FTIR spectroscopy, SEM-EDX, as well as by N_2 adsorption-desoption isotherms. The loading of amikacin was performed in aqueous solutions and the drug uptake on the support was estimated by an indirect UV-vis spectroscopic method. The hybrid materials were characterized by FTIR spectroscopy, low-angle and wide-angle XRD. The drug release studies were performed in a phosphate buffer solution. Only up to 70% of the total drug content in the hybrids is released in 24 h emphasizing a strong interaction between amikacin and support.

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AN INVESTIGATION OF TIN THIN FILMS DEPOSITED ON FECOV AND TI SUBSTRATES BY MAGNETRON SPUTTERING TECHNIQUE

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In this paper, the research results regarding to the TiN thin layer deposition on a FeCoV/Ti solid diffusion bonded assembly in order to be used as a pivot in dental field, have been presented.

Applied as a thin layer, TiN is a non-toxic coating with intrinsic biocompatibility which is used to harden and protect ferromagnetic surfaces and, also to improve their wear resistance. With this aim, an investigation on titanium nitride (TiN) film deposited on the ferromagnetic (FeCoV) and biocompatible pure Ti substrates by DC-magnetron sputtering from a Ti metal target using a mixture of nitrogen (N2) and argon (Ar) gases has been offered.

These TiN layers have been deposited both at RT (Room Temperature) and 200°C with the flow rate ratio of the N2:Ar were 4:1 and the sputtered time fixed at 1200 seconds. After deposition, the morphology, crystalline orientation and composition of deposited TiN films were characterized by AFM, XRD, and FE-SEM respectively.

Also, the tribological (friction coefficient, wear rate) and mechanical characteristics (Vickers hardness, Young modulus and depth of penetration) on the hard developed TiN coatings have been investigated.

OPTICAL PROPERTIES FOR THE POLYMERS (PEG:PLGA) OBTAINED BY MATRIX ASSISTED PULSED LASER DEPOSITION (MAPLE)

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Thin films of biodegradable polymers (Polyethylene glycol (PEG) and Poly(lactide-co-glycolide) (PLGA)) offer promising perspectives for controlled drug delivery implants. In this work, thin layers of PEG:PLGA blends were obtained by matrix assisted pulsed laser deposition (MAPLE) on silicon substrates. The films were analyzed in terms of surface topography and optical constants. These properties were particularly discussed in correlation with the laser fluence used for MAPLE deposition of the films. The optical characterization was carried out using spectroscopic ellipsometry technique (SE). The measurements were performed in the range of wavelengths 350-700 nm, at different angles of incidence. In order to extract the optical parameters (refractive indices and extinction coefficients) an optical model was required. This model consists of 4 layers: substrate (silicon), native SiO_2, the blend of polymers and a rough top layer. The thickness of the thin polymer layers and the thickness of the rough layer were determined by Atomic Force Microscopy (AFM). The preliminary values of optical constants were obtained using the Cauchy dispersion in the range of 600-700 nm. The final values of the refractive indices and of the extinction coefficients were calculated using Gaussian oscillators, which were fully Kramers-Kronig consistent. In all, the findings indicate that spectroscopic ellipsometry is a suitable technique for determining the changes of the optical properties as a function of the deposition parameters i.e. laser fluence for thin films of biocompatible polymers deposited by MAPLE.

COMBINATORIAL PULSED LASER DEPOSITION OF CALCIUM PHOSPHATES

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Compositional libraries of calcium phosphates (CaPs) were fabricated by combinatorial pulsed laser deposition (CPLD) technique on glass, fused silica and silicon substrates at room temperature. In view of obtaining different combinations for calcium phosphate mixtures, we employed in our studies CPLD technique. The aim of this study was to evaluate the solubility in SBF for a specific CaPs mixture. The composite coatings obtained by CLPD were characterized by different morphological and physico-chemical properties along one axis, necessary to tabulate solubility libraries corresponding to each ratio of calcium phosphate mixture. These libraries are of great importance in finding the optimal quality of coatings with a precise solubility related to the composition of calcium phosphate mixture. The combinatorial films were deposited by simultaneous ablation of two targets with different composition (hydroxyapatite (HA) + beta-tricalcium phosphate (beta-TCP) or (HA) + octocalcium phosphate (OCP)). Morphological and topological profiles were determined by microscopy techniques (AFM, SEM and SNOM). Mapping and profile of composition were obtained by energy dispersive spectroscopy (EDS). In vitro test showed a distribution of cells as a function of composition. The combinatorial PLD experiments conducted in our study allowed us to emphasize in a comprehensive manner, the potential of this approach to evaluate the properties of the CaPs mixture by deposition of only few libraries.

CHEMICAL MODIFICATION OF MULTIWALL CARBON NANOTUBES WITH DIAZONIUM SALTS

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Modifying the surface chemistry of carbon materials has been a goal of chemists for many decades, especially carbon nanotubes (CNTs). Modification of carbon nanotubes is essential for their use in many proposed applications such as drug delivery systems, biosensor devices, composite materials and electrochemical energy devices.

The oxygen-containing functional groups were the widely recognized and the most common species formed on the surface of carbons, which significantly influenced their performance for practical applications. Several chemical and physical modifications of anode surfaces have successfully been used to increase microbial fuel cell (MFC) power densities.

The purpose of this research was to investigate the effect of diazonium salts on carbon nanotube before and after being modified by Raman and electron spectroscopy (XPS).

To modify the CNT surface, we treated the multiwall carbon nanotubes with diazonium salts (4-nitroaniline and 4-Nitrobenzenediazonium tetrafluoroborate). Carbon based material were modified with diazonium salt in order to increase nitrogen-containing functional groups. The addition of carbon radicals from diazonium salts to CNTs has proved particularly successful in the covalent chemical modification of CNTs. Introduction of nitrogen-containing functional groups at the anode surface could be useful method to improve microbial adhesion and power generation of MFC.

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OBTAINING THE METAL SUPPORT OF DENTAL BRIDGES BY ELECTRO-GALVANIC DEPOSITION

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To obtain prosthetic dental works with high mechanical strength, with increased precision of execution, with high biocompatibility and physiognomic aspect, were implemented in dentistry the modern technologies used in engineering.

This paper proposes a new methodology for obtaining the resistance support of the dental bridges by using electro-galvanic deposition of the metals used in dental technique, namely gold and chrome, combined with innovative technologies of Reverse Engineering and Rapid Prototyping.

To obtain the virtual model of the prosthetics field is used the 3D scanning directly in the oral cavity or scanning the plaster model obtained by casting in an impression of the prosthetic field. After scanning is obtained the point cloud of the prosthetic field. Using specialized software the point cloud is processed in order to obtain the 3D solid model of the prosthetic field. In the next stage takes place the virtual modeling of the resistance structure of dental bridge fixed on the abutments of the prosthetic field. This modeling will take into account, from dimensional point of view, the technology used to obtain the metal support, namely electro-galvanic deposition of metal.

In order to deposit the electro-galvanic metal, first, is made the support on the Rapid Prototyping machine. By a controlled electro-galvanic deposition, the support will be covered with a metal layer of 0.25 - 0.3 mm. Depending on the type of work, the base metal can be gold or chrome. On the new metal structure can be deposited physiognomic materials such as composite or porcelain. Using the proposed method allows obtaining of prosthetic works with high precision, with very small thicknesses of the walls but with high mechanical resistance. It is, also, obtained a very good adaptability on the prosthetic field, due to the lack of deformations that occur when materials are processed at high temperatures or sintered, and the cost price is much lower.

NEW $CaCO_3$ / POLY(2-ACRYLAMIDO-2-METHYLPROPANESULFONIC ACID–CO-ACRYLIC ACID) POLYMORPHS, AS TEMPLATES FOR CORE/SHELL PARTICLES

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The special mineralization process in the biological system has attracted many scientists to biomimetically synthesize inorganic/organic materials. An important method of biomimetic synthesis is to use a soft organic template and to control the morphogenesis of inorganic materials with complex forms. Therefore, the crystallization of $CaCO_3$ particles from aqueous solutions in the absence and presence a strong/weak anionic copolymer, poly(2-acrylamido-2-methylpropanesulfonic acid–co-acrylic acid) (PAMPSAA) is followed in this study. The influence of polymer chemical structure and concentration on the nucleation and growth of calcium carbonate has been deeply investigated, showing a strong effect on the morphology of the formed composite materials. Vaterite/calcite polymorphs with different sizes and spherical shapes were obtained. The new materials were characterized by infrared spectroscopy, X-ray powder diffraction, X-ray photoelectron spectroscopy, flow particle image analysis, laser diffraction technology, and SEM and optical microscopy. The encapsulation of porous $CaCO_3$ microparticles can be realized by nanoengineered layer-by-layer self-assembly, which is a self-assembly technique where multiple nano-sized layers are built up on the template using oppositely charged polyelectrolytes. Thus, oppositely charged polyions were assembled onto new prepared carbonate colloidal particles in a layer-by-layer manner to yield core-shell particles.

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INFLUENCE OF THE DIFFUSION MEDIUM ON THE MIGRATION OF THE CROSS-LINKERS AND ON THE PROPERTIES OF THE HYDROGELS OBTAINED THROUGH DIFFUSIVE CONTROL

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In order to obtain alginate based hydrogels through ionic cross linking of guluronic blocks from alginic acid with divalent cations by controlling the reaction rate, two different methods for controlling the migration of crosslinking ions were used. In this way hydrogels with large pores with excellent properties for growth and proliferation of stem cells from human body were realized.

To find out the influence of the diffusion medium on the hydrogel properties, 3-D scaffolds were realized under the same conditions but with different diffusion medium (filter paper) which differs by the pores size. For all hydrogels thus obtained, the following characterizations were performed: induction period (DMA), the time stability of visco - elastic properties (DMA), hydrogel morphology (SEM), cell growth and proliferation. It was observed that the three different diffusion mediums different marks the properties of the resulted hydrogels. In all cases, were obtained hydrogels with large pores of about 200 to 500 μ. The diffusion mediums with small pores lead to hydrogels with homogenous "egg - box" structure. In case of the diffusion medium with large pores the resulted hydrogels are strongly inhomogeneous considering the pore sizes. The cell growth and proliferation in such hydrogels are strongly dependent by the structural homogeneity of the supports, the most convenient support being those obtained with "blue code" paper filter as diffusion medium.

ELEMENTAL ANALYSIS OF BIOCOMPATIBLE METALLIC MATERIALS TYPE 316L STAINLESS STEEL

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Among the best used biomaterials for implants execution are stainless steel 316L.

A biomaterial as the type AISI 316 L steel , has to meet very stringent tests (established by rules and stas-s), before it can be applied in medical implants. One important test to impose a type of metallic material as a biomaterial is to fulfill the criterion on certified chemical composition and homogeneity. The most adequate technique to determine the elemental composition of metallic biomaterials is optical emission spectrometry technique (SEOASE), due to very high correspondence between performance and costs.

Consequently, optimization of mechanical and physical properties of austenitic stainless steel type 316 L for osteosynthesis, requires careful balancing of chemical composition.

Thus, this paper presents the elemental analysis of steel samples of AISI 316 L with SEOASE method as well as, estimates of uncertainty associated the results of this method. The purpose of these experimental research is to highlight the need to estimate the quality of experimental results under the new European requirements and practices specified in the standards EN 17025 and EN 13005.

S4

Thin Films of Multifunctional Materials for Advanced Applications

Invited Papers

GENERAL BEHAVIOUR OF Ti 3D BANDS AT THE N-DOPED $SrTiO_3/LaAlO_3$ INTERFACE AND THEIR 2-DIMENSIONAL LOCALIZATION

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The formation of a 2 dimensional electron gas (2DEG) at the interface between $SrTiO_3$ and $LaAlO_3$ (STO/LAO) is one the most outstanding scientific achievements of the last few years. While the role of the $LaAlO_3$ polarity in injecting charge on STO by a Zener Breakdown mechanism has been widely debated, less attention has been paid to the equally essential ability of STO conduction bands to create rapidly decaying surface states, where the excess charge is eventually confined. In this paper we want to stress the importance of the chemical behaviour of 3d bands for what concerns transport and thermoelectric properties of the 2DEG, and the perspective it opens for alternative STO based hetero-structures for building the 2DEG.

We will first illustrate our theoretical results for the electronic transport and thermoelectric power of bulk and 2D conduction bands of STO, and then discuss the possibility of having 2DEG formation in systems without polar discontinuity. In particular we will compare band structure calculations on STO/LAO junctions with heterostructures based on $SrTiO_3/SrZrO_3$ (STO/SZO) junctions. In this case the conduction band discontinuity is given by the large conduction band offset, and an excess of electron carriers has to be introduced in the system by doping impurities. We will illustrate in detail the behaviour of La and Nb impurities at varying concentration and thickness of the SZO epi-layer.

All the presented computational results have been obtained by a recently formulated beyond-local-density First-Principles approach, i.e. the variational Pseudo Self interaction correction; this method was showed able to compute realistic band gaps in a variety of magnetic and non-magnetic complex oxides; this virtue is of the outmost importance here, since the accurate description of band alignment is an essential ingredient for the 2DEG mechanism. For transport and thermopower calculations we used the Bloch-Boltzmann approach, with energy-dependent modelling of the relaxation time approximation.

TWO-DIMENSIONAL GROWTH AND PROPERTIES OF MAGNETOELECTRIC $BaTiO_3$-$CoFe_2O_4$ EPITAXIAL BILAYERS ON $SrTiO_3$(001) AND Si(001)

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Multiferroic epitaxial layered heterostructures combining ferroelectric (FE) and ferromagnetic (FM) phases can permit magnetoelectric response via interfacial charge field effect or elastic coupling at the interfaces. Fabrication of heterostructures combining a FE perovskite like $BaTiO_3$ (BTO) and a FM spinel oxide like $CoFe_2O_4$ (CFO) is extremely challenging because they are highly dissimilar in terms of lattice parameters and surface energy. Here we use kinetic growth limitations to obtain by pulsed laser deposition atomically-flat (001)-oriented bilayers (either CFO/BTO or BTO/CFO) on $SrTiO_3$(001) with $La_{2/3}Sr_{1/3}MnO_3$ or $SrRuO_3$ bottom electrodes. Reflection high-energy electron diffraction (RHEED) is used for in-situ monitoring of intensity oscillations and lattice strain relaxation, which is complemented by ex-situ X-ray diffraction measurements. The remnant FE polarization in CFO/BTO, depending on BTO thickness, ranges from bulk value (~26 $\mu C/cm^2$) to above 50 $\mu C/cm^2$, and in BTO/CFO can be higher than bulk value. We observe a large variation of dielectric permittivity up to 40 % under application of magnetic field, occurring at the ferroelectric phase transitions. This temperature sensibility and the insulating nature of CFO strongly signal that the observed magnetoelectric coupling is induced by elastic coupling.

Finally, CFO/BTO bilayers are integrated with Si(001). Since BTO on Si(001) tends to orient its polar axis in-plane due to the thermal mismatch between both materials, a $LaNiO_3/CeO_2$/YSZ buffer layer structure causing high epitaxial compressive stress was used to favour c-axis orientation of BTO. Carefully controlled growth conditions permit two-dimensional epitaxial growth of BTO, and X-ray diffraction measurements reveals that the polar axis is out-of-plane. Measurement of ferroelectric loops in CFO/BTO bilayers showed a high remnant polarization above 20 $\mu C/cm^2$.

FUNCTIONAL PROPERTIES OF CHARGED DOMAINS WALLS IN LA-BIFEO$_3$ THIN FILMS

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Ferroelectric domain walls are inherently nanoscale 2D structures that separate domains of aligned polarization and have recently generated a huge amount of attention due to their potential functional properties; conduction at domain walls was recently demonstrated[1] and additionally they play a crucial role in the above-bandgap photovoltaic effect[2]. Furthermore an emerging aspect of study is the appearance of enhanced properties at charged domain walls[3,4] (entities in which components of the spontaneous polarization lie in a head-to-head or tail-to-tail arrangement thus resulting in charge accumulation in the wall vicinity). However, charged domain wall are generally energetically unfavourable and as such do not spontaneously form under normal circumstances. A method to reliably engineer charged domain walls on the nanoscale is therefore lacking.

Current work involves attempts to address this issue by precisely controlling domain wall types and orientations in thin films of the room-temperature multiferroic 10%La-BiFeO$_3$ by means of scanning probe microscopy. The rhombohedral {100}-orientated films (in the pseudocubic notation) possess 8 possible <111> polar directions which can be switched through application of a sufficiently large electric field. Thus through appropriate biasing of a scanning probe, each polar direction can be selectively chosen with nanoscale precision thereby allowing for creation of both neutral and charged domain walls with any desired topology. Piezoresponse force microscopy and conductive atomic force microscopy can subsequently be used for observation and characterization of functional properties.

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FULL FIELD ELECTRON SPECTROMICROSCOPY OF FERROELECTRICS

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The analysis of chemical and electronic states in complex and nanostructured material systems requires electron spectroscopy to be carried out with high lateral resolution. Here we present two examples of electron spectromicroscopy [1] applied to the study of ferroelectric domain surfaces.

Standard electrical characterization of thin films is made difficult due to the high leakage current in the tunnel regime. The electrostatic surface potential and the work function of FE domains in an ultra-thin BiFeO$_3$ were studied using low energy electron microscopy (LEEM) and energy filtered photoelectron emission microscopy (PEEM), [2] providing insights into the ferroelectric polarization as a function of nanometric film thickness.

The second example presents an energy filtered PEEM study of the band structure of a micron scale region in a strained BaTiO$_3$ epitaxial thin film. Using suitable electron optical configuration a spatially resolved reciprocal space image of the electron dispersion relations in the first Brillouin zones is obtained [3] and compared with first principles calculations, confirming the surface termination layer of BaTiO$_3$. These examples demonstrate that the combination of real and reciprocal space imaging of ferroelectric surfaces using full field electron spectromicroscopy is a powerful new tool for unraveling the electronic structure underpinning ferroelectric order and screening.

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NEW FUNCTIONAL PROPERTIES DRIVEN BY NANOSCALE INTERFACE REACTIONS IN MAGNETOELECTRIC COMPOSITES

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Nanoscale interface reactions in combinations of di-similar materials can drive to interesting functional properties. A better control of these reactions and large ratio of active interfaces can be obtained in core-shell powders, for which an appropriate sintering strategy generates desired microstructures with various degrees of connectivity in composite ceramics. Magnetoelectric Mag@$BaTiO_3$ (Mag=α-Fe_2O_3 [1], Ni,$ZnFe_2O_4$ [2]) core-shell nanocomposites were densified by classical or by spark plasma sintering. By various sintering strategies, dense and homogeneous ceramics were produced as result of the interface reactions between constituents: (ii) di-phase compositions with fully isolated magnetic regions within a $BaTiO_3$ matrix, and (ii) multi-phase compositions. Besides the parent materials, variable amounts of secondary phases (Fe_3O_4, $BaFe_{12}O_{19}$ and $Ba_{12}Fe_{28}Ti_{15}O_{84}$ [3]) were induced and they gave rise to new funtional properties of the ceramic composites. Dielectric constant of 100-300 and low losses by comparison with $BaTiO_3$-based magnetoelectric composites produced by other methods and reduced hoping conductivity contribution due to the isolation of the low-resistivity magnetic phase were produced. Dielectric tunability with multipolar contributions to the dielectric non-linearity was reported and peculiar magnetic properties, including “wasp-waisted” constricted M(H) loops were determined as result of the formation of magnetic phases with contrasting magnetic coercivities (hard and soft phases). The present results demonstrate the usefulness of the core-shell approach and the role of sintering strategy in driving new functional properties in multifunctional composites, by an appropriate control of the in situ solid-state nanoscale interface reactions.

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TUNNELING ACROSS A FERROELECTRIC BARRIER: A FIRST-PRINCIPLES STUDY

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Ferroelectric tunnel junctions (FTJ), including a ferroelectric as the barrier material, have recently attracted a lot of interest since they are potentially interesting for technological applications such as data storage. In such system, the barrier has to be sufficiently thin to allow for tunneling but must at the same time remain ferroelectric, a property that tends to be suppressed in ultrathin films. The first-principles modeling of FTJ remains challenging mainly because typical density functional calculations within the LDA and GGA significantly underestimate the band gap of insulators and so produce erroneous Schottky barriers. In a first part, we consider a prototypical model system made of a ferroelectric oxide film between two gold electrodes and compute the I-V curve of the system using density functional theory and Non-Equilibrium Green's Function (NEGF+DFT) formalism. Our atomic-scale approach allows for the first time accurate and self-consistent description of the screening at the metal ferroelectric interface and of the atomic relaxation. It predicts that the asymmetry in the I-V curve for up and down polarizations is sufficiently large to be detected experimentally at finite bias even for symmetric FTJ, therefore opening the door to applications in which the tunneling current allows the reading of the polarization state [1]. In a second part, we introduce a new hybrid approach for the correct description of Schottky barrier at metal/ferroelectric interfaces and discuss trends for typical ferroelectrics and metals. Work supported by the European project OxIDes and the EMMI.

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THIN FILMS OF HYBRID ORGANIC-INORGANIC NANOSTRUCTURED MATERIALS – A COMBINED COMPUTATIONAL AND EXPERIMENTAL APPROACH

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The recent discovery of spin injection in hybrid organic-inorganic spin valves based on films of V[TCNE]$_x$ (x~2, TCNE = tetracyanoethylene), has brought back to the front line the first room-temperature molecule-based magnet. We present the results of a theoretical study correlating the structure and the special magnetic properties of the M[TCNE]$_2$ (M = V, Mn, Fe, Ni, Co) family of materials, with focus on the V[TCNE]$_2$ system. Starting from the available experimental data on M[TCNE]$_2$ systems we perform Density Functional Theory (DFT) calculations with periodic boundary conditions to optimize the geometry. The DFT calculations indicate antiparallel spin alignment resulting in ferrimagnetic ordering, but heavily overestimate the value of the exchange coupling. Better estimates of the exchange coupling parameters, J, in the expected range of -175 to -150 K, are obtained by means of *ab initio* multiconfiguration calculations, performed on smaller molecular models cut from the optimized crystal lattice. Further spin-orbit and ligand-field calculations allowed the modeling of the environment of the V(II) ions yielding a $10Dq$ ~18400 cm^{-1} parameter, a uniform magnetic anisotropy of the bulk, D = -0.03 K, and a random anisotropy of D_r = 0.45 K.

In the second part of the talk we report results of combined experimental and theoretical studies of metal-free dyes used as sensitizers for nanocrystalline TiO$_2$ dye-sensitized solar cells. Based on DFT calculations we discuss, for each dye, the adsorption onto the substrate, the matching of the absorption spectrum of the dye with the solar spectrum, the energy level alignment with the semiconductor and the electrolyte, and the charge transfer to the substrate. Examples of dyes used include 5-(4-sulfophenylazo)salicylic acid disodium salt, four azodyes derived from different heterocyclic systems, and various natural pigments based on betalains, studied also experimentally in standard AM1.5 conditions.

EPITAXIAL DESIGN OF NOVEL PEROVSKITE-TYPE FERROELECTRICS

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Epitaxial heterostructures of complex metal oxides represent one of the hottest topics in modern nanoelectronics and condensed matter physics. The main foreseen area of related devices includes morphic computational systems. Epitaxial films of perovskite-type ferroelectrics (FEs), whose polarization and/or resistivity can be modified electrically in a non-volatile manner, may be relevant for such systems. However, much broader field of applications of FE films is connected with the diversification of electronic devices and systems, where epitaxial single-crystal films may have superior performance compared to nanocrystalline films (e.g. higher permittivity) and may bring novel functions (e.g. strong magnetoelectric effect).

Compared to bulk samples or thick layers, nanoscale effects of misfit strain and interfacial phenomena can result in novel phases and properties in epitaxial thin-film FEs. The concept of strain-induced changes formulated 15 years ago [N. A. Pertsev, A. G. Zembilgotov, and A. K. Tagantsev, Phys. Rev. Lett. 80 (1998) 1988] is now well established in theoretical studies. Interfacial phenomena primarily considered at the film-electrode boundary in models of FE capacitors are now expanded to include unusual charge, polar, and magnetic ordering. Development of FE heterostructures with novel properties by controlling these nanoscale effects captures imagination. Some of our experimental achievements in this direction as well as unexpected observations will be briefly overviewed.

FERROMAGNETIC SURFACE ALLOYS SYNTHESIZED BY MOLECULAR BEAM EPITAXY AND CHARACTERIZED BY INNER-SHELL SPECTROSCOPIES

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Surface magnetic properties may differ considerably from bulk. In this Contribution I will stress on the occurence of unusual magnetic properties for films with thicknesses in the monolayer regime. The contribution will first state on the synthesis of ferromagnetic systems from materials which are not ferromagnetic in the bulk state: Mn [1], Cr [2], V [3]. For each metal, a different strategy was employed, but all techniques employ molecular beam epitaxy. The magnetic properties are measured by X-ray magnetic circular dichroism (XMCD). Ni monolayers deposited on Cu(001) exhibit magnetic properties with a 2D character of spin waves [4]. Another class which will be discussed is represented by magnetic metals deposited on semiconductors. Co deposited on GaAs(011) may stabilize the *bcc* structure, as seen by X-ray photoelectron spectroscopy (XPS) and X-ray photoelectron diffraction (XPD) [5]. A comparison between Fe deposited on GaAs(001) and on InAs(001) exhibits in the latter case a lower reactivity (as seen by XPS) and enhanced magnetic properties (XMCD) [6,7]. Ref. [7] presents also an explanation of the huge enhancement (300 %) of the magnetic orbital moment for ultrathin layers. Other examples which will be discussed are represented by Fe/Si(001) [8], Fe/Sm(001) and Mn/Ge(001), characterized by XPS, X-ray absorption fine structure (XAFS), surface magneto-optical Kerr effect (SMOKE), and Auger electron spectroscopy (AES).

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TOWARD INTEGRATION OF EPITAXIAL PIEZOELECTRIC THIN FILMS ON SILICON SUBSTRATE FOR MEMS APPLICATIONS

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There is an increasing worldwide interest, particularly from industrials, in piezoelectric thin films like $Pb(Zr,Ti)O_3$ (PZT) for MEMS applications, such as sensors and actuators. PZT thin films grown onto silicon substrates typically show polycrystalline structures, which results in degraded properties compare to bulk single crystal material. Since epitaxial films usually exhibit performances superior to polycrystalline counterpart, it is of great interest to integrate epitaxial piezoelectric thin films onto silicon wafers for MEMS devices. Although epitaxial growth of PZT films onto single crystal substrate has been widely studied, it remains a major technological challenge when the substrate is silicon. In the case of PZT, the only reliable route so far to obtain single crystal thin films on silicon is utilizing an isotype perovskite structure oxide buffer layer $SrTiO_3$ (STO) grown by Molecular Beam Epitaxy (MBE). The possibility to grow epitaxial $SrTiO_3$ (STO) on Si substrate then opens the way to integration of complex perovkiste-type oxide onto silicon substrate. We successfully deposited epitaxial $Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT) thin film on STO-buffered Si(001) substrate by both sol-gel and pulsed laser deposition (PLD) methods. $SrRuO_3$ film could also be epitaxially grown by PLD on top of the STO layer in order to act as back electrode for capacitors.

ENERGY BAND ALIGNMENT OF FERROELECTRIC MATERIALS

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Ferroelectric materials have energy gaps around 3 eV and can therefore be considered as wide gap semiconductors. The energy band alignment of semiconductors describes how the valence band and conduction band energies of two materials are aligned when the two materials are brought into direct contact. In the case of semiconductor/metal contacts, the discontinuities are known as Schottky barriers. These determine the rate of charge injection at the contact, which can become a determining factor of thin film devices.

We have determined energy band alignment at a large set of interfaces including ferroelectric materials using photoelectron spectroscopy (XPS). The results give insights into the fundamental properties which govern energy band alignment of such materials and how the band alignment relates to electrical properties of the materials. We further show that the barrier height at oxide/metal interfaces is modified by chemical oxidation and reduction and, in the case of ferroelectric materials, also by polarization.

MULTILAYERED MAGNETOSTRICTIVE THIN FILMS AND NANOWIRES PREPARED BY ELECTRODEPOSITION

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Organic and inorganic materials artificially created can be used as active components in implants or other prosthetic devices. Among them, the magnetostrictive nanowires can mimic the cilia which can be found in organs of the human body, and have unique electrical and magnetic properties which can be exploited for the use in implants or medical imaging. The magnetoelastic properties of Fe-Ga alloys are very sensitive to the microstructure, the solubility of Ga in bcc α-Fe and the fabrication process [1], these effects being more prominent for Fe-Ga electrodeposited alloys [2]. This work focuses on our new results concerning the preparation and characterization of electrochemically deposited [$Fe_{1-x}Ga_x/Fe_{1-y}Ni_y$] x n (x = 0.1÷0.3 at. %; y = 0.4÷0.8 at. %) and [$Fe_{1-x}Ga_x/Co_{60}Fe_{20}B_{20}$] x n (x = 0.1÷0.3) multilayered films and nanowires arrays. Multilayered films 3-4 µm thick have been obtained by electrodepositing 50 consecutive sequences of amorphous Co-Fe-B or crystalline permalloy (Ni-Fe) (20 nm, 50 nm or 60 nm) and Fe-Ga (60, 50 nm or 20 nm) successive layers. 200 to 300 sequences multilayered nanowires were electrodeposited into commercially available nanoporous alumina membranes with pore diameters ranging from 35 to 250 nm, and lengths of 50-60 µm. The layers have been deposited successively by changing the electrodeposition potential in the range from 0.75 to 1.9 V. The composition and thickness of the different layers were tailored by optimizing the deposition parameters, such as the buffer additives and applied voltage. The combination of $Fe_{1-x}Ga_x$ magnetostrictive material and crystalline $Fe_{1-y}Ni_y$ or $Co_{60}Fe_{20}B_{20}$ amorphous soft magnetic material shows good magnetic softness (H_c does not exceed 60 Gs for permalloy and 20 Gs for the amorphous alloy) and gives novel magnetostrictive behavior caused by the formation of twisted spin structures. All these aspects will be discussed in detail, considering also the magnetic and structural coupling between layers and the surface/interface phenomena.

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FERROELECTRIC THIN FILMS DEPOSITED BY RADIOFREQUENCY ASSISTED PULSED LASER DEPOSITION

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Results on the deposition and characterization of $Sr_xBa_{1-x}Nb_2O_6$ (SBN) , (1-x) $Na_{0.5}Bi_{0.5}TiO_3$ – x$BaTiO_3$ (NBT-BT) and La doped $Pb(Zr_xTi_1.x)O_3$ – PLZT are presented. The influence of the deposition parameters (laser wavelength and fluence, substrate type and temperature) and of the beam consisting in excited and ionised species produced by radiofrequency discharge both during deposition and cooling are investigated in connection with layers texture, ferroelectric and electro-optic properties.

Oral Presentations

INVESTIGATION OF MOS INTERFACE OF 4H-SiC C-FACE EPITAXIAL LAYER

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4H-SiC carbon(C)-Face silicon carbide (SiC) epitaxial layer had been grown by using a low-pressure, hot-wall chemical vapor deposition system. The FTIR spectra (reflectance mode) show that the intensity and shape of the epitaxial layer are much better than these of the substrate. XRD spectra show 4H-SiC (004) plane at 2θ angle of about 35.6°. The peak is very high and narrow. Results of FTIR and XRD indicate the good crystal structure of the epitaxial layer. The doping concentration is about $1.5x10^{16}$ cm^{-3} and the root-mean-square (RMS) surface roughness is 0.3 nm.

Subsequently, MOS-Capacitors were fabricated. The soft breakdown starts at around 4.5 MV/cm and oxide breakdown occurs at around 6.5 MV/cm when the wet oxidation was carried out at 1150°C for 90 min, followed by re-oxidation at 950°C for 2 hours and immediately annealed with NO at 1175°C for 2.5 hours. The oxide thickness is about 5000 Å. As the oxidation temperature decreases to 1000°C and 950°C, the soft breakdown starts at around 3.5 MV/cm and oxide breakdown occurs at around 8 MV/cm in the oxide thickness range between 750 Å and 400 Å. However, the lowest interface trap density was obtained from the wet oxidation at the temperature of 950°C. The oxide breakdown fields for Al- and N-implanted samples with Ar and graphite-cap post-implant anneals were also investigated. RMS surface roughness significantly reduces to 0.6 nm with a graphite-cap anneal. The doped samples were oxidized at 950°C in the wet oxygen ambient without re-oxidation and followed by NO anneal at 1175°C. The soft breakdown and oxide breakdown of N-implanted samples are 4.5 MV/cm and 8 MV/cm, respectively; the oxide breakdown of Al-implanted samples is only 3 MV/cm, no matter whether a graphite-cap or Ar anneal was used.

STRUCTURAL AND MAGNETIC PROPERTIES OF TRANSITION METAL IONS DOPED ZnO THIN FILMS

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For new applications it is a great interest the investigation of diluted magnetic semiconductor (DMS) because the semiconductor properties can be integrated with magnetic properties to realize the transparent spin-based devices. Ferromagnetism in transition metal (TM)-doped Zn O is theoretically predicted by Sato and Katayama-Yoshioda using ab initio calculations based on local density approximation (LDA). The ferromagnetism of TM doped Zn O thin films is considered through a double exchange mechanism, without requiring additional carrier incorporation. The preparation conditions and levels of doping are crucial for the achievement of this goal.

In this work we investigated the structural, magnetic and conduction properties of Ni-doped Zn O ($Zn_{1-x}Ni_xO$, x = 0.03÷0.15) and Fe-doped Zn O ($Zn_{1-x}Fe_xO$, x = 0.03÷0.15) thin films, prepared by sol-gel method. X –Ray Diffraction (XRD) and Atomic Force Microscopy (AFM) were used to characterize their morphology and structures of TM-doped Zn O thin films. The magnetic studies of DMS were done using vibrating sample magnetometer (VSM) at room temperature. To make a correlation between the electrical properties and structure of (TM)-doped Zn O thin films were used the method of four contacts disposed in line for electrical characterization. The measurement system consists in a current source Keithley 6221, a nanovoltmeter Keithley 2182A and Jandel Universal Probe equipment. The influence of dopant ion (Ni^{2+}, Fe^{3+}) concentration on the structure and magnetic properties of semiconductor oxide $Zn_{1-x}TM_x O$ (x = 0.03 ÷ 0.15) thin films revealed that substitution of TM ions for Zn^{2+} in the Zn NO wurtzite lattice take place for low content, x=0.03 ÷ 0.05 (Fe^{3+}) and x=0.03 ÷ 0.10 (Ni^{2+}) and that Zn O thin films doped with the transition metal have the room temperature ferromagnetism.

$LiMo_2$ (M=Co, Ni, Mn) THIN FILM CATHODE MATERIALS: ELECTRONIC PROPERTIES AND BATTERY PERFORMANCE

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$Li(Ni,Co,Mn)O_2$ layered oxide is a perspective high voltage cathode material for Li-ion rechargeable batteries. The thin film cathode materials can be considered as a prototype for the 3D battery structures with the potential interest for auto-industry as an alternative energy storage source.

$Li(Ni,Co,Mn)O_2$ thin films (200 - 300 nm thickness) are grown by RF magnetron sputtering at room-temperature in the O/Ar gas mixture. Post-deposition annealing of the films in oxygen atmosphere leads to crystallization of the cathode material. Higher crystallinity is achieved at high annealing temperatures resulting in the improved battery performance. Crystallographic structure of the films is characterized by X-ray diffraction (XRD). Performance of the batteries is studied by electrochemical cycling of the $Li(Mn,Ni,Co)O_2$ with $LiPF_6$/EC/DMC liquid electrolyte and Li-foil as anode material. Dependence of stoichiometry and surface electronic properties of the grown films on the growth parameters such as deposition temperature, O/Ar gas ratio etc. is investigated by X-ray photoelectron spectroscopy (XPS). The valence state of the transition metals in the crystallized thin film cathode materials is Ni^{2+}, Co^{3+} and Mn^{4+}. Annealing of the films at high temperature, $T_{ann} \geq 700°$ C, leads to lithium deficiency which is linked to the lattice oxygen loss in the Li_2O form previously also observed for the $Li_x(Ni,Co)O_2$ thin film material [1].

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EVIDENCE FOR TUNNELING FROM INTERFACE STATES IN NON-NITRIDATED n-4H-SiC/SiO$_2$ CAPACITORS

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Non-nitridated n-4H-SiC/SiO$_2$ capacitors exhibit anomalous discontinuities in the capacitance-versus-voltage (C-V) characteristics at temperatures below 150 K compared to capacitors with a nitridated SiC/SiO$_2$ interface. Two abrupt minima appear in the C-V curves always at the same specific gate voltages. In order to understand the origin of these minima, we have developed a model invoking charge transfer between the n-type SiC layer and some oxide states near the interface via a tunneling process, described by the semi-classical Wentzel-Kramers-Brillouin approximation. The presence and the magnitude of these minima can be explained by considering some neutral donor states at the SiC/oxide interface enabling electron tunneling into distinct energy levels in the oxide. Numerical simulations based on our model give close agreement with the anomalous C-V characteristics observed experimentally. The proposed model is shown to account for the appearance, position and amplitude of the minima by considering two defect levels in the oxide layer close to the interface. Both defect levels were experimentally detected via Thermally Dielectric Relaxation Current measurements. Thus, the model implies that under given conditions, i.e., the existence of a sufficient density of neutral donors at the SiC/oxide interface and empty electron states in the oxide layer, abrupt minima are in general to be expected during C-V measurements of n-4H-SiC/oxide capacitors. Indeed, similar discontinuities in the C-V characteristics but, at higher temperatures, are also observed when SiO$_2$ is replaced with Al$_2$O$_2$.

FUNDAMENTALS ON SYNTHESIS AND CHARACTERIZATION OF CARBON NANOPOWDER FILLED POLYMER COMPOSITES

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In this work we report the synthesis of free-standing films and sandwich layers of polymer/nanocarbon composites trough an original approach called the inverse stamping method, which consists in forming a thin film of carbon nanoparticles onto the water's surface.

The almost spherical carbon encapsulated iron nanoparticles with narrow size distribution were prepared via laser co-pyrolysis method in which the CW CO$_2$ laser beam irradiates a gas mixture containing iron pentacarbonyl (vapors) and ethylene/acetylene hydrocarbons. Specific flow geometries were used in order to synthesize iron particle first followed by stimulate hydrocarbon decomposition at iron surface. High-resolution transmission electron microscopy images reveal the core-shell feature of synthesized nanostructures with around 2 nm thick carbon layers and 3-7 nm diameters iron-based core dimensions. The mean diameter could be experimentally controlled. It was found a decreasing trend of particle size with the decreasing of pressure and total reactant gas flow.

Polymer matrix consists of the **Syntalat A 077** type acrylic polymer (SpolChemie): hydroxyl content 2.6%; density 1.02 g/ml; OH groups cross linked with an aliphatic isocyanate. The polymer was chosen for it's high adherences on to the substrate. The isocyanate was used to hurry the reticulation process.

Trough the free infiltration of the polymer between the nanoparticles which form the film, after the polymer is cured, we obtain free-standing films of polymer/nanocarbon composites. The TEM, SAED, SEM, HRTEM procedures has been used in order to characterization such carbon based nanostructures.

The incorporation of conductive particles into a polymer matrix modifies fundamentally the electrical conductivity of the composite. A sudden change of several orders of magnitude often undergoes in the electric conductivity of polymer composite, when the mass fraction of the included material passes a critical threshold. This phenomenon, known as percolation threshold, is related to the existence of conducting clusters. A major problem in the production of polymer nanocomposites is the filler content, that must be as low as possible, otherwise the mixture processing becomes difficult, the mechanical properties of the composites are poor, and the final cost is high. High-grade conductive carbon black is indeed expensive. The only way to reduce the carbon black content in polymer composite materials is to favour inhomogeneities in the material.

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DIELECTRIC FUNCTION MODEL USED IN OPTICAL CHARACTERIZATION OF “HIGH-K” CERAMIC THIN FILMS; APPLICATIONS TO Ni:YSZ thin films growth by PLD

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Transparent high-k ceramic thin films grown by PLD have been studied in connections with applications like MOSFET, sensors, thermal barriers and ionic devices. Cauchy and Cauchy – Urbach functions were mainly used in optical simulation for spectroelipsometric data interpretation due to transparency on a large domain of electromagnetic spectrum of a big part of such materials. Some limitations in using GenOsc and EMA are generated by performance of soft-ware package WVASE32. Applications for YSZ and Ni:YSZ thin films are presented.

Keywords: high – k ceramic thin films; PLD; WVASE32; Cauchy model; Cauchy – Urbach model;Ni: YSZ thin films.

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THIN FILM TANDEM SEMICONDUCTOR SYSTEMS FOR DYES VIS-PHOTODEGRADATION

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Multi-materials with enhanced photocatalytic properties, based on associates of semiconductors are designed considering the band gap value and position. Photocatalyst thin layers were obtained by spray pyrolysis deposition (SPD) using ethanol solutions of inorganic precursors to obtain: (a) SnO_2 ($SnCl_4$, 99.8%, Alfa Aesar); (b) TiO_2 ($TiCl_4$, 99.9%, Alfa Aesar); (c) WO_3 (WCl_6, 99.8%, Alfa Aesar); (d) Cu_xS ($CuCl_2 \cdot 2H_2O$, 99%, Scharlau Chemie SA). The samples were deposited in a wafer structure, consisting of two mono-component layers (the thin film surface was 4 cm^2), on microscopic glass (Heinz Herenz) and FTO (F doped SnO_2 coated glass – Libbey Owens Ford TEC 20/2.5 nm). After deposition, each layer was cooled down, at room temperature, followed by annealing in air.

The composition, crystallinity and surface properties of the tandem layers were correlated with the photocatalytic activity of the samples in the degradation of a 0.0125 mM methylene blue (MB) solution, under VIS irradiation, measuring the absorption spectra for each sample, after 6h. Hydrogen peroxide (4 mL H_2O_2 30%/L of dye solution) was used as electron acceptor to improve the photocatalytic activity of the semiconductors. Stability and reproducibility were also investigated: after the first series of photocatalysis experiments, the films were washed in bi-distilled water, dried and again subjected to photocatalysis with a fresh solution of 0.0125 mM MB; this procedure was twice followed.

In the experimental conditions, the tandem photocatalysts WO_3/TiO_2, SnO_2/TiO_2 and Cu_xS/TiO_2 have photodegradation efficiencies of 35%, 40% and 97%, respectively.

The photodegradation mechanism of MB under visible light irradiation and Cu_xS – based photocatalysts is detailed discussed; it involves a series of reactions that include, in systems containing H_2O_2, the enhanced production of $HO^{\bullet}$ radicals due to the electrons from the photoexcited Cu_xS; supplementary the copper cations can form additional radicals $HO_2^{\bullet}$ and $HO^{\bullet}$.

MAGNETOSTRICTION EFFECT IN $La_{0.7}Sr_{0.3}MnO_3$ THIN FILMS ON $SrTiO_3$

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Manganites are a unique class of materials with an extremely strong coupling between three fundamental degrees of freedom: electronic, spin and lattice order [1]. Of particular interest is $La_{0.7}Sr_{0.3}MnO_3$ (LSMO), showing both a Curie temperature of 360 K and an almost full spin polarization [2]. Epitaxial thin films can be subjected to elastic strain due to structural mismatch with respect to substrates. $SrTiO_3$ (STO) substrates may induce a biaxial strain on the growing film due to the tetragonal distortion occurring below the antiferrodistortive phase transition (T_S) at 105 K.

In this work LSMO thin films were grown on oriented STO (001) substrates by pulsed laser deposition. X-rays diffraction, atomic force microscopy and scanning electron microscopy were used to analyse film orientation and texture. Comparison of temperature dependence of the magnetizations obtained under magnetic fields parallel and perpendicular to the substrate, confirms the large anisotropy of LSMO films grown onto STO substrates. Below T_S, the magnetization starts to deviate from the expected high temperature Brillouin behaviour. The relation between the excess of magnetization relative the one obtained from Brillouin fitting and the square of the order parameter of the STO phase transition is linear. This result is in favour for the existence of magnetrostriction coupling between LSMO film and STO (001) substrate. This result is also confirmed from magnetoresistance and resistivity measurements, which also show an anomaly at T_S.

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COMPARISON BETWEEN ELECTRICAL PROPERTIES OF $Pb(Zr,Ti)O_3$ AND $BaTiO_3$ CAPACITORS WITH BOTTOM $SrRuO_3$ CONTACT AND DIFFERENT METALS AS TOP ELECTRODE

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Here we present the electric properties of metal-ferroelectric-metal (MFM) structures based on 300 nm epitaxial $Pb(Zr,Ti)O_3$ (PZT) or $BaTiO_3$(BTO) films grown on a common $SrRuO_3$ (SRO) bottom contact and having different metals as top electrode (SRO, Pt, Cu, Al, Au). The PZT, BTO and SRO layers were deposited by PLD method on single crystal $SrTiO_3$ substrates with (001) orientation. The electric properties were investigated by performing the following measurements: hysteresis, capacitance-voltage (C-V), current-voltage (I-V). These measurements were performed at different temperatures, between 150 K and 400 K. The dielectric constant determined from the capacitance measurements has different values for different metals, suggesting a significant influence of the interface with the top electrode. The height of the potential barrier was estimated from the leakage current measurements using the Schottky – Simmons equation. It was found that the potential barrier is about the same for top and bottom interfaces, and is almost independent on the top metal except Al. The results suggest a strong effect of the polarization charges on the electronic properties of the interfaces in MFM structures.

STRUCTURAL, ELECTRIC AND MAGNETIC PROPERTIES OF $Pb(Zr_{0.2}Ti_{0.8})O_3$ – $CoFe_2O_4$ HETEROSTRUCTURES

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We have investigated symmetric and non-symmetric $Pb(Zr_{0.2}\ Ti_{0.8})O3$ – $CoFe_2O_4$ (PZT/CFO) heterostructures grown on SrTiO3 (100) substrates by using pulsed laser deposition (PLD). PZT /CFO layers were sequentially deposited on the STO substrate with a bottom electrode of $SrRuO_3$ (20 nm). X-ray diffraction (XRD), and transmission electron microscopy (TEM) investigations reveal crystalline structure and the epitaxial relationship between PZT and CFO. The electric properties of symmetric and non-symmetric heterostructures were characterized by using P-V loop, C-f, and C-V measurements. The remnant polarization and the coercive field are significantly affected by the CFO layer. The M-H loop measurements shows that the non-symmetric PZT/CFO heterostructure possess a weak ferrimagnetic response even at room temperature.

MOLECULAR BEAM EPITAXY GROWTH AND X-RAY PHOTOELECTRON SPECTROSCOPY ANALYSIS OF Au/PZT HETEROSTRUCTURES

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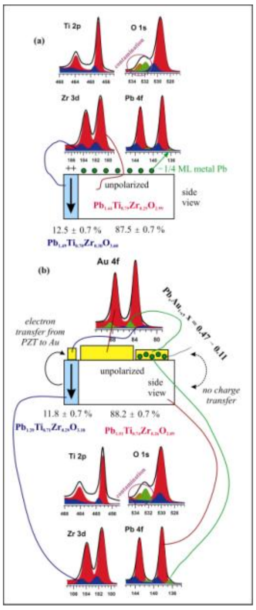
Growth of gold layers on $Pb(Zr_{0.2},Ti_{0.8})O_3$ (PZT) deposited on $SrTiO_3$ is *in situ* investigated by X-ray photoelectron spectroscopy in the thickness range 2-100 Å. Two phases are identified, with compositions close to nominal PZT resulted from the target's composition. The 'standard' phase is represented by all binding energies (Pb 4f, Ti 2p, Zr 3d, O 1s) sensibly equal to the nominal values for PZT (as derived from the NIST X-ray photoelectron spectroscopy database), whereas the 'charged' phase exhibits all core levels are shifted by ~ 1 eV towards higher binding energies. Au deposition proceeds in the formation of an ohmic contact with a band bending of $\Phi_{PZT} - \Phi_{Au}$ ~ 0.4-0.5 eV for both phases, identified as similar shifts towards higher binding energies of all Pb, Ti, Zr, O core levels with Au deposition. The Au contact on the 'charged' phase reflects in a shift of Au 4f core level by about 1 eV towards lower binding energies with respect to 'nominal' Au 4f binding energy position (metal Au). This implies electron transfer from PZT to Au, driven by the polarization field. The free PZT phase is occupied by ~ one quarter monolayer of Pb; this Pb forms a surface alloy with Au, producing a Au 4f component shifted by ~ 0.7 eV towards higher binding energies with respect to metal Au.



Poster Presentations

EFFECT OF NICKEL DOPING ON STRUCTURAL, MORPHOLOGICAL AND MAGNETICALPROPERTIES OF SOL-GEL SPIN COATED ZnO THIN FILMS

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In recent years, the researches and developments of ZnO films produced by doping with transition metals (TM) have attracted intense interest due to their properties of dilute magnetic semiconductor (DMS). Dilute magnetic semiconductors (DMS_S) are potential candidates for technological applications in optoelectronics, magnetoelectronic and microwave devices.

In this paper, $Zn_{1-x}Ni_xO$ (x = 0.03 - 0.15) thin films were synthesized by sol-gel method and deposited by spin coating onto Si/SiO_2 substrate.

The thin films have been characterized structurally by X-ray measurements and FTIR spectra, optically by electronic spectra measured in UV-Vis, morphologically by SEM and AFM microscopy and magnetically by Vibrating Sample Magnetometry (VSM) method.

X-ray diffraction (XRD) structural characterization shows that all samples have single phase with the ZnO – like wurtzite structure. The FTIR spectra confirm the formation of tetrahedral coordination of the oxygen ions surrounding the zinc ions and a shift in the frequency of bands determined by the substitution of Zn^{2+} ions in ZnO lattice with Fe^{3+} or Ni^{2+} ions.

Atomic force microscopy (AFM) was used to study the crystallinity and growth mode of the films. The films showed an improvement in crystallinity and preferential c-axis orientation. The thickness of films deposited on Si/SiO_2 substrate was 35 nm.

Magnetic measurements indicated that all samples are ferromagnetic at room temperature. The origin of the ferromagnetism in $Zn_{1-x}Ni_xO$ was mainly due to Ni ions substituted into the ZnO lattice.

Keywords: thin films, sol-gel method, ferromagnetism, diluted magnetic semiconductor.

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ELECTRICAL AND OPTICAL BEHAVIOUR OF DC SPUTTERED TANTALUM OXYNITRIDE THIN SOLID FILMS

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The main purpose of this work is to present and to interpret the electrical and optical behaviour of TaN_xO_y thin films as a function of the deposition parameters and structure. The tantalum oxynitride films were produced by DC reactive magnetron sputtering. The reactive gas mixture (85% N_2 and 15% O_2 the proportion was kept constant for all depositions) was increased from 5% gradually up to 60%, while keeping the argon flow constant for all depositions. A temperature of 100°C was applied to the substrate.

The electrical resistivity was determined using the four point probe method for the low resistivity samples. For the high resistivity samples contacts for the determination of the I-V characteristic were deposited on the thin film surface. Optical measurements for reflectivity and transmittance were made in the wavelength region of 250 to 800 nm, and for colour determination in the 400 to 700nm wavelength region, respectively.

The results were correlated to the crystallographic structure exhibited by the samples. An influence of the chemical composition and crystallographic structure on the electrical resistivity and optical properties was noticed.

$YBa_2Cu_3O_{7-x}$ THIN FILMS OBTAINED FROM A FLUORINE – FREE AQUEOUS PROPIONATE PRECURSOR SOLUTION

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$YBa_2Cu_3O_{7-x}$ (YBCO) films have gained considerable interest due to their intrinsic properties. These films can be obtained both trough physical and chemical methods. The most common chemical method used is TFA – MOD CSD, but the main disadvantage of this method is the release of hydrofluoric acid (HF). Due to this major drawback, a large effort has been made towards obtaining YBCO films from fluorine – free precursor solutions. Using this type of solutions the elimination of hydrofluoric acid during the thermal decomposition is avoided. YBCO films have been obtained starting from a fluorine – free aqueous propionate precursor solution. The precursor solution was prepared by dissolving metal acetates solutions in an excess of propionic acid. Further, the solution was concentrated by distillation under sever conditions. The as-prepared precursor solution was spin-coated on (100) $SrTiO_3$ single crystalline substrates. The precursor films were subjected to a single – step thermal treatment comprising both pyrolysis and crystallization. In order to determine the thermal decomposition behaviour of the precursor solution during the pyrolysis, the precursor powder, previously obtained by drying the precursor solution, was submitted to thermal analysis (DTA – TG). The final YBCO films were structurally and morphologically characterized by X-ray diffraction and atomic force microscopy, respectively. The electrical characterization of these films was made by transport measurements.

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PHASE DEVELOPMENT DURING ANNEALING IN NdFeB-TYPE THIN FILMS

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The formation and the evolution of the main phases, starting from the amorphous state to the crystalline one was studied through a combined set of experiments performed on the thin films DC magnetron-sputtered. In order to reproduce the well-known technological conditions for develop the nanostructured materials based on Nd-Fe-B alloys, the XRD dynamic analysis were performed, starting from room temperature to 760°C, with an 20°C increment. The formation of the desired microstructure was confirmed also by the SEM and EDX analysis, performed before and after XRD analysis.

PROPERTIES OF $Ba_{1-x}Sr_xTiO_3$ THIN FILMS OBTAINED BY PULSED LASER DEPOSITION

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Barium strontium titanate (**$Ba_{1-x}Sr_xTiO_3$**) thin films were obtained by Pulsed Laser Deposition (PLD) techniques on Pt/Si and MgO substrates. Control of the BST (**$Ba_xSr_{1-x}TiO_3$**) thin film epitaxial growth and microstructure quality are important factor for the electronic devices applications. A study on the influence of strontium content (x=50 and 75) on the properties of the thin BST thin layer was carried out. Films texture and surface topography of BST layers were studied by X-ray diffraction and atomic force microscopy (AFM); the element distribution along the layer was identified with secondary ion mass spectroscopy (SIMS). Optical properties in the 300 − 1700 nm range of wavelengths have been investigated using spectroscopic ellipsometry (SE). In a first step, the thicknesses of the films and of their rough layer are extracted from Cauchy model. Then, the final values of refractive indices and extinction coefficients were calculated using a Gauss oscillator model, which is fully Kramers-Kronig consistent. The thicknesses of rough layer were found to be in agreement with atomic force microscopy results.

Keywords: SE, spectroscopic ellipsometry, PLD, AFM, XRD, SIMS, BST, barium strontium titanate

ELECTROPLATED IN MAGNETIC FIELD OF ALLOYS CoNiMnP ONTO COPPER SUBSTRATE

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The electrochemical deposition technique is especially interesting due to its low cost, high throughput and high quality of obtained deposit. In the present study, preliminary experiments of electrodeposition in magnetic field of CoNiMnP alloys onto Cu substrate were performed. Also, the CoNiMnP magnetic film characteristics were studied. Copper substrate of 99% purity was in shape of sheets. The electrolyte bath consisted in 26g/L $CoCl_2.6H_2O$, 24g/L $NiCl_2.6H_2O$, 3.6g/L $MnSO_4$, 24g/L H_3BO_3, 4.6g/L NaH_2PO_2, 23g/L NaCl, 0.8g/L saccharin, 0.2g/L sodium lauryl sulphate and 0.01g/L cerium(III) sulphate. The operating conditions for deposition in magnetic field were the followings: pH 3.5-5, current density 1.5-9.5mA/cm^2, voltage 2-5V, room temperature, magnetic stirring, 1-15 h electrolysis time. Both Ni and Co plates with large area were used as soluble anodes. Deposits of CoNiMnP alloy having about 50 μm thickness were obtained working with 5mA/cm^2 current density during 10h. The CoNiMnP alloy layer showed good appearance and was characterized by uniformity, fine-grained and brightness. The structure, texture, crystallite size and Vickers microhardness were evidenced and measured by SEM and XRD investigations and microhardness tests, respectively. The magnetic measurements on the CoNiMnP alloy films were performed through magnetometry.

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RADIOFREQUENCY ASSISTED PULSED LASER DEPOSITION (RF-PLD) OF In_2O_3 THIN FILMS

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Indium oxide is used in some types of batteries, thin film infrared reflectors transparent for visible light, some optical coatings, and some antistatic coatings. They can be used as diffusion barriers ("barrier metals") in semiconductors, e.g. to inhibit diffusion between aluminium and silicon.

In this paper is presented an experimental work for obtaining thin films of In_2O_3 deposited by Pulsed Laser Deposition (PLD) assisted by radiofrequency on glass substrates in a different condition. The deposition parameters as number of pulses, laser wavelength, substrate temperature, radiofrequency power were varied. Techniques as Atomic Force Microscopy, X-ray Diffraction, Secondary Ion Mass Spectroscopy, Scanning Electron Microscopy and Spectroscopic Ellipsometry (SE) have been use to characterize the deposited thin layers.

Keywords: SE, spectroscopic ellipsometry, RF-PLD, AFM, XRD, SEM, SIMS, Indium oxide

THE OPTICAL NONLINEARITY OF SILVER OXIDE NANOPARTICLES THIN FILMS PREPARED BY Dc-MAGNETRON SPUTTERING METHOD

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Metal nanoparticles possess unique linear and large nonlinear optical properties. They exhibit wide applications in many fields, such as labelling of biological molecules, biosensors and nonlinear optical devices. In this work, we presented our studies of nonlinear optical refractive index of AgO_x thin films were prepared by using a cylindrical direct current reactive magnetron sputtering system at 10^{-5} torr initial pressure on BK7 glass substrate. The samples at different thickness were investigated by x-ray diffraction, scanning electron microscopy (SEM), linear and nonlinear optical absorption. The linear optical absorption data were measured in the UV-visible-near infrared spectral regions and the surface Plasmon resonance (SPR) were determined from these data .The nonlinear refractive index(n_2) of thin films are evaluated by the moiré deflectometery technique. A Nd:YAG^{+2} laser beam (wavelength of 532 nm) was focused by lens L1 and is re-collimated by lens L2. Two similar Ranchi gratings G_1 and G_2 with a pitch of 0.1 mm were used to construct the Moiré fringe patterns. The distance between planes of G_1 and G_2 is important, which it must be one of the Talbot distances of the used gratings. The Talbot distances are given by $z(t)= tp^2/\lambda$,where p is the periodicity of the grating; λ is the wavelength of light, and t is an integer. Therefore, the Moiré fringes were observed on a screen that was attached to the second grating. The Moiré fringe patterns were projected onto a computerized CCD camera by lens L3, placed at the back of the second grating. Moiré deflectometery is a sensitive method for measuring changes in the refractive indices of material. We performed as the thickness increased the nonlinear refractive index of AgO_x thin films increased.

ANALYSIS OF TRANSPARENT OXIDE THIN FILMS FOR $CuIn_xGa_{1-x}Se$ SOLAR CELLS

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In this paper we present recent results, regarding growth and characterization of some oxide thin films (e.g. ZnO:Al, SnO2, ITO) deposited by rf magnetron sputtering and vacuum thermal evaporation techniques. These oxide thin films have conductive properties and can be individually used as transparent contact electrodes to create CuInxGa1-xSe (CIGS) type solar cells.

The experimental results following the investigations performed on the three compounds ZnO:Al, ITO and SnO2, are showed. Thicknesses of 300 nm for ZnO:Al, 250 nm for SnO2 and 230 nm for ITO, were determined using the interferential microscope (i.e. White Light Interferometer from Ambios). From atomic force microscopy (AFM) measurements we found that roughness values (R_{rms} = 7 nm for ZnO:Al, R_{rms} = 37 nm for SnO2 and R_{rms} = 53 nm for ITO) of the studied oxide thin films (i.e. ZnO: Al, SnO2 and ITO) are on the order of nanometers and tens of nanometers.

Based on X-ray diffraction investigations, oxide thin films show strong orientation after the main planes perpendicular to the substrate, e.g. (002) and (022) for SnO2 film, (002) for ZnO:Al film and (222) for ITO film. Information about the crystalline structure and surface topography of the samples was obtained using scanning electron microscopy (SEM).

It is highlighted that ITO is a good conductive and transparent material (90 - 94%), ZnO:Al has the advantage of an improvement of these properties depending on the used doping with Al (3%), and SnO2 shows very small values of the electrical resistance on the order of Ohms.

CHARACTERIZATION OF RF SPUTTERED $MTaO_3$ (M=Li, Na, K) THIN FILMS

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Among the family of transition metal oxides, the $MTaO_3$ (M=Li, Na, K) compounds have attracted a great interest from researchers from a fundamental and technological point of view. $LiTaO_3$ is isomorph to $LiNbO_3$ and both exhibit ferroelectricity at room temperature. Owing to the polar characteristics, these materials can be used in transducers, sensors, and optical applications. Contrarily, $NaTaO_3$ and $KTaO_3$, though possessing polar soft modes, do not exhibit ferroelectricity at finite temperatures. All these materials possess a highly polarizable lattice, yielding their properties very sensitive to defects, impurities, elastic, and applied electric fields. These factors can easily induce ferroelectricity and intermediate glass- or relaxor-like states. Though a relative high number of works has been published in bulk $MTaO_3$ systems, their study in thin film form is still scarce and their behaviour is yet not fully understood.

In this work $MTaO_3$ thin films were prepared by radio frequency (rf) magnetron sputtering by using 2 inch diameter ceramic targets of $LiTaO_3$+Li_2O, $NaTaO_3$+Na_2O, and $KTaO_3$+K_2O with an average target-to substrate distance of 5 cm. Afterward, the films were deposited on platinized substrates by changing a specific parameter. The experimental parameters handled in this work were the argon pressure in the chamber, the rf power and the deposition time.

We have found out that some of the experimental conditions such as the argon pressure and the rf power have a large effect on the composition of the films. Both X-ray diffraction and Raman spectroscopy were used for determination of the phase in the as-grown films. Only for some experimental conditions, secondary phases of $M_XTa_YO_Z$ were found. Polarization reversal was also studied by using a modified Sawyer-Tower set-up in order to sort out the existence of ferroelectric ground state in the $MTaO_3$ as-grown films at room temperature.

THICKNESS DEPENDENCE OF STRUCTURAL, ELECTRICAL AND OPTICAL PROPERTIES OF PLD GROWN SrRuO3 THIN FILMS ON MgO

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Heteroepitaxial strain in ferroelectric thin films is known to have a significant impact on both their low and high frequency dielectric properties[1]. $SrRuO_3$ (SRO) has become the most popular epitaxial electrode for complex oxide heterostructures [2] owing to its favorable fatigue characteristics, chemical and thermal stabilities and also its nearly isotropic conductivity.

Conductive oxide thin films can be engineered through the deposition conditions [3]. The three major influences on their structural, electrical and optical properties are strain, stoichiometry, and thickness.

SRO films of varying thickness have been deposited on MgO (001) substrates by pulsed-laser-deposition (PLD) using a KrF excimer laser (LAMBDA Physik), λ=248nm, $\nu_{repetition}$ =8Hz, at a fluence of 0.8J cm^{-2} in an oxygen pressure of 40mTorr. Four samples of uniform thickness (10 to 75nm) have been produced. The thickness was measured with an INNOVA DEKTAK surface profilometer. Their structure and orientation have been checked by XRD. AFM (INNOVA Dimension 2000) in the ac mode was employed to investigate the surface morphology showing rather smooth surfaces for all samples. Morphology control is critical in achieving sharp interfaces in device structures. Spectroscopic ellipsometry in the UV-VIS and IR ranges (UVISEL Ex-Situ, Horiba Scientific) was used to study the variation of the optical constants of the films with thickness. Electrical conductivity and electron mobility were investigated using the van der Pauw method.

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S5

Advanced Ceramics: From Micro- to Nanoscale

Invited Papers

MILLIMETER WAVE ELECTROMAGNETIC CHARACTERIZATION BY MEANS OF WHISPERING GALLERY MODE TECHNIQUE: FROM DIELECTRIC PROPERTIES TO MICROSTRUCTURE INFORMATION

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The application of the whispering gallery mode (WGM) technique to the dielectric characterization of low-loss materials is described. The technique consists in making an overmoded dielectric resonator in form of a disc composed by the material of interest. The dielectric properties of the material can be determined studying the electromagnetic spectrum of the device. In this contribution, it will be shown how very high measurements accuracies can be reached also at millimeter and submillimeter wavelengths, on the basis of a simple procedure for WG modes excitation, analysis, and identification. The application of the WGM technique to ceramics shows relevant peculiarities, related to the characteristic disordered structure of these materials, usually composed by different small crystalline phases separated by irregular regions. The relatively dense spectrum of resonance modes typical of a WGM resonator allows a detailed analysis of the frequency behaviour of the dielectric properties. It will be shown that such property can be decisive in the identification of the transition between regimes of dielectric losses dominated by intrinsic factors (the materials forming the crystallites) to regimes dominated by extrinsic factors (the 'defects'). This information is in turn important to assess the possible improvements in the preparation of the ceramic samples. Moreover, the WGM technique is extremely sensitive to the irregularities of the resonant structure, owing to the probing electromagnetic modes, which exist in couples having degenerate resonance frequency in ideal configurations based on reciprocal materials. In real cases, both the irregularity of the geometry and the inhomogeneity of the material induce a splitting in the ideally degenerate modes. It will be shown that this modal splitting can give indications on the size of the defects with resolution orders of magnitude beyond the electromagnetic wavelength, being the contribution of the geometric irregularities typically negligible. Finally, it will be shown that the influence of the modal splitting on the quality factor determination can be minimized by studying the WGM resonance spectrum at various temperatures.

TERAHERTZ TIME-DOMAIN SPECTROSCOPY (THZ-TDS) APPLIED TO THE EVALUATION OF PHONON-POLARITON DISPERSION RELATION IN FERROELECTRIC CRYSTALS

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In ferroelectric materials, the dielectric properties in the terahertz (THz) region are of great importance due to the physical and chemical properties of ferroelectricity dominantly originated in this region. Recently the generation of coherent terahertz radiation through the femtosecond pulse laser irradiation on a photoconductive antenna has enabled the very promising new technique of the terahertz time-domain spectroscopy (THz-TDS), of which spectrometric technique has been in progress with advantages over conventional spectrometric techniques (Raman, FTIR).

The THz-TDS technique has better signal-to-noise ratio than the FTIR in room temperature detection, in which the time-domain signals (TDS) make it possible to measure directly not only the spectral transmission intensities $T(\omega)$ but also the intrinsic phase shifts $\Delta\varphi(\omega)$ of propagating THz radiations within a sample specimen. The intrinsic phase shifts $\Delta\varphi(\omega)$ enables us to make experimental estimation of the complex dielectric constant $\varepsilon^{*}(\omega)\{\varepsilon'(\omega), \varepsilon''(\omega)\}$ free from the uncertainty caused by Kramers-Kronig analysis, and also of the dispersion relations for various excitations coupled with the propagating THz radiations.

A versatile compact THz-TDS instrument developed newly with the advantages of wide wavenumber coverage and high dynamic range is now being applied for spectrum measurements on dielectric functional materials and for characterization of the dielectric properties on the phonon-polariton dispersion of ferroelectrics, the photonic band gaps of photonic crystals, crystalline morphology of organic compounds and pharmaceutical products, and boson peaks of grassy and amorphous materials.

In this paper, an overview of the instrument techniques, the spectrometric performances, and the THz-TDS measurement results will be described in focus on the typical perovskite families of current industrial interest, which includes bismuth titanate $Bi_4Ti_3O_{12}$ (BIT), lithium heptagermanate $Li_2Ge_7O_{15}$ (LGO), and lithium niobate $LiNbO_3$ (LN). On these ferroelectrics crystals, the complex dielectric constants are estimated. The nonliner dispersion relations determined from the phase shifts are consistently interpreted by taking account of phonon-polariton propagation in the crystals, that is consistently reproduced by the calculated phonon-polariton dispersion relations based on Kurosawa's formula.

FROM CONVENTIONAL TO ABERATION CORRECTED TEM IN MATERIALS SCIENCE

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Since 1931, when the first magnified images have been obtained by using electrons, electron microscopy has become one of the most powerful microstructural investigation techniques, through its spatial resolution, variety of electron beam manipulation techniques (imaging, several electron diffraction modes, electron energy filtering) and analytical capabilities. Nowadays, a complex transmission electron microscope can be operated in more than 15 different modes, being able to reveal structural and chemical details down to a space resolution well below 1 Angstrom.

Electron microscopy has been developed at the Institute of Atomic Physics in Magurele since 1953. Today, the National Institute of Materials Physics (NIMP) holds the most powerful high-resolution analytical transmission electron microscope (HRTEM) in South-Eastern Europe, with a resolution of 0.8 Angstrom in scanning transmission electron microscopy mode (STEM). The C_s-corrected microscope is also equipped with highly sensitive EDS (Energy Dispersive X-ray Spectroscopy) and EELS (Electron Energy Loss Spectroscopy) analytical units.

The current talk is aimed to present the TEM facilities at NIMP in terms of hardware, available working modes and problem solving capabilities based on the results obtained on various types of materials throughout the recent years. A special emphasis will be put onto currently running analytical microstructural investigations on epitaxial multiferroic thin films using the sub-Angstrom resolution electron microscope JEM ARM 200F installed at NIMP in 2011. The distribution of the atomic species in the analysed heterostructures is presented as a 2D chemical mapping obtained by STEM-EDS and Energy Filtered Transmission Electron Microscopy (EFTEM). The lattice strain, extended defects and local structure around interfaces are investigated by STEM, HRTEM and quantitative image processing using the Geometrical Phase Method.

EXISTENCE OF DILUTE MAGNETISM IN OXIDE PEROVSKITES?

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Recently, oxide semiconductors doped with transition metals have been investigated for two major reasons; either as a quest for new dilute magnetic oxides (DMO) or new multiferroic/magnetoelectric systems. Doping of the oxide semiconductors with transition metals has often been considered as a way to induce a long-range magnetic ordering in dielectric. The studies have been performed on simple oxides such as ZnO, Sn_2O, TiO_2, In_2O_3 as well as on more complex oxide compounds, $BaTiO_3$, $KTaO_3$, $SrTiO_3$ etc. The common characteristics of the studies on these materials are that they are mainly quite controversial. Very often they only have focused on a fine characterization of the physical properties of the magnetic response but have not reported on detailed microstructural and crystallographic analyses.

We showed that under proper conditions 3% of Mn can fully incorporate in $SrTiO_3$ predominantly on the A-site as Mn^{2+} or the B-site as Mn^{4+}. Such samples do not exhibit any evidence of long range magnetic interaction or spin-glass down to as low as 5K. The magnetic anomaly has only been observed for the samples, in which Mn is misplaced due to changes in the processing conditions and the valence state of Mn. Our findings conflict with claims, reported in several papers, that Mn-doped $SrTiO_3$ exhibits intrinsic spin coupling via frustrated antiferromagnetic superexchange, low temperature spin glass state and magnetoelectric coupling.

We have also shown that no intrinsic coupling of magnetic moments of dopant Mn ions occurs for thermodynamically equilibrated Mn-doped $KTaO_3$ even at cryogenic temperatures. We also found no evidence of the spin glass behaviour in our well-processed $(K_{1-2x}Mn_x)TaO_3$. If the magnetic anomaly is detected it is of an extrinsic nature from Mn clustering inside the $KTaO_3$ lattice or Mn-oxide precipitates. We also showed that incomplete processing can result in an antiferromagnetic response due to a presence of residual Mn_3O_4.

SHAPING OF NANOSTRUCTURED MATERIALS THROUGH SPARK PLASMA SINTERING

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Spark Plasma Sintering (SPS) is known to be very efficient for superfast full densification of nanopowders. This capacity is attributed to the simultaneous application of high density pulsed dc currents and load, even though the sintering mechanisms involved remain unclear. In the first part of the talk, the mechanisms involved during the SPS sintering of insulating oxide nanopowders (Al_2O_3, Y_2O_3 and stabilized Zirconia) or metallic particles will discussed.

In the second part of the talk we will illustrate how SPS can also be a useful tool to: (1) Consolidate mesoporous powders to obtain monolithic materials and to elaborate materials with controlled level of porosity. (2) Nearly fully densify core (BT or BST)/shell (SiO_2 or Al_2O_3) nanoparticles with limited or controlled reaction at the interface in order to modulate the dielectric properties of the ceramics obtained. (3) Sinter nanocomposites made of double walls carbon nanotubes dispersed in ceramic or metallic matrices and how the hardness, friction, wear, toughness and electrical properties of the nanocomposites obtained can be tuned.

DOPING EFFECTS IN WIDE GAP OXIDE FILMS

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Oxide-based films and nanostructures have emerged as important and promising materials for a wide range of applications such as photovoltaics, photocatalysis, optoelectronics, gas sensing and electronics. Many of these technologies require the use of transparent electrodes and charge collectors. At present, indium tin oxide (ITO) is the commonly large scale manufactured and used material. Many materials are under study to replace ITO with cheaper and more abundant materials. The doping effects of niobium-doped titanium oxide films were produced by RF co-sputtering of TiO_2 and Nb in argon, argon-H_2 and argon-O_2 plasmas, followed by vacuum thermal annealing.

In view of application in the photovoltaics field, new materials are also explored and developed to increase the conversion efficiency of solar cells through the increase of the most efficient and useful fraction of the solar spectrum which hits the cells, thanks to spectrum conversion processes such as frequency down-shifting. In that sense, the development of oxides doped with rare earths offers new opportunities to realize active materials for the efficient absorption and conversion of the solar radiation in solar cell. In this view, wide band gap oxide films doped with rare earth ions able to insure such processes were synthesized.

To develop an appropriate understanding of the properties of the doped oxide films, it is necessary to address the material preparation methods and also structural and defect issues. The effect of doping on the optical properties of the films was studied and a combined use of optical and x-ray photoemission spectroscopies provided information about the electronic properties of the films. Fundamental properties regarding the valence band, the Fermi level and the work function of the un-doped and doped films were studied.

MAGNETIC THIN COATINGS FABRICATED BY MAPLE FOR BIOLOGICAL APPLICATIONS

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Potential applications of core/shell nanoparticles raise from their ability to exhibit properties that neither individual materials could exhibit. This research is focused on functionalized Fe_3O_4 core/shell/adsorption-shell nanoparticles assembled thin coatings fabricated by matrix assisted pulsed laser evaporation (MAPLE) as vehicle for antibacterial drugs targeted delivery. Thin coating systems were characterized by FTIR, SEM, TEM, XRD, and investigated by *in vitro* biological assays, using Gram negative and Gram positive bacterial reference strains with known antibiotic susceptibility profiles as well as eukaryotic cell monolayers for cytotoxicity assays. Higher fluencies result in thin films degradation whereas ~200 mJ/cm^2 lower fluencies result in thin films that have a chemical structure similar to the starting material. SEM micrographs revealed a uniform morphology of nanocomposite nanoparticles while TEM investigations showed 2-20 nm Fe_3O_4 core nanoparticle diameters. The antibiotic charged nanoparticles influenced the viability as well as the microbial and eukaryotic cell adherence to the inert substrate. We concluded that magnetic nanoparticles could be used for the development of novel anti-microbial materials or strategies for fighting the microbial adherence and further development of the medical biofilms formed by different pathogenic agents on different substrata, frequently implicated in the aetiology of chronic and hard to treat infections. This lecture will discuss how careful consideration of the structure and composition of thin coatings can assist in the development of truly functional materials.

ELECTROCALORICS AS A SOLID STATE COOLER; EVALUATION FROM DIRECT AND INDIRECT MEASUREMENT OF FERROELECTRIC RELAXORS

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The need for more efficient and environmentally-friendly materials for refrigeration has lead to a significant effort towards solid state cooling such as electrocaloric cooling.

In polar crystals the spontaneous polarization increases with application of an external electric field. Under adiabatic conditions the entropy, S, stays constant where the system compensates the dipole alignment with an increase in temperature. The temperature change is reversible when the field is removed. This phenomenon is called the electrocaloric (EC) effect.

For the development of electrocaloric materials, a complete picture of the thermo-physical properties and its polar dynamics is necessary. Therefore it is necessary to characterize fully both thermal and electrical properties of each and every sample, as these properties vary between different material syntheses

Here, a direct EC measurement system based on a modified-Differential Scanning Calorimeter (DSC) is presented. The set-up allows the acquisition of thermal data (EC effect and heat capacity), leakage current and polar state (P-E loops) simultaneously for each sample. The results are discussed by comparing indirect EC evaluation, based on the change of polarization over temperature and the direct measurements from the estimated heat flux when a DC pulse is applied to the sample.

Different predictive theories will be explored that characterise the electocaloric when varying the temperature and applied electric DC field for ferroelectric relaxor systems.

Oral Presentations

MgB_2 WITH ADDITIONS OBTAINED BY SPARK PLASMA SINTERING

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MgB_2 is a candidate for different superconducting application owing to its relatively high critical temperature of about 39 K, availability and low price, low density, non-toxicity, and its unconventional physics. However, enhancement and control of the properties is highly desired and necessary to fulfill its potential.

MgB_2 bulk samples with relative density above 90% were prepared by ex-situ Spark Plasma Sintering technique. Pristine MgB_2 and added samples with Sb, Sb_2O_3, Bi, Bi_2O_3, Te, TeO_2, RE_2O_3 (RE=Eu, La, Ho), SiC or C_{60} were investigated from the viewpoint of consolidation and reactive processes, structural, morphological, superconducting and mechanical properties. Obtained materials take advantage of their final composite nature or of the C-substitution effects when using C-containing additions. We also show that for the same oxide additive, the raw state of the additive powder can induce very different properties exceeding or not the properties of the pristine reference MgB_2 sample. Finally we prepared samples with co-additions. These samples often exceed the critical current density J_c and/or the irreversibility field H_{irr} of the pristine and of the samples with one individual addition. Presented results are useful to develop a methodology to design, control and eventually maximize J_c and H_{irr} of MgB_2 based materials.

Apart from the superconducting properties we investigate biodegradable and other properties of MgB to establish its potential for medical applications.

FORMATION OF HYDROGENATED TiO_2 FILMS BY OXIDATION OF TI IN WATER VAPOR PLASMA

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Titanium dioxide (TiO_2) absorbs only ultraviolet light, and several approaches, including the use of dopants, have been taken to narrow the band gap of TiO_2. To enhance solar absorption we fabricated the hydrogenated nanocrystalline thin TiO_2 films on the basis of a conceptually different approach performing oxidation of Ti thin films in water vapor plasma at room temperature. It is shown, that plasma irradiation of wetted surface areas leads to the formation of hydroxyl radicals resulting from strong oxidation of Ti due to water supper-hydrophilic properties and split into their atomic components H and O. The mobile H atoms are trapped at radiation defects while the H atoms detrapped from trapping centers diffuse through the oxide layer into the bulk and, taking into account titanium's high affinity for hydrogen, are absorbed by the titanium, and consequently are stored in the bulk. Oxygen atoms diffuse through the oxide layer and form TiO_2. The atomic hydrogen concentration depends on the thickness of oxide layer. In thicker oxide layers, the atomic H atoms wander around the oxide layer until they either are trapped, or encounter another atomic H to recombine into H_2 and diffuse out of the oxide layer. The trapping process dominates in the implant zone.

SYNTHESIS OF CERIUM OXIDES MODIFIED WITH SAMARIUM FOR SOFC ANODES

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In this work we obtained four solid solutions based on the system $Ce_{1-x}Sm_xO_2$ (x = 0.0; 0.05; 0.10 and 0.15), with the purpose of develop materials for solid oxide fuel cell applications (SOFC). The characterization by infrared spectroscopy (FT-IR) and thermal analysis (TGA-DTA) of the precursors, revealed the formation of different kind of citrate species. The analysis of X-ray diffraction (XRD) and electron microscopy (SEM-TEM), allowed us determine the morphology, surface characteristics and texture of materials, results consistent with oxides of nanometric scale (< 20 nm), regularly oriented in the plane (111) with interplanar distances of 0.31 nm. The composition evaluated by microanalysis of energy dispersive X-ray (EDS) and X-ray fluorescence (XRF) indicated an excellent agreement between the proposal and obtained compositions, whereas the analysis of reducibility (TPR-H_2) and impedance spectroscopy (IS), demonstrated the feasibility in the use of these ceramics in partial oxidation reactions and showed a good response under operating conditions of a solid oxide fuel cell.

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MICROANALYSIS OF Al_2O_3-(Al-Fe) CERAMIC-METAL INTERPENETRATING PHASE COMPOSITES USING SCANNING AND TRANSMISSION ELECTRON MICROSCOPY TECHNIQUES

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Ceramic-metallic interpenetrating phase composites (IPC's) produced by reactive metal penetration (RMP) have unique characteristics due to the fine intermixing of two or more phases with individual properties that are distinctly different from one another. Typical applications envisioned for IPC's are high wear/corrosion resistant refractory materials for handling of molten materials, lightweight vehicle braking components, and high performance military body and vehicle armour. Preformed parts made of vitreous SiO_2 were immersed in molten Al-7.5wt.%Fe alloy for several hours. During transformation, vitreous SiO_2 reacts with the molten metal while the shape of the preform is preserved. Structural and chemical analysis of transformed material, using SEM/XEDS methods on metallographic and FIB-polished samples, demonstrates the coexistence of Al_2O_3 and Al interpenetrating phases, and indicates the presence of an Al-Fe phase. XRD investigation confirmed the presence of an Al-Fe phase but determination of the exact nature of the phase was difficult based on the X-ray diffraction pattern alone. Samples for S/TEM investigation were prepared using FIB methods. Through combining S/TEM imaging methods with XEDS and electron crystallography, two new metal phases were observed and investigated. Morphology, chemical composition and crystallographic structure of the micron-scale Al-Fe binary and nano-scale Al-Fe-Si ternary phases, will be reported.

THE STRUCTURAL DISORDERING AND SUPERIONIC PHASE TRANSITION IN THE $Li_{0.12}Na_{0.88}Ta_yNb_{1-y}O_3$ SOLID SOLUTION

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The structural disordering and phase transitions in the $Li_{0.12}Na_{0.88}Ta_yNb_{1-y}O_3$ solid solution were investigated by Raman spectra. Phase transition from ferroelectric to antiferroelectric when t=300÷350°C is clearly seen. In this case the line corresponding to the stretching modes of oxygen atoms disappears. Thermal disordering of the $Li_{0.12}Na_{0.88}Ta_yNb_{1-y}O_3$ solid solution structure introduced by mobile lithium ions happens gradually. It is facilitated by misorientation and considerable deformation of the oxygen octahedra with the changeof their symmetry. The static disordering of the structural units in the Nb^{5+} and Ta^{5+} sublattice makes the point of ferroelectric-antiferroelectric phase transition lower and can facilitate the transition to the superionic state. However, from the data obtained, it is clear that the phase transition to the superionic state doesn't appear in the Raman spectra.

HgI_2 NANOPARTICLES OBTAINED BY ELECTRON BEAM IRRADIATION

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Mercuric iodide is a vast studied semiconductor because of its properties for ionizing radiation detection. For imaging applications, a uniform and oriented film must be grown onto amorphous substrates. This growth is mainly guided by the first nucleation. In order to obtain adequate HgI_2 nuclei for nucleation, nanotechnology was employed.

HgI_2 particles were synthesized by the hydrothermal method from $Hg(NO_3)_2H_2O$ and NaI or KI as iodine sources. The dependence of the obtained product with iodine source and time of synthesis was studied. A synthesis time varying from 0 to 24 hours was studied, always at a furnace temperature of 120 ºC. The products obtained were then washed with distilled water and left to dry. The initial obtained clusters were characterized by X-ray diffraction (XRD) confirming alpha-HgI_2 phase identity by the Rietveld method. Clusters were also characterized by scanning electron microscopy (SEM) observing that no size uniformity was reached. In order to modify size and morphology of these clusters, they were subjected to the electron beam of a transmission electron microscope. When HgI_2 nanoparticles synthesized from NaI clusters were irradiated with a 100kV electron beam, we obtained plate nanoparticles 20-200 nm in size, and when clusters synthesized from KI were irradiated with the same electron beam we obtained nanoparticles 20-70 nm in size. For all samples, coalescence was observed after nanoparticle formation, giving nanoparticles of larger size. Crystallinity of mercuric iodide nanoparticles formed after irradiation was not yet confirmed. The synthesis method followed by electron beam irradiation promises accurate control of size and morphology of heavy metal iodide nanostructures, which is very propitious for improving HgI_2 film growth orientation and, thought it, the current applications of this material, as far as for the opening of new ones.

DIELECTRIC RESONATOR ANTENNAS WITH INCREASED DIRECTIVITY BY USING $(Zr_{0.8}Sn_{0.2})TiO_4$ CERAMIC

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$(Zr_{0.8}Sn_{0.2})TiO_4$ (ZST) cylindrical resonators with dielectric constant ε_r around 37, with values for $Q \times f$ product greater that 50,000 GHz at microwave frequencies have been prepared by solid state reaction. Dielectric resonator antennas (DRA) using ZST materials were designed by using Ansys High Frequency System Simulator. The feeding circuit consists in a microstrip line on a FR4 substrate. The $TE_{01\delta}$ mode of the antenna is controlled by using a using a circular metallic disk placed on the top surface of the ZST resonator. The antenna was tuned to 2.45 GHz central frequency. In addition, an antenna array was developed by using four DRA distanced by half a wavelength. The reflection loss and the antenna pattern were measured in an anechoic chamber with an Agilent PNA-X N5245A network analyzer. The measured response agrees well with the simulated data. The DRA array exhibits a significant increase in the directivity than the single resonator DRA. The developed antennas are attractive for ISM (Industrial, Scientific and Medical) band applications.

ELECTRON PARAMAGNETIC RESONANCE OF THE MAGNETIC DEFECTS IN NANOSTRUCTURED ZnO:Mn THIN FILMS

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The wide-band-gap (3.37 eV) transparent semiconductor ZnO is presently one of the most studied materials following the prediction of room temperature ferromagnetism in *p*-type Mn-doped ZnO [1], which was later confirmed by Sharma et al. [2]. Moreover, ZnO has been successfully used for many other applications in optics, electronics, solar cells, sensors, energy, medicine, etc. [Ref. 3 and references therein].

The uniform doping of thin films is very important for most applications. Electron paramagnetic resonance (EPR) spectroscopy is the method of choice for the identification and determination of the localization of paramagnetic impurities in insulators and semiconducting materials. With this method it is possible to distinguish substitutional, interstitial, surface and/or agglomerated magnetic impurities in concentrations from ppm to several percents [4, 5].

In this report we present an in-depth EPR study of the Mn^{2+} ions localization in Mn-doped (0 – 5%) ZnO thin films grown by rf magnetron sputtering on sapphire substrates. XRD and TEM, including HRTEM and EDS have been also used to complete the structural characterization of the thin films and their nanoscale defects.

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THIN FILM FIELD EFFECT TRANSISTORS USING PAPER AS GATE DIELECTRIC

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We report on the development of high performance hybrid flexible field effect transistors (FET) using paper as substrate and gate insulator, and indium oxide (In2O3) thin films as channel layer and source, drain and gate contacts. The In2O3 thin films were grown by pulsed electron beam deposition method (PED) at room temperature, in O2 atmosphere in the pressure range 10-2-2x10-2 mbar. By varying the deposition conditions, we can grow by PED low resistivity films (~10-2 Ω·cm) for the gate, source and drain contacts, and highly resistive (102 - 103 Ω·cm) semiconductor films for the FET channel. A Hall mobility of ~20cm2/Vs was measured for the conductive films grown on paper. Capacitor paper having 10 μm thickness was used as substrate and gate insulator. A 150 nm In2O3 film was grown on one side of a paper sheet to be used as gate contact. On the opposite side of the paper, 80 nm source and drain In2O3 films were grown using shadow mask patterning, resulting in an active channel geometry of 1.5 mm width and 150-200 μm length. The 60 nm channel film was grown on top of these contacts, also by shadow mask deposition. The electrical characterization of the FET was performed in the dark, in ambient air at room temperature. We measured a gate leakage current of ~20 nA at 5 V and an on/off current ratio of ~6x104. Drain saturation currents up to 3.5 mA were measured. A memory effect, with -0.4 V switch-on voltage and -4 V switch-off voltage was revealed by the transfer characteristics. The subthreshold swing was 0.33 V/dec in the switch-off regime and 0.48 V/dec in switch-on regime.

DEFECT CHEMISTRY IN PIEZOELECTRIC CERAMICS

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Experimental results on local fluctuations around the stoichiometric composition in complex substituted piezoelectric ceramics are presented. The geometry of defects generated by donor and/or acceptor substituents is simulated by using the supercell method. The effects of ion substitution on host cell parameters, lattice energy and electrostatic potential are calculated by using the General Utility Lattice Program (GULP).

OXIDE/METAL/OXIDE TRANSPARENT ELECTRODES FOR PLASTIC ELECTRONICS AND PLASTIC SOLAR CELLS

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In this paper we present the physical properties of two types of multilayer structures: oxide/metal/oxide obtained by successive sputtering depositions of metallic targets (In:Sn, Zn, Ag, Au) in reactive atmosphere (for oxide films) or inert atmosphere (for metallic interlayer films). Very good quality transparent conducting thin films structures ($\rho=2\cdot10^{-5}\Omega\cdot$cm, T ~ 90%) were obtained. The morphological, optical and electrical properties were analysed and compared for the multilayer films deposited in identical conditions on glass and PET substrates. The influence of substrate nature on the morphological properties is more pronounced in the case of zinc oxide films. The Haake figures of merit at λ=550nm are comprised between $4\cdot10^{-3}\Omega^{-1}$ and $29\ \cdot10^{-3}\Omega^{-1}$ in function of the nature of the metallic interlayer. The stability of electrical properties with the temperature of oxide/metal/oxide films is remarkable in comparison with the usual behaviour of single oxide films.

TOWARDS MODELING TUNGSTEN-BORATE-TELLURATE GLASSES

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The structural origins of glassy materials are very important in science up today. For clarifying the structure of the binary tellurate glasses some work is done in analyzing the basic structural units whereby [TeO_4] trigonal bypiramid and [TeO_3] trigonal pyramids are found in crystals and in glasses. Alone pair of electrons occupies one equatorial site of the Te sp^3d hybrid orbitals in the [TeO_4] unit and also the apex of the sp^3 hybrid orbitals in the [TeO_3] unit. The axial Te-O bonds of the [TeO_4] unit are little longer than the equatorials bonds. Tungsten ions have an intermediate behaviour between network formers and modifiers.

In brief, despite many characterizations, the structure of tungsten-borate-tellurate glasses is still subject to discussion. The purpose of this paper was to approach the structure of these glasses using the XRD, IR, UV-VIS, Raman spectroscopy and DFT calculations.

The incorporation of WO_3 into borate-tellurate glasses causes both the diminishing of trigonal bypiramids and the forming of non-bridging oxygens. The band situated about 650cm^{-1} due to Te-O_{ax} vibrations also shifts slightly towards lower frecquencies. This can be attributed to the formation of non-bridging oxygens Te-O^-, that, in addition, cause the progressive band broadening.

The accommodation of the network with the excess of oxygen and the higher capacity of migration of the tungsten ions inside the host network can be associated with a change of tungsten coordination, from [WO_4] to [WO_6] structural units.

Poster Presentations

EARLY PROCESSES IN SiC/SiO_2 CORE-SHELL NANOWIRES GROWTH VIA CHEMICAL VAPOUR DEPOSITION

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One dimensional nanostructures like nanotubes or nanowires are studied with increasing interest because of quantum confinement effects and remarkable optical, electronic and mechanical properties compared to bulk materials.

Many different materials have been used to obtain nanowires, among them, silicon carbide is one of the most promising for its superior properties such as temperature stability, thermal conductivity, hardness, chemical stability. Furthermore, its biocompatibility makes it possible to use SiC nanowires for nanomedical applications.

For device development it is very important to fully understand the mechanisms responsible for the growth of this kind of nanostructures.

In this work we report the analysis of the first stages of the growth of self-assembled β-SiC/SiO_2 core/shell nanowires (NWs) on Si substrate.

The synthesis of the NWs was carried out in a home-made CVD system using carbon monoxide as precursor and nitrogen as carrier gas. The substrates were previously dipped in a $Ni(NO_3)_2$ ethanol solution that act as catalyst. A non-ionic surfactant was added to the solution in order to enhance the wettability of the substrate.

The samples have been analysed using Field Emission Gun scanning electron microscopy (SEM FEG) JEOL 6400F and transmission electron microscopy (TEM) JEOL 2200FS.

Nickel nitrate is first thermally decomposed to nickel oxide, then a reduction owing to the presence of carbon monoxide takes place, producing metallic nickel, which forms a liquid alloy with the silicon of the substrate. The surface tension of the liquid creates some clusters that serve as nucleation sites for nanowires growth.

Immediately after heating, no aggregation of the catalyst could be found, with a time of two minutes, cluster formation (the so called “dewetting” process) could be observed. After a growth time of only 5 minutes, nanowires with core-shell structure were already present. The most superficial layers of the substrate were analysed using TEM Planar sections.

DOPED $Ba(Zn_{1/3}Ta_{2/3})O_3$ DIELECTRIC CERAMICS FOR MICROWAVE APPLICATIONS

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Barium tantalate based complex perovskites are very attractive solutions for microwave applications due to their very low dielectric loss. Moreover, the microwave devices miniaturization is very important for filters, antennas and oscillators used in the communications field at frequencies higher than 10 GHz. The ceramics of Nb^{5+}, Zr^{4+} or Eu^{3+} doped $Ba(Zn_{1/3}Ta_{2/3})O_3$ (BZT) complex perovskite oxide were obtained by the conventional solid-state reaction method from high purity starting materials. Preparation of such complex dielectric materials requires special thermal treatments in order to control the densification, the cation ordering, the secondary phases presence and to enhance the quality factor. BZT samples were sintered in air, at temperatures between 1400 and 1600 °C, for 2 h. X-ray diffraction, scanning electron microscopy and dielectric measurements were used for compositional, structural, morphological and dielectric characterization.

All BZT ceramics present single-phase compositions after polishing the surface in order to remove the layer in which zinc volatilization occurred. The 1:2 ordering of zinc and tantalum cations on the octahedral positions of the perovskite structure was observed with the increase of the sintering temperature. The SEM images evidence a porosity decrease, as well as an intense granular growth with sintering temperature increasing; the bigger grains, with quasi-spherical or polyhedral shape, reach 10 μm when sintering at 1600 °C. The microwave dielectric parameters of BZT resonators were measured by using Hakki-Coleman method. The dielectric constant varies between 17 and 31. BZT ceramic doped with 1 % Nb_2O_5 and sintered at 1600 °C exhibits the highest value for the quality factor, $Q \times f \sim 157$ THz at 10 GHz.

SYNTHESIS, CHARACTERIZATION AND GAS SENSING PROPERTIES OF CeO_2-Y_2O_3 BINARY OXIDES

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Ceria-based materials are attractive due to their many applications in catalysis, fuel cells , optical films, polishing materials, gas sensors, and other fields. The binary oxide system CeO_2-Y_2O_3 was prepared by sol gel methods using citric acid 10% aqueous solution like chelating agent at 80^oC under constant stirring. The molar ratio was Ce/Y=4:1. Then, ethylene glycol was added to promote the citrate polymerization by the polyesterification reaction. The citric acid ethylene glycol ratio was fixed at 2:1. The gel resulted was calcined at 800^oC for 2 hours**.** For the sensor fabrication, the disc of 4 mm diameter and 1 mm thickness was prepared by pressing of CeO_2-Y_2O_3 powder at 10 tone force/cm^2. The circulars gold electrodes with ϕ 1 mm were deposed on the both faces of the disc by e-beam evaporation in the conditions: $P=10^{-5}$ Torr, current 8 mA, time 60 s. The thickness of the gold electrodes 2.5 μm was determined with white light interferometric microscopy (VEECO Instruments) method. For the powder oxides the X-ray diffraction analysis was evidentied: the CeO_2 phase with a cubic structure with lattice parameter a=b=c= 5.409 Å. The size crystallites calculated with Scherrer formula for the powder calcined at 800^oC was 107,3 nm for the (111) peak. For the Y_2O_3 phase was identified a cubic structure with a lattice parameter a=b=c= 10.616 Å and a size crystallites 22,6 nm. Secondary was evidentied a phase with composition: $Ce_{0.6}Y_{0.4}O_{18}$. The SEM analysis was evidentied a large porous aggregates. From BET analysis was resulted a specific area 3,13 m^2/g, a pores size of 8.93 Å and a pores volume of $1{,}066 \times 10^{-3}$ cm^3/g. The sensor was developed a voltage of 376.95 mV for 140^0C function temperature, a sensitivity of 0.3 μV/ppm and a response time 30 seconds.

ELECTRICAL PROPERTIES OF LEAD TITANAT ZIRCONAT (PZT) CERAMICS DOPPING WITH NIOBIUM

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Ceramics with composition of $Pb_{(1-x/2)}(Zr_{0.52}Ti_{0.48})_{1-x}Nb_xO_3$ with x = 0.01 - 0.025, were prepared by conventional mixed oxide route were investigated.

A microstructure with mean grains sizes of 1– 3μm were found and the apparent density of 7.1–7.6 g/cm^3.

The dielectric and piezoelectric properties were determined on ceramic discs with a diameter of 10 – 18 mm and 1mm thick. Dielectric constant and losses have been measured in the range of frequencies at 40 Hz - 400 kHz using impedance analyzer. The piezoelectric constant k_p were calculate using the resonance and antirezonance values. It showed that the Nb additives were helpful in improving both of the dielectric and piezoelectric properties.

Keywords: PZT ceramics, ferroelectrics, piezoelectric properties.

EPITAXIAL INDIUM OXIDE THIN FILMS ON C-CUT SAPPHIRE SUBSTRATES

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Indium oxide (In_2O_3) thin films were grown by pulsed electron beam deposition method on c-cut sapphire substrates at 10^{-2} mbar oxygen pressure and rather low growth temperatures (up to 500°C). Smooth and stoichiometric In_2O_3 thin films were obtained as evidenced by Scanning electron microscopy and Rutherford backscattering spectrometry. X-ray diffraction analyses showed that the In_2O_3 cubic phase is observed even for room temperature growth, while increasing temperatures lead to a better crystallinity. Epitaxial thin films were obtained at substrate temperatures as low as 200 ºC and the epitaxial relationships with the single-crystal substrates were measured by asymmetric X-ray diffraction. Up to 300°C two phases, the classic and a disordered bixbyite phase are present with a "hexagon on hexagon" epitaxial growth. At 500°C the sole disordered bixbyite phase is observed with an epitaxy corresponding to a 30° rotation of the (222) plane with respect to the (00.2) c-cut sapphire basal plane. These films show a high optical transparency in the visible wavelength range and electrical properties which recommend them for applications that require semiconducting thin films.

SPECTROSCOPIC PROPERTIES AND THEORETICAL INVESTIGATIONS OF THE GADOLINIUM-LEAD-TELLURATE GLASSES

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Among many oxide glasses, tellurate glasses are preferred over silicate and phosphate glasses due to their relatively low phonon energy, high refractive index, high dielectric constant, good corrosion resistance, thermal and chemical stability.

The purpose of this paper was to approach the structure of gadolinium-lead-tellurate glasses with compositions $xGd_2O_3 \cdot (100-x)[7TeO_2 \cdot 3PbO]$, x=0-50mol% using the XRD, FTIR, Raman, EPR and UV-VIS spectroscopy.

IR data results show that the intensity of the bands corresponding to the $[TeO_3]$ and $[TeO_4]$ structural units modifies slowly with the increasing of the gadolinium ions. This compositional evolution of the structure could be explained by considering that the excess of oxygen may be accommodated by the deformation of Te-O-Te linkages and the formation of $[PbO_3]$ and $[PbO_4]$ structural units. By introduction of higher Gd_2O_3 content (x>30mol%) in the host matrix, the absorption UV bands become largely due to the fact that the position of the $Te\text{-}O_{equatorial}$ bonds are also affected.

The Gd^{+3} ions doped host matrix exhibits six resonance signals at $g \approx 2.0$, 2.8, 3.3, 4.3, 4.8 and 6. In the sites with $g \approx 2$, 2.8 and 6, gadolinium ions are characterized by a coordination number higher than six. The intensity of these signals decreases with the increase of the Gd_2O_3 content up to 50mol%. The weakening intensity of the clusters EPR line could be due to the Gd^{+3} migrations inside the glass network. It can be pointed that the Gd^{+3} ions are generally suspected to improve their environment because the Gd^{+3} ions in these glasses can be considered as isolated in the sense of the absence of clustering.

MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF COMPLEX SUBSTITUTED PZT CERAMICS

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A solid-state reaction technique was used to prepare samples of $(Pb_x,Sr)(Zr,Sb,Ti,Mn)O_3$ with different lead contents. X-ray diffraction (XRD) technique and scanning electron microscopy (SEM) were used to study the structural and microstructural properties of the materials. The XRD study confirmed the formation of the tetragonal phase. The variation of the lead content induces changes of porosity and grain size. The dielectric properties depend on the lead content. The ceramic samples containing 1 wt% lead in excess exhibits the highest dielectric constant and the dielectric losses are less than 1%.

Keywords: solid-state reaction, PZT based- ceramics, morphotropic phase boundary, dielectric and piezoelectric properties

INFLUENCE OF ELAIDIC ACID ON ZnO NANOPARTICLES SURFACE OBTAINED BY SOL-GEL METHOD

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Many important researches have been made in the domain of nanocomposites materials based on polymeric matrices with inorganic nanoparticles fillers, because were find widespread applications for the development of functional materials.

The aim of the paper was to obtain ZnO nanoparticles by sol-gel method and surface modification with elaidic acid, trans isomer of the oleic acid ($C_{18}H_{34}O_2$), used as coupling agent.

Firstly, ZnO nanoparticles were prepared by sol-gel method from the solutions of zinc acetate dihydratate and ethylene glycol as precursors. The powder obtained after drying the gel was synthesized by calcinations at the temperature over 500°C for 3h at a rate of 7°C/min. Then, the surface of ZnO nanoparticles was modified with coupling agent in order to reduce the tendency of agglomeration of inorganic components. In order to obtain this we formed an organic layer to ensure the compatibility between inorganic nanoparticles and different polymeric matrices used depending of the desired applications.

The morphology of the ZnO sample was characterized by scanning electronic microscopy (SEM) and showed the presence of a mixture of nanoparticles with various sizes and shapes. Also, X-ray diffraction (XRD) and FTIR spectroscopy show that ZnO nanoparticles have a wurtzite crystalline structure (hexagonal phase) and point out the existence of the organic layer in surrounding of the oxide nanoparticles.

DIELECTRIC PROPERTIES OF Ni-Zn FERRITE DOPED WITH Dy

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Lately, extensive research has been focused on investigating Ni-Zn ferrite properties obtained by various synthesis methods. Also, it was proved that by adding small quantities of rare earth ions into Ni-Zn ferrite lattice the related properties could be improved.

Rare earths are playing an important role in defining dielectrical and magnetic properties of spinel-type ferrites for microwave and high frequency applications. The aim of our study is to show the effect of small quantities of Dy^{3+} in Ni-Zn ferrite synthesized by sol-gel autocombustion method using tartaric acid as fuel agent. Thus, two spinel-type nanostructured ferrites with chemical composition $Ni_{0.8}Zn_{0.2}Fe_2O_4$ and $Ni_{0.8}Zn_{0.2}Fe_{1.98}Dy_{0.02}O_4$ were obtained and studied after structural characterization by means of IR, DRX, SEM techniques.

Interesting correlations between dielectric properties and cation distribution of as-synthesized ferrites were done.

$Ni_{0.8}Zn_{0.2}Fe_{1.98}Dy_{0.02}O_4$ shows a lower dielectric loss value compared to $Ni_{0.8}Zn_{0.2}Fe_2O_4$, meanwhile the resonance frequency is suggesting possible performances for microwave applications.

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STUDIES ON THE TEXTURAL FEATURES OF SOME LAYERED DOUBLE HYDROXIDE MATRICES

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Layered double hydroxides (LDHs) porous layered matrices, in which magnesium was partially substituted by copper or nickel, were synthesized by a coprecipitation method. LDHs, known as hydrotalcite-like anionic clays, have received considerable interests in recent years owing to their applications as catalysts, catalyst precursors, adsorbents and ion exchangers. The micromorphology and structural characteristics of the substituted LDHs were characterized by field-emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD). The obtained results point out that the layered double hydroxides textural features can be tailored as a function of their composition. The possibility to shape the micromorphologic characteristics of layered double hydroxides can contribute to obtain LDHs with tailored nanoporous properties.

TERAHERTZ TIME-DOMAIN SPECTROSCOPY OF LOW-LOSS MICROWAVE CERAMICS

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Low-loss microwave ceramics continue to have a decisive influence on the evolution of the satellite broadcasting, telecommunications, and global positioning systems. These materials, which exhibit high dielectric constant and good temperature stability, are required to reduce the equipment size, enhance its reliability, and lower the manufacturing and operational costs. The characterization of such materials in submillimeter and terahertz waves is a challenge for the research field as the communications are moving towards higher frequencies.

Terahertz Time-Domain Spectroscopy (THz-TDS) is a powerful technique used to characterize the dielectric properties of materials in far-infrared domain. The THz-TDS technique has the advantage of better signal-to-noise-ratio than the conventional FTIR technique. Moreover, the measurements of spectral transmission intensity and intrinsic phase shifts of the propagating THz radiation through sample/reference allow direct determination of complex dielectric constant without using of the Kramers–Kronig relations.

Titanates and tantalates based ceramics prepared by conventional solid-state reaction were investigated by using THz-TDS. The transmission spectra were recorded with an Aispec pulse IRS-2000 Pro time-domain spectrometer (40 GHz – 7 THz) on about 0.2 mm thick samples. It was found that the THz values of the complex dielectric constant of the samples studied are in good agreement with the microwave data. In particular, from 5 GHz to about 100 GHz, a low frequency dispersion of the relative permittivity was observed.

INFLUENCE OF THERMAL TREATMENT ON THE FORMATION OF ZIRCONIA NANOSTRUCTURED POWDER BY THERMAL DECOMPOSITION OF DIFFERENT PRECURSORS

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In this paper we present a study on the preparation of nanostructured zirconia (ZrO_2) from different precursors, in order to establish the optimal conditions for obtaining the tetragonal zirconia phase. The precursors used were zirconium hydroxyde and two zirconia carboxylates, obtained by the redox reaction between zirconium nitrates and 1,3-propane diol or poly(vinil alcohol). The zirconia powders obtained by thermal decomposition of these precursors were characterized by means of differential thermal analysis (DTA), thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The presence of the tetragonal phase was clearly observed at temperatures higher than about 400°C. Monoclinic zirconia appears as a secondary phase starting at about 400°C in the case of zirconium hydroxide precursor, and at about 500°C in the other two cases. In all the three cases, the tetragonal phase is almost completely transformed into the monoclinic one at 1000°C. The most stable tetragonal phase was obtained by the redox reaction between zirconium nitrates and poly(vinil alcohol).

COMPARATIVE SYNTHESIS AND CHARACTERIZATION OF OXIDE NANOPOWDERS WITH LUMINESCENCE PROPERTIES

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Luminescent nanomaterials, a relatively new domain, provide challenges to both fundamental research and breakthrough development of technologies in various areas. Phosphors are efficient luminescent materials and irreplaceable in white light-emitting devices. In present paper the white light-emitting devices were manufactured combining InGaN-based blue light-emitting diodes with nanophosphors from garnet family, yttrium aluminum garnet ($Y_3Al_5O_{12}$ or YAG) and terbium aluminum garnet ($Tb_3Al_5O_{12}$ or TAG) doped with rare earth. We present the sol-gel method used for preparations of garnet nanopowders doped with rare earths and formation of garnet’s at 1000°C. The chemical structure, morphology and luminescence *properties* of garnets were studied

IN SITU VARIABLE TEMPERATURE X-RAY DIFFRACTION STUDIES ON SIZE SCALE OF $CuCrO_2$ POLYTYPES WITH DELAFOSSITE STRUCTURE

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We had first studied the thermal stability of 2H and 3R polytypes of $CuCrO_2$ nanocrystals obtained by hydrothermal method from room temperature up to 1100 °C under vacuum conditions. The variable temperature X-ray patterns had shown that the thermal stability depends by the polytypes of $CuCrO_2$ nanocrystal, 2H-$CuCrO_2$ has been more stable phase than 3R-$CuCrO_2$. Also, our work had revealed the effect of the synthesis method on the thermal stability of 3R-$CuCrO_2$ microcrystals obtained by hydrothermal method and conventionally solid state method. An interesting result is that synthesis method has no effect on the thermal stability of 3R-$CuCrO_2$ delafossite phase, the decomposition temperature has been found about 1000°C. High temperature X-ray patterns of 3R-nano and micro crystals had shown the size dependence thermal stability, the 3R-CuCrO2 micro crystal had kept the delafossite structure up to 1000°C compared with 500°C for 3R-CuCrO2 nanocrystal.

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LUMINESCENCE OF Ce^{3+}- AND Eu^{2+}- DOPED SILICA GLASSES UNDER UV AND X-RAY EXCITATION

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Ce^{3+}- and Eu^{2+}- doped silica glasses were prepared by impregnation of Ce and Eu ions, respectively, into porous silica glasses followed by high temperature sintering in a CO reducing atmosphere. The characteristic emission band of Ce^{3+} 5d → 4f transition peaking around 375 nm is observed in the luminescence spectra of Ce^{3+}- doped glass under UV and X-ray excitation. Its photoluminescence decay is governed by several tens of nanoseconds decay time. For Eu^{2+}-doped glass, the Eu^{2+} 5d → 4f emission band peaking around 430 nm is observed in the luminescence spectra and its photoluminescence decay is governed by a few microseconds decay time. The X-ray excited integral scintillation efficiency of about 82% and 120% of that of the $Bi_4Ge_3O_{12}$(BGO) crystal is obtained, respectively, for the Ce^{3+}- and Eu^{2+}- doped glasses. Scintillation light yield under gamma-ray excitation was also measured and compared with that of the BGO and CeF_3 crystals.

Keywords: Ce^{3+}, Eu^{2+} , Luminescence, Porous materials, Scintillation, Photoluminescence decay

SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF La0, 45Sr0, 55MnO3 PEROVSKITE FOR SOLID OXIDE FUEL CELL CATHODES

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La1-xSrxMnO3 perovskite solid solutions are the most common cathode materials used for solid oxide fuel cells (SOFC), which combined with YSZ electrolyte lead to optimal performance for high temperature operation of SOFC.

The purpose is to have a lower temperature for SOFC functionality, by reducing the electrolyte resistivity, increasing the electrode efficiency and using physical and chemical compatible materials.

In this paper, the structural characteristics and microstructures of the La0,45Sr0,55MnO3 (LSM) layer deposited on electrolyte support were investigated. Cathode and electrolyte were analyzed by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) to characterize the interface phase between the cathode and the electrolyte. CeO2-based electrolyte doped with various oxides (strontium, calcium and yttrium) was obtained by uniaxial pressing and then it was sintered in air at 1400oC. The cathode composition prepared by solid state reaction method was deposited on the electrolyte using a spray coating technique. The amorphous layers were sintered at temperatures ranged between 1000 -1050oC for 1- 4 hours to obtain the perovskite structure.

Preliminary results indicate the formation of specific phases and the cathode compatibility with this electrolyte.

OPTICAL AND STRUCTURAL PROPERTIES OF Au:TiO_2 THIN FILMS PREPARED BY MAGNETRON SPUTTERING: THE EFFECT OF THERMAL ANNEALING

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Noble metals embedded in a dielectric matrix may display specific optical properties due to the surface plasmon resonance (SPR) effect. The aim of this paper is to demonstrate that the optical properties of TiO_2 coatings with embedded Au nanoparticle, obtained by magnetron sputtering, can be controlled in the nanoscale region using thermal annealing at different temperatures. The as deposited samples were subjected to thermal annealing, in vacuum, at temperatures ranging for 300 to 800^0C. The thermal annealing affected the shape and size of Au nanoparticles, and also the phase of the dielectric matrix. The role of the shape and size distribution of the nanoparticles, as well the influence of the crystallinity and phase composition of the host matrix on the optical response of the thin films was also studied. Further analyses like surface coloration and reflectivity were used to determine the optical properties of the samples.

X-ray diffraction (XRD) was used to determine the thin-film structure and crystallinity as a function of the annealing temperature. XRD analysis confirmed the presence of gold in all the samples and the crystallization of the TiO_2 matrix for the samples annealed at temperatures above 400^0C. The XRD data also shows clear evidence of the crystallization of the TiO_2 matrix in the anatase phase at 400^0C. With further increase of the annealing temperature, there is a change from the TiO_2 anatase phase into rutile-type structure. Simultaneously, the Au atoms are organized in crystalline nanoparticles (revealing an fcc-type structure, with the (111) preferential growth orientation).

SYNTHESIS BY HYDROTHERMAL METHOD OF $BaTiO_3$-BASED NANOMATERIALS

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The high permittivity, relatively low losses and good ferroelectric properties make the barium titanate-based materials widely used as dielectric material for multilayer capacitors or dynamic random access memories, among other applications [1]. However, modern electronics demand very small functional structures but it was observed that there is a strong dependency of the functional properties on the particles size and shape. For example, there is a critical size for the $BaTiO_3$ (BT) where the ferroelectricity no longer exists, although for nanoparticles with “cylindrical” shape the critical size concept is no longer valid [2] Therefore, the synthesis of nanoferroelectrics with different size and shapes are of key importance to understand the effect of the dimensionality on the ferroelectric properties. The present authors demonstrated that size and shape control of BT nanoparticles can be achieved by hydrothermal method [3]. Here we show the formation of tetragonal BT nanocubes with ~ 90 nm in size when barium acetate is hydrothermal treated at 135 ºC for 5h with titanium isopropoxide in the presence of sodium hydroxide as mineralizer. Adding strontium acetate, round shaped nanoparticles of $Ba_{0.7}Sr_{0.3}TiO_3$ (BST), with cubic structure and 50 nm in diameter, have been obtained in the same synthesis conditions. BT and BST ceramics have been obtained starting from the as prepared hydrothermal powders and their dielectric properties have been evaluated. This study demonstrates the potentiality of the hydrothermal method in the morphology control of the BT based nanomaterials.

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BROADBAND DIELECTRIC SPECTROSCOPY of PARAELECTRIC $Ba_{0.6}Sr_{0.4}TiO_3$ CERAMICS

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Ferroelectric materials are very attractive solutions for tuneable devices in RF and microwave domain. Most of the applications require materials with moderate dielectric loss at room temperature. $Ba_{1-x}Sr_xTiO_3$ solid solutions have received much attention for their high electric-field tuneability. However, in the ferroelectric state, the $Ba_{1-x}Sr_xTiO_3$ materials exhibit high dielectric loss. Therefore, for most of the electrically controlled devices, the BST materials can be used only in the paraelectric state.

$Ba_{0.6}Sr_{0.4}TiO_3$ (BST) ceramics were prepared by conventional solid-state reaction method from high purity starting materials. Four sintering temperatures (1300, 1350, 1400, 1450 °C) were used in order to investigate the influence of the microstructure on the dielectric properties of the materials. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were employed for morphological and structural characterization of BST samples. The broadband frequency behaviour of the complex dielectric constant was carried out using a broadband Alpha Analyzer from Novocontrol. The samples were placed in a parallel plate capacitor and the temperature was controlled using a Quatro Cryosystem. Dielectric spectra were recorded in the frequency range from 10 mHz to 10 MHz and in a temperature range between −100 and +100 °C. The data were acquired as a function of frequency under nearly isothermal conditions. All BST samples show a sharp ferroelectric-paraelectric transition with the Curie point near 0 °C. A monotonically decreases of the dielectric constant with the increase of the frequency was observed. At frequencies lower than 100 Hz, the conductivity contribution became significant, especially at high temperature. The microwave investigations at room temperature revealed dielectric constant around 4000 and loss smaller than 1% at 0.7 GHz. The results indicate that the paraelectric BST ceramics can be utilized for RF and microwave devices.

DETERMINATION OF THE TENSIONS APPEARING IN CERAMIC LAYER APPLIED TO THE BLADES FOR AIRCRAFT ENGINE

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The achievement of the ceramic layer on the turbine blades for aircraft engine is now a usual method used for increasing the strength and durability of the blades. Turbine blades for aircraft engine are subdued, during functioning, to thermal and mechanical stresses, which can lead to their deterioration.

One of the problems which appears to the ceramic coatings is the resistance of the deposited layer, to the tensions in the turbine blades under the action of the centrifugal force and force produced by the gas stream.

In this paper is carried the analysis of these solicitations onto a layer of ceramic material deposited on the moving blades of aircraft engine Tumanski R13 which equips MIG 21.

A calculation model was developed, using the finite element analysis module, from the software CATIA V5R19. The calculation has taken into account/consideration the composed load due to the forces that are acting on blades. There is a rigid connection between the ceramic layer model and the model of the base material. The resulting tension was determined on the basis of Von Mises’s equivalence theorem.

The analysis was made on the moving blades from both turbine stages of the engine and this led to a comparison of the way how the different geometry of the blades can influence the stress distribution in the ceramic layers due to tangential shear stress occurring at the interface between the coating layer and the base material.

PERMITTIVITY BEHAVIOR in FERROELECTRIC TRANSITION of PURE TGS CRYSTALS

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Triglycine sulphate (TGS) is a ferroelectric crystal, which exhibits a typical second order transition of order-disorder character. Usually, at Curie temperature, ferroelectric materials show a very rapidly increase of dielectric constant to a very high peak value. This increase, for TGS crystals, is related to the evolution of the ferroelectric domains and it was estimated that about 80% of the dielectric constant value is caused by the ferroelectric domain walls oscillation.

The relaxation mechanism was characterized by dielectric spectroscopy. The dielectric dispersion was investigated on the frequency range 1 Hz to 10 MHz, over the temperature range from -120 to +65 ^{o}C, crossing up and down the Curie Point. Both components of the complex permittivity of pure TGS crystals show different frequency behavior related to ferroelectric or paraelectric state of the sample. Cole-Cole diagrams of the two components of permittivity clearly exhibits two types of relaxation in the ferroelectric phase, with characteristic time LOW~10^{-3} sec, HIGH ~10^{-7} sec and unexpectedly a fade third MIDDLE relaxation time of ~10^{-5} sec. The first, the lower frequency relaxation mechanism, is recognized to be related with ferroelectric domain wall relaxation (domain cropping) and has an important thermal evolution, with activation energy of 0.7 eV (approximately 28 k_BT). The second one, the higher frequency relaxation mechanism, is ascribed to the "critical slowing down" effect, similar to the relaxation in the paraelectric phase. This relaxation time was found to be independent of temperature. A third, middle relaxation mechanism, apparently does not have a specific physical support. It seems to be a sort of interaction of the two previously mentioned relaxation mechanisms.

CHARACTERIZATION AND OPTICAL PROPERTIES OF LANTHANUM-CERIUM OXIDE NANOPARTICLES BY HYDROTHERMAL GROWTH

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$La_2Ce_2O_7$ nanocrystals were synthesized via the co-precipitation & hydrothermal method. Thermo gravimetric analysis (TGA), X-ray diffraction (XRD), Electron Diffraction Spectroscopy (EDS), Raman Spectroscopy, Transmission Electron Microscope (TEM) and Fourier Transform Infrared Spectroscopy (FT-IR) were utilized to characterize the thermal decomposition, phase structure and morphology of the products. The Lanthanum Cerium Oxide nanoparticles show UV absorption and room temperature Photoluminescence (PL).

TRITIATED MgB_2 SUPERCONDUCTING CERAMICS

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Dense superconducting MgB_2 ceramics (95% of theoretical density), obtained by spark sintering technique were loaded with tritium for 2 and respectively 264 hours in ampoules fed by a uranium bed generator. X-ray diffraction shows an increase of the *c*-axis lattice parameter to 3.5263 Å and 3.5364 Å after 2 hours and 264 hours, respectively. Two contradictory features were observed as a result of tritium loading: *i*) the tritiated samples have the same critical temperature and *ii*) they display a spectacular increase of the irreversibility (Fig. 1), hence, of the critical current density. The increase of the critical current density is expected as the result of the structural damages produced by the tritium intake but the increase of the critical temperature by doping was not anticipated in two-gap superconductors. It might be the result of the change in phonon frequency due to the presence of the lighter atoms of tritium. The extension of the phonon frequency to higher values, leads in turn to the increase of the critical temperature, which is dependent on the high frequency modes.

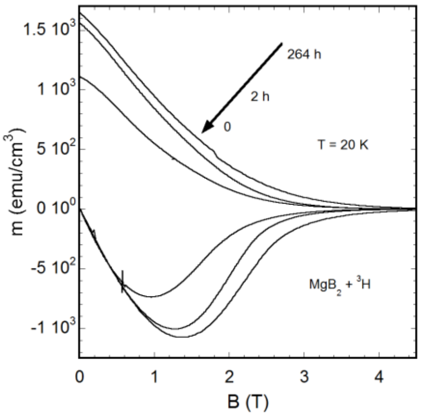

Fig. 1. Field dependence of the magnetization of tritium loaded MgB_2.

STRESS ASPECTS FOR DEFECT EMISSION IN ULTRASONIC SPRAY PYROLYSIS DEPOSITED FLUORINE DOPED ZINC OXIDE (FZO) THIN FILMS

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Zinc oxide thin films are the most attractive and also promising substitution material for the ITO film as transparent conducting oxides (TCO) electrode in optoelectronic devices. FZO thin films with varying Fluorine dopant level (0-5%) were prepared on corning glass substrates by ultrasonic spray pyrolysis. Structural and electro-optical properties of as deposited films were investigated in order to optimize the growth of FZO thin films as TCO for photovoltaic applications. The films are nanocrystalline and fit well with the polycrystalline hexagonal wurtzite structure with preferred orientation along c-axis. ZnO thin films exhibits hexagonal platelets morphology analysed by Field emission scanning electron microscope (FESEM). Crystallinity deteriorated with increasing fluorine dopant. Fluorine incorporation in ZnO thin films confirmed by Energy dispersive X-ray analysis (EDX). Raman spectroscopy and photoluminescence (PL) analyses identified the stress due to defects introduced by Fluorine in the ZnO thin film. A special emphasis was devoted to E_2 high phonon mode analysis by Raman spectra. PL spectra show the formation of a shoulder in band emission induced by fluorine dopant and also shoulder peak intensity increases with increasing percentage of Fluorine content, consistent with Raman results.

S6
Advanced Materials for Photovoltaics and Applications

Invited Papers

CURRENT RESULTS AND FUTURE STEPS OF PV SILICON GROWTH IN EXTERNAL FIELDS

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The present and future demands of R&D for industrial crystal growth of photovoltaics are concentrated on improved crystal quality, increased yield, and reduced costs. To meet these challenges, the size of the melt volume must be markedly increased. As a result, violent con-vective perturbations appear within the melts due to turbulent heat and mass flows. They disturb the single crystal growth and give rise to compositional inhomogeneities. Further, the control of the melt-solid interface shape and morphological stability is of crucial importance to minimize the thermomechanical stress and growing-in defects, respectively. The application of external force fields proves to be an effective tool to master these targets [1].

After introducing different stabilizing external fields, like constant and accelerated melt rotation, mechanical vibrations, and electric current, the paper focuses on use of magnetic fields. Especially, non-steady fields became very popular, since in this case the needed strength of the magnetic induction is much lower than for steady fields. A new low-energy low-cost technology, named KRIST*MAG*® [2], combines heat and travelling magnetic field (TMF) generation in the heater placed close to the melt crucible.

Today, multicrystalline (mc) ingot crystallization and single crystalline (sc) Czochralski growth are the main production techniques for photovoltaic (PV) silicon. Considerable efforts are directed on monocrystalline seeding and drastic oxygen reduction, respectively. In both cases the application of magnetic fields are of increasing interest. A spectacular development of yield per run has been taken place by increase of ingot mass from 250 kg to 800 kg. However, numerous process problems become more importance with increasing ingot size, such as i) the control of temperature field and curvature of melting point isotherm even at masses > 600 kg, ii) reduction of the diffusion boundary layer to avoid SiC and Si_3N_4 inclusion generation near to the interface, and iii) minimization of the interaction between melt and container wall, for example. For the first time, a KRIST*MAG*® heater-magnet module [3] was tested in a commercial mc-Si crystallization equipment with G5 ingot geometry of >600 kg melt charge. First of all the TMF component was applied to enhance the melt mixing in order to remove the harmful diffusion boundary layer. Additionally, the TMF frequency, phase shift and travelling direction turned out to be very sensitive parameters for tailoring of the interface shape. Finally, a double-frequency regime was applied to minimize the melt flow at the container wall and to shift its velocity maximum towards the bulk centre [4].

In case of Czochralski growth the TMF can help to decrease the oxygen content and radial temperature gradient that develops large {110} facets [5]. As a result a crystal of rectangular cross section is growing - a very favourable shape for solar wafer production.

Finally, during Si ingot crystallization ultrasonic vibration is able to reduce the harmful diffusion boundary layer very effectively. As was shown by numeric modelling a strong Schlichting stream is generated affecting the diffusion boundary layer immediately at the growing interface very effectively [5].

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GROWTH OF HIGH-QUALITY SI MULTICRYSTALS FOR SOLAR CELLS USING DENDRITIC CASTING METHOD AND NONCONTACT CRUCIBLE METHOD

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Part 1: The structure control for the growth of Si multicrystals should be done to reduce crystal defects. Dislocation is the most important crystal defect to be reduced, and the main origin of dislocations is random grain boundaries. We proposed the dendritic casting method to control the crystal structure and reduce random grain boundaries.

Dislocations in Si multicrystals can be largely reduced by decreasing the density of random grain boundaries, especially at the bottom of ingots. The dislocation density near a random grain boundary also becomes lower as the coherency of the random grain boundary increases. The arrangement of dendrite crystals grown along the bottom of ingots is effective for realizing such Si multicrystals, and dendrite crystals should be controlled to be fairly parallel to each other, because the arrangement of dendrite crystals is effective for decreasing the dislocation density by improving the coherency of random grain boundaries.

A method of controlling thermal conductivity under crucibles to control the arrangement of dendrite crystals was proposed [1]. Graphite plates with different thermal conductivities were used all over the bottom surface of crucibles. A highly cooled part for obtaining a highly supercooled part in a Si melt was intentionally and locally introduced on a wide graphite plate. Using the graphite plate with a line-shaped highly cooled part, most of the dendrite crystals appeared from the highly supercooled part, and their distribution was well arranged fairly parallel to each other. Using this method, solar cells with a high conversion efficiency have been obtained using large scale wafers cut from several ingots prepared by this method.

Part 2: Stress control is necessary when preparing high-quality multicrystalline Si ingots for solar cells using crucibles because crystal defects such as dislocations are mainly generated by stress in the ingots. Conventional crystal growth methods using crucibles cannot control the stress caused by expansion of Si. We proposed a noncontact crucible method using conventional crucibles that reduces the stress in Si multicrystalline ingots [2].

In the present method, the Si melt has a low-temperature region in its central upper part. Nucleation occurs on the surface of a Si melt using a seed crystal, and a crystal grows inside the Si melt without touching the crucible wall. The crystal grows naturally in the low-temperature region, then it is slowly pulled upward while ensuring that crystal growth remains in the low-temperature region. In the growth for Si crystals, it is difficult to prevent the growth from crucible wall coated with a Si_3N_4 layer which has many nucleation sites. The diameter of the ingots increased almost linearly with the temperature reduction. The temperature reduction is defined as the difference between the starting temperature at which an initial crystal is first observed to grow from the seed crystal and the final temperature. A large ingot diameter of 26 cm was obtained. The maximum minority carrier diffusion length of 610 μm was measured at a point on a cross section. We have confirmed that such ingot growth is feasible in a crucible and large ingots can be grown even in a small crucible. Si multicrystalline ingots with few dislocations and only twin boundaries are expected to be realized by this method.

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SINGLE-CRYSTAL CdTe THIN FILMS AND NANOCYLINDERS

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Exceptional quality, epitaxial films of CdTe have been developed using the Pulsed Laser Deposition method. The films have been extensively characterized by X-ray diffraction, transmission electron microscopy and photoluminescence. The tendency of CdTe films to twin along <111> directions is suppressed via careful control of growth conditions especially deposition rate and substrate temperature. A novel approach to developing electronic devices on a range of platforms will be described. At somewhat higher growth temperatures, catalytically seeded nanocylinders are developed. These structures form with a narrow height dispersion making them interesting active elements to be embedded in flexible structures.

ORIGIN AND DEVELOPMENT OF GRAIN AND TWIN BOUNDARIES IN PV SILICON

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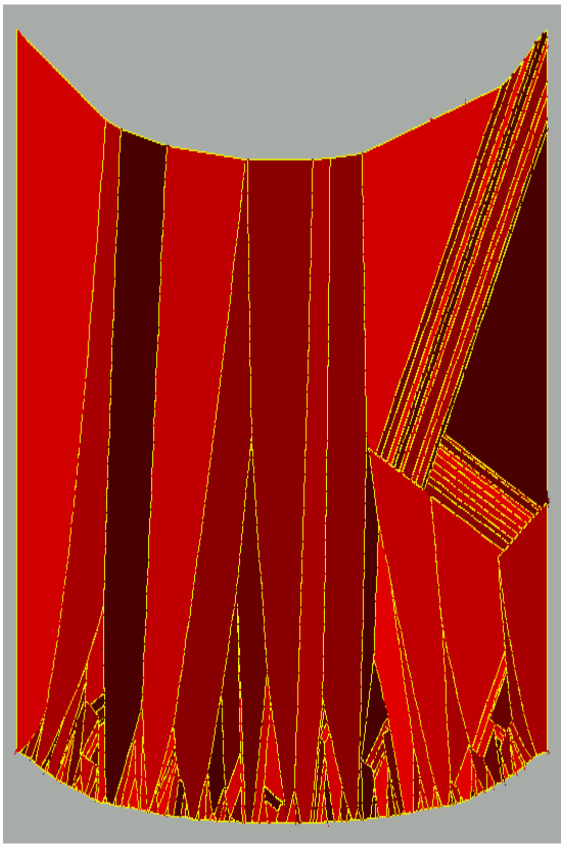
Industrials involved in photovoltaic silicon production are facing a shortage of silicon raw material and should use less pure silicon than previously. The decrease of PV properties associated to impurities has renewed the interest in the defect structure of the ingots. Among these defects, the grain boundaries are of particular interest because they are places for carrier recombination but also because they attract impurities. A bibliographic review [1] shows that Si ingots present three different grain structures:

- Large grains, with a centimeter size,
- Twins,
- Small grains, millimeter in size or less, also known as grits.

In order to get a better understanding of the physico-chemical phenomena leading to the grain structure, a series of theoretical investigations and numerical approaches is developed. Our overall model takes into account:

- Time dependent heat transfer during the whole ingot casting, giving the solid-liquid interface shape and velocity, as well as liquid and solid thermal gradients, versus time.
- Random or orientated nucleation on the crucible bottom.
- Grain boundary by grain boundary analysis of the grain/grain/liquid triple line. Its morphology, faceted or rough, is deduced from crystallographic, thermodynamic and kinetic local conditions [2].
- Distinction is made between coherent (Σ3) and incoherent (all other) grain boundaries.
- In case of rough triple line, a specific kinetic law is used in order to find the growth direction of the grain boundary.
- In case of facetted-rough triple line, the facet is overrun by the rough grain, which needs less undercooling. The growth direction of the grain boundary is given by the facet.
- In case of facetted triple line, the grain boundary follows the bisector of the two facets and the probability of twinning is taken into account [3], which generates new grains in the structure.
- The intersection of grain boundaries is considered.
- A model of grit solidification, including segregation and precipitation of impurities, growth of SiC or Si3N4 precipitates and subsequent nucleation and growth of Si on these precipitates has shown good agreement with experimental results [4, 5].

Numerical results in the simplified approximation of 2D axi-symmetric thermal field and 2D Cartesian grain structure will be presented and discussed.



Grain structure predicted by a 2D preliminary model. The nucleation at the bottom of the axi-symetric crucible is random. 10% of the grain-grain-liquid triple lines are facetted. Some of then give birth to twins, with a probability of 10±0,5 cm-1. Grain colours are arbitrary.

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CRYSTALS FOR THE LOW CARBON ENERGY TECHNOLOGIES

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Technological innovations have enabled economic and social progress for mankind since centuries. Thereby, the development of new materials has triggered the introduction of new technologies. This is obvious from the Kontratieff-waves, which describe the modern world economy since the beginning of the 19th century. New steel alloys allowed the use of steam engines (1st wave) and railways (2nd wave), the progress in chemistry was the basis for the 3rd wave, the refinery of petrochemicals was the prerequisite for mobility using automobile (4th wave). The 5th wave from 1970 to 2010 was the information technology, which became possible only to the availability of crystals with tailored properties, such as silicon crystals or compound semiconductors, and the corresponding methods for their industrial production. Affordable eco-friendly energy and its efficient usage is one of the mega topics for the next decades and therefore might become the 6th Kontratieff-wave. In this context crystals are key components again: Multi- and monocrystalline silicon for energy generation, wide band gap semiconductors for an increase of the energy efficiency during energy transmission and conversion to mentioned only two classes of crystalline materials.

In this paper some contributions of Fraunhofer IISB in the field of crystal growth of silicon for photovoltaics will be highlighted. According to the International Technology Roadmap for Photovoltaic there will be a tremendous increase of the market share of so-called mono-like silicon which is grown by directional solidification (DS) using seed crystals. However, the growth of large silicon mono-crystals by DS is still a big challenge because the interaction of the growing crystals with the crucible wall can cause heterogeneous nucleation. This leads to a poly-crystalline structure and therefore reduces the yield of monocrystalline material. It will be shown, that most of the misoriented grains at the crystal periphery are caused by a twinning process. This problem can be minimized if proper seed orientations and growth conditions are chosen. Generally the <100> seed orientation turns out to be most difficult with respect to mono-crystalline growth. Heterogeneous nucleation originating from the crucible walls seems to be a minor problem.

EFFECTS of PROTON and ALPHA PARTICLES IRRADIATION on the PERFORMANCES of CdS/CdTe PHOTOVOLTAIC CELLS

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Due to their physical and chemical properties (such as suitable band gaps, large absorption coefficients and good chemical stability) CdS and CdTe thin films are interesting materials for electronic and optoelectronic devices, including particularly, the photovoltaic cells for terrestrial and space applications. For this specific application, it is of prime importance to study the influence of ionizing radiations both on the structural, electrical and optical properties of the component materials and on the photovoltaic structures based on them.

In this paper, the photovoltaic cells based on CdS/CdTe thin films, produced by thermal vacuum sublimation, were irradiated with protons and alpha particles, at room temperature. The irradiation energy for both protons and alpha particles was 3 MeV and the fluencies were 10^{14} protons/cm^2 and 10^{13} alpha particles/cm^2, respectively. The prepared samples configuration was a”superstrat” one having CdS as ”window” layer and CdTe as ”absorber” layer, respectively. The films were deposited by conventional thermal vacuum evaporation technique. The CdS/CdTe is given a post deposition $CdCl_2$ heat treatment which enables grain enhancement, reduces the defect density in the films, promotes the interdiffusion of the CdTe and CdS layers and thereby improves solar cell efficiency. The effects of irradiation were studied by investigating the changes in the electrical and optical properties of the cells. The structures were electrical characterized before and after protons and alpha particles irradiation by measuring the I-V characteristics both in dark and in AM 1.5 conditions and the results were compared. The parameters characterizing a photovoltaic cell, short-circuit current, open circuit voltage and fill factor were calculated before and after protons and alpha particles irradiation and the obtained values are comparable, but less for irradiated samples. Moreover, the maximum power obtained for a photovoltaic cell half decreased after alpha particles irradiation. A discussion about the possible origin of those defects is given. In this sense, it was found that proton irradiation in the above mentioned conditions results mainly in the introduction of defects at the CdS/CdTe interface.

Keywords: Solar cells, Cadmium Telluride, Thin Films, Proton and Alpha Particles Irradiation

Oral Presentations

RAMAN INVESTIGATION AND ELECTRO-PHYSICAL PROPERTIES OF II-VI WIDE-BAND GAP FILMS

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In recent time II-VI wide-band gap semiconductor compounds, such as ZnS, ZnSe, ZnTe pay a higher attention in photovoltaic applications, biological and gas sensors, photoelectronic devices, etc. Owing to its optical band gap (~3,7 eV for ZnS, ~2,7 eV for ZnSe, ~2,3 eV for ZnTe) these compounds may be used as a window layers in tandem solar cells, light-emitting diodes, etc.

II-VI semiconductor films were deposited onto glass (Raman investigations) substrates and ITO (indium tin oxide) substrates with conduction sub-layer (molybdenum, chrome, ITO) (investigations of electro-physical properties) in vacuum equipment VUP-5M at a residual pressure of ~5•10-3 Pa. Temperature conditions were: evaporate temperature - Te = 1073 K (ZnSe); 1273 K (ZnS); 973 K (ZnTe), substrate temperature was varied in the range of Ts = (373 – 673) K, evaporation time was t = 1-3 min (Raman investigations), 10 min (electro-physical investigations).

Raman spectroscopy investigations were performed on spectrometer TRIAX 320 (1200 grooves) by using a He-Ne line-focused laser with wavelength of 441,6 nm and power of 50 mW at room temperature (297 K). Exposure was 5 sec., acquisition – 20 sec.

For investigation of electro-physical properties were measured dark current-voltage characteristics (I-V curves) of sandwich-structures. Furthermore, σ-T dependencies were investigated on ohmic, in some cases – on quadrant areas of I-V characteristics.

As a result of Raman investigations, we interpreted the series of some peaks due to the so-called longitudinal optical LO-phonon replicas in condensates. The replicas are better resolved at excitation conditions close to resonance Raman scattering conditions.

As a result of electro-physical investigations, I-V curves have a view of multilayer structures ITO-semiconductor-metal are determined by chalcogenide films condensation conditions, by their crystalline structure and by the material of cup- and bottom current-conduction contacts.

COMBINATORIAL SCREENING OF Sn-Sb-S THIN FILM LIBRARIES FOR THE APPLICATION OF NEW SULFOSALT-BASED PHOTOVOLTAIC DEVICES

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Sulfosalt thin film Sn-Sb-S composition-spread libraries were grown onto un-heated glass substrates by thermal vacuum evaporation using two separate Sb2S3 and SnS sources at an operating pressure of 10-5 mbar. The libraries were annealed in vacuum for one hour in the temperature range 100-500 °C (50 °C increments). The combinatorial synthesis approach allows for the rapid exploration of new compounds in the Sb2S3-SnS binary phase diagram and to assess their suitability to act as absorber layers in terrestrial photovoltaic devices. The effects of compositional change and annealing temperatures on the structural, morphological, opto-electronic and thermoelectrical properties have been experimentally investigated. Composition and film thickness (~2 μm) were measured using X-ray fluorescence (XRF). The crystallographic structures and phase evolution were assessed by X-ray diffraction (XRD). Crystallinity and grain size were found to strongly depend on the composition and the post deposition annealing conditions. While lower Sb concentrations promoted polycrystalline growth, strong preferred orientation prevented the identification of which specific phases were present. Raman spectroscopy was further used to compliment the XRD technique for the identification of intermediate phases. The films were found to exhibit an amorphous structure at temperatures below 150 °C. Above this transition temperature, a polycrystalline structure was identified. Photoconductivity measurements indicated ideal optical band gap values of between 1.3 and 1.5 eV across the whole compositional range, irrespective of whether the film was amorphous or crystalline, with the strongest photo-response found at the higher Sn concentrations. Hot probe measurements generally showed n-type conductivity, with some evidence of p-type conductivity at higher annealing temperatures (> 300 °C).

FLEXIBLE PROTECTIVE LAYER FOR THIN FILM SOLAR CELLS APPLICATIONS IN SPACE

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In our research group the epitaxial lift-off (ELO) method has been developed as a method to reduce the costs of III-V solar cells [1]. The ELO method encompasses the separation of the solar cell from its growth template (wafer) by the etching of an intermediate release layer. The cost reduction is achieved by reusing the wafers for the production of subsequent solar cells. The use of the ELO method results in flexible, thin-film solar cells, with efficiencies equal to that of cells on a wafer.

Most of the III-V solar cells produced nowadays are used for powering satellites. The ELO technique produces flexible solar cells, meaning that flexible solar panels for space can be designed. Besides new deployment options flexible panels will also significantly reduce the weight of the panels, which in turn will reduce launch costs of the satellites. The only additional requirement for a flexible panel configuration is the replacement of the brittle cover-glass shielding the solar cell by a flexible alternative. These shielding layers should besides being flexible also be transparent, since they are applied on top of a solar cell. Also, they should remain flexible and transparent in the harsh space environment (e.g. high energy UV and particle radiation, and atomic oxygen). Due the this environment most polymers, which are the obvious choice if transparent and flexible materials are desired, will degrade rapidly.

In this study we present the synthesis and characterisation of a candidate material for a flexible shielding layer for space application. This candidate material is silica/silicone that is synthesized by a sol-gel reaction to minimize the organic content. Also, first results of some space environmental tests (electron and UV irradiation) on this material will be presented.

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STRUCTURAL AND COMPOSITIONAL PROPERTIES OF CZTS THIN FILMS FORMED BY RAPID THERMAL ANNEALING OF ELECTRODEPOSITED LAYERS

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The wide use of photovoltaic technologies in the future needs development of new cost-effective processes based on earth abundant, low cost and low toxicity materials. Among the most interesting new materials Cu2ZnSnS4 is attracting due to its excellent photoelectrical properties and due to possibility of using cheap chemical technologies for materials preparation. In this work Cu2ZnSnS4 (CZTS) thin films were formed by rapid thermal annealing (RTA) of sequentially electrodeposited Cu-Zn and Sn films in 5% H2S containing atmosphere. The precursor and annealed films were characterized by x-ray diffraction (XRD), micro-Raman spectroscopy, atomic force (AFM) and scanning electron microscopy (SEM) investigations.

The influence of the RTA parameters, such as the speed of raising temperature, duration of annealing (from 15 to 60 minutes) and the H2S annealing temperature (450-530˚C) on the morphology on the elemental and phase composition and properties of the Cu2ZnSnS4films was investigated. It was shown, that the fast rise of temperature (10-15˚C/sec) leads to the formation of multiphase films. At the same time the rise of temperature with lower speed (3-4˚C/sec) leads to the formation of the uniform Cu2ZnSnS4 films.

NEW PHOTOELECTRICAL PROPERTIES OF P3HT:PCBM BLEND THIN FILMS

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Polymers:fullerene bulk heterojunctions ITO/PEDOT:PSS/P3HT:PCBM/LiF/Al solar cells are at present ones of the most studied organics solar cells. Power conversion efficiency (PCE) which exceed 5% [1-6], or 6.5% for tandem solar cells [7] were demonstrated. However all the physical properties and the instability of these systems are not yet well understood and controlled. In this paper we present new electrical and photoelectrical properties of P3HT and P3HT:PCBM thin films subject at different cyclic measurements .

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COMPARATIVE STUDY OF $CuIn_{1-x}Ga_xSe_2$ AND $CuIn_{1-x}Al_xSe_2$ THIN FILMS OBTAINED BY SELENIZATION

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$CuInSe_2$ (CIS) is the most well-known material of a wide family of copper based chalcopyrites used as absorbent layer in thin film solar cells. In order to increase the CIS band gap for an optimal match with solar radiation spectrum CIS is usually alloyed with Ga to obtain $CuIn_{1-x}Ga_xSe_2$ (CIGS). Alternatively to Ga, alloys with Al (abundant and inexpensive) $CuIn_{1-x}Al_xSe_2$, (CIAS) allows a wide range of band gap energy. CIGS and CIAS thin films have been prepared by a two stage process consisting on the evaporation of precursor metallic layers and the subsequent selenization in controlled atmosphere. The different behaviour of the structural, optical and electrical properties of both chalcopyrites with a common base, the ternary $CuInSe_2$, has been studied depending on critical parameters such as the films thickness and the Ga or Al proportion, *x*. Polycrystalline CIGS and CIAS thin films have been obtained with chalcopyrite structure and orientation along the (112) plane for a wide range of Ga or Al proportion and film thicknesses. XRD diagrams have shown that the coexistence of CIGS and CIAS phases with different Ga and Al incorporation due to an incomplete substitution depends on the x values and the film thickness. For thicknesses lower than 2μm, CIGS samples have shown a single peak in the (112) reflection for a wide range of x values. However, the presence of several peaks around the (112) reflection appear from 1μm thickness and for x>0.2 increasing with the thickness. Band gap energy increase nonlinerly with the x values with different bowing parameters depending on the film thickness.

A COMPARATIVE STUDY OF THE ZnO FILMS DEPOSITED AT TWO WAVELENGTHS BY PICOSECOND LASER ABLATION FOR DYE SENSITIZED SOLAR CELLS APPLICATIONS

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ZnO films deposited by picosecond laser ablation using a Nd:YVO_4 laser (8 ps, 50/10 kHz, 0.17 J/cm^2), at 532 and 355 nm wavelengths were investigated. Depositions were carried out at room temperature (RT) starting from a pure Zn target in oxygen atmosphere (150-900 mTorr) at two gas flow rates (0.25 and 10 sccm) on indium tin oxide (ITO) and fluorine doped tin oxide (FTO) substrates. After deposition, the films were subjected to an annealing treatment for 2 hours in oxygen atmosphere (250-550 ^{0}C).

The influence of the wavelength, oxygen pressure, flow rates, type of substrates and annealing temperature on the morpho-structural characteristics of ZnO films were investigated. The optimal parameters of ZnO films for dye sensitized solar cells (DSSC) applications were determined.

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CIBALACKROT – A NEW MATERIAL FOR ORGANIC PHOTOVOLTAICS

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Recent efforts implying natural materials and synthetic derivatives of natural molecules for organic photovoltaics were performed. In this paper, a new organic material is presented. Cibalackrot is a relatively new material, obtained from indigo by one step condensation reaction. It proved to be a suitable organic semiconductor showing ambipolar behavior, with balanced mobilities for electrons and holes of $5x10^{-3}$ cm^2/Vs and $9x10^{-3}$ cm^2/Vs respectively. XRD, SEM, cyclic voltammetry, UV-Vis spectroscopy were used for characterization.

This material was tested for the development of photovoltaic devices such as solar cells, organic field effect transistors and light emitting diodes.

INFULENCE OF THE PHOTOVOLTAIC MATERIAL ON THE EFFICIENCY LOSS IN MOUNTAIN IN-FIELD CONDITIONS

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Commercial PVs are used in developing building integrated systems, installed in a broad variety of locations. The pre-dimensioning of the systems considers the PV general properties and usually generic data of the location (latitude, longitude, altitude, and clearness index) based on which solar irradiation data are generated. These data have an average degree of confidence and their use without an extended analysis of the weather data of the particular in-field location may lead to significant miss-estimations of the real output of the installed system. This becomes even more important for tracked PV systems (modules, strings or platforms).

The paper presents the results obtained in analysing four types of PV modules installed on a tracked PV platform, installed in the Transilvania University of Brasov, Colina Campus, a mountain region with ambient temperatures ranging from -25^o to 35^{o}C, with snowy winters and rather rainy weather. The platform consists of mono-, poly and amorphous silicon and CIS modules (overall power 1.5kWp) and has a step-wise pseudo-equatorial tracking system. The current, potential and energy output data are comparatively analysed in terms of conversion efficiency and conversion relative losses, considering a reference period of one year (2010); the weather data (solar total, diffuse and direct radiation, wind, precipitations) are correlated with the modules’ temperature, outlining significant differences and smaller relative losses for the thin film modules. The data are further compared with those collected from a fixed 10kWp PV platform (poly-crystalline silicon) and the overall effect of tracking is analysed, considering the increase in the amount of harvested solar radiation and in temperature on the module, which proved to have the most significant effect. The results represent input data in developing optimised tracking algorithm, considering the optimal abatement from 90^o of the incident direct radiation, for avoiding over-heating. The data also outline the importance of accurate PV selection and system design, considering the implementation location.

GROWTH MORPHOLOGY AND ORIENTATION ANALYSIS OF MULTICRYSTALLINE SILICON INGOTS GROWN BY NONCONTACT CRUCIBLE METHOD

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Dislocations work not only as recombination sites for photogenerated carriers but also shunts of PN junctions of solar cells. Therefore, reduction of dislocation density is demanded to improve the conversion efficiency of solar cells. Crucibles used in conventional casting methods by unidirectional solidification constrain expansion due to the solidification of the Si melt during whole processes of crystal growth, then, dislocations are introduced in Si crystals by stress. Once dislocations are generated, they increase in subsequent unidirectional growth process. This occurs also in the case of mono-like crystal which investigated actively these days. Recently, we proposed a noncontact crucible method (NCM)[1] which enables reduction of stress due to constraint by crucible. In this method, nucleation occurs on the surface of a Si melt using seed crystals, and crystals grow inside the Si melt without touching the crucible walls. Then, the ingots continue to grow while being slowly pulled upward maintaining the front of crystal growth in the low-temperature region in the Si melt.

In the present study, growth morphology, defects, electrical properties and their relations in the Si ingots grown by NCM were studied with orientation analysis by electron back scattering diffraction patterns (EBSP), observation of etch-pit densities (EPD) and lifetime measurements. From Si ingots grown by NCM, vertical cross-section near Si seed and wafers with 180 μm in thickness were obtained. They had multicrystalline structure and the percentage of Σ3 among the whole grain boundaries was relatively high.

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ELECTRONICS AND PHOTONICS: TWO SCIENCES IN THE BENEFIT OF SOLAR ENERGY CONVERSION,

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This paper gives a global point of view on two sciences: Electronics and Photonics towards the solar energy conversion. The new research directions in these two sciences are pointed out in connection with the renewable energies. Building with sand…. The human being intelligence transformed the sand in buildings, glass, optical fibers, computers, solar cells etc. One of the most abundant chemical compounds in nature is the silicon dioxide, also known as silica. Silica is most commonly found in nature in sand or quartz. Silicon is prepared starting from silica and quite the whole electronics today is based on silicon. Photonics is based fundamentally on silicon dioxide (glasses, optical fibers, lenses). So finally the whole world today, based on electronics and photonics, with computers, optical transmission data, mobile phones, solar cells, use sand as starting material. Of course there are other elements and sand alone couldn’t be use to get all these ends. Beside inorganic materials, the new research achievements show that organic materials could be also used successfully for electronics and photonics. The new trends in physics and technology are the plastic electronics and plastic photonics. Most recent studies proved that in the next future the sand will be replaced by graphene. This talk presents a parallel and the equivalence between the electronics and photonics. Starting from electron in electronics, photon, solitons and plasmons in photonics, electrical cables – optical fibers, plasmonic wave guides, electrical circuits - optical circuits, electrical transistors – optical transistors, plasmonster, electrical generators – pulsed lasers and spasers, photonics gets step by step all the tools already existing in electronics. Solar energy could be converted in many ways, the most known is the conversion in electricity. Today we need that the energy is in form of electricity because most of the apparatus that we use are based on electricity: informatics, motors. However the progress in photonics with optical circuits, optical transistors, etc shows that the photonics informatics will be possible. Also the optical manipulation and optical engines concept were already demonstrated experimentally. If the laser propulsion will be achieved, and the optical engines will work, the question that will rise tomorrow is “Shall we still use the electricity in the future ? What will be the solar devices tomorrow ?

EFFECTS OF GROWTH PARAMETERS ON THE QUALITY OF MULTI-CRYSTALLINE SILICON GROWN BY A BRIDGMAN TECHNIQUE

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Because of its low costs and his similarities with directional solidification, Brigman growth of multi-crystalline silicon is a suitable technique for a fundamental study of the influence of growth parameters (like growth rate and temperature gradients, crucible coating) on interface shape, grains size and impurity distribution (carbon, oxygen), which are important parameters for the photovoltaic applications. In order to study the influence of growth parameters on the crystals quality different crystals were obtained using a Bridgman set-up. The set-up was designed to obtain materials of high purity and high melting points and was adapted for silicon solidification because of its large flexibility due to the existence of 4 heating elements protected by a special casing.

Numerical modelling (using CrysMAS software) have been performed in order to optimize the temperature field. A good agreement between numerical results and experimental data was found for the axial temperature profile.

Using a thin crucible (3cm diameter) several crystals were obtained for various growth parameters (pulling rate, temperature profile, crucible coating). Grain size analysis has been performed for all these crystals and it was found that the average grain size decrease when the pulling rate increase. The distribution of carbon and oxygen along the length of the crystals has been analyzed using IR absorbtion spectroscopy [1, 2]. It was found that due to the segregation process the concentrations are higher in the upper part of the crystals. The influence of growth parameters on interface shape and deflection was also studied.

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ZnO NANOPARTICLES INFLUENCE ON THE PHOTOVOLTAIC PERFORMANCES OF POLYMERIC BLENDS BASED ON MEH-PPV:PCBM

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Polymeric photovoltaic cells based on MEH-PPV:PCBM were prepared on optical glass and flexible substrates, respectively. ZnO nanoparticles were added on PEDOT:PSS solution, varying the concentration. All polymeric films were deposited by spin-coating technique, at room temperature. For all samples were performed electrical and photo-electrical measurements, at room temperature. Current-voltage characteristics were drawn both in dark and in AM 1.5 conditions. Parameters characterizing a photovoltaic cell were calculated and compared. Photoelectrical measurements indicate that the ZnO nanoparticles have a positive influence on the conversion efficiency, reducing the serial resistance of the structure and improving the stability of the cells.

Keywords: ZnO nanoparticles, photovoltaic cells

TRANSPARENT THIN FILMS FOR POLYMERIC PHOTOVOLTAIC CELLS

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Indium zinc oxide (IZO) thin films were prepared on optical glass substrates by pulsed laser deposition technique using targets with an In/(In+Zn)atomic ratio of 80%. A planetary ball mill was used to obtain a homogeneous mixing of the In_2O_3 (Aldrich, 99.9% purity) and ZnO (Aldrich, 99.99% purity) powders. IZO films were used as transparent electrodes in polymer-based poly(3-hexylthiophene) and 1-(3-methoxycarbonyl)-propyl-1-phenyl-(6,6)C_{61} (1:1) blend photovoltaic cells. Samples were morphological, structural, electrical and photoelectrical characterized and the results were compared with those obtained for similar structures having as transparent electrode indium-tin-oxide (ITO). The action spectra measurements revealed that IZO-based photovoltaic structures have performances comparable with ITO-based devices.

Keywords: IZO, photovoltaic cells, PLD

DYNAMIC SIMULATION OF IMPURITY TRANSPORT AND REACTIONS IN A BRIDGMAN FURNACE FOR DIRECTIONAL SOLIDIFICATION OF MULTI-CRYSTALLINE SILICON

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In directionally solidified multi-crystalline silicon (mc-Si) oxygen and carbon are the impurities that are present by far the highest level. Oxygen dissolves in the melt due to the dissolution of the silica crucible and evaporates by bonding with a silicon atom to SiO at the melt free surface. The argon gas flow carries the SiO to the graphite fixtures, where it reacts with carbon forming carbon monoxide (CO). Furthermore CO is carried to the melt free surface, where it dissociates into carbon and oxygen. Finally, the dissolved oxygen and carbon are incorporated into the crystal. Oxygen related defects, like thermal and new donors can reduce the minority carrier lifetime. If the solubility limit of carbon is exceeded, SiC precipitates can form locally in the melt and in the crystal, which can cause ohmic shunts in solar cells and result in nucleation of new grains in silicon ingots.

We present, for the first time, a dynamic coupled global simulation of mc-Si directional solidification in a VB furnace, considering the argon gas flow, melt flow, impurity transport and reactions for the full melting and solidification phase. Results from simulation are compared with experimental data, considering the impurity distribution in the ingot and CO gas concentration above the melt free surface.

Poster Presentations

THE SOLAR CELLS CdS-CdTe WITH CdO INTERMEDIATE LAYER

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Solar cells (SC) based on thin layers of CdS-CdTe have a greater prospect for widespread use in the energy sector. This is explained by the fact that the width of the CdTe band is Eg = 1,46 eV, which corresponds to a wider spectral window.

The purpose of this work is an attempt to improve the energy parameters of CS by introducing a thin dielectric layer between the layers of CdO CdS and CdTe.

It is well known that introduction of such a thin dielectric layer (to allow tunneling), increases the potential barrier height in the heterojunction, reduces considerably the influence of interface states in which the nonequilibrium carriers are lost, and accordingly improves the conditions for distribution of charge carriers.

Therefore, for this research we prepared samples in which with the same support was obtained heterojunction (HJ), with CdO layer and without it in the same technological conditions. For comparation we used two methods of production: The cuazi closed volume method and the hot walls method.

To assess the effectiveness of these samples V-A (Volt-Ampere) characteristics were measured at 100 mW/cm^2 lighting. The study of V-A and V-F (Volt-Farad) characteristics has allowed the quality parameters of the HJ and to investigate the space charge region.

Our research has shown that HJ containing CdO interlayer have better parameters compared with HJ which lacks the CdO interlayer; this fact is valid for both methods of obtaining SC.

In particular it is observed for the open circuit voltage (Uoc). In some samples this increase reaches 10%. However, upgrading of energy parameters takes place up to a certain thickness of oxide, since a higher thickness of the layer, makes it become nontransparent for carriers, tunneling is not possible and therefore Isc (short circuit current), Uoc, FF (Fill Factor) and η (efficiency) decreases significantly. Thus with the introduction of CdO, we have obtained the best evidence, where:

Isc = 20,45 mA/cm^2, Uoc = 0,842V, FF = 0,572 and η=12,3%.

MICROSTRUCTURAL AND PHOTOELECTROCHEMICAL PROPERTIES OF THERMALLY EVAPORATED SnS THIN FILMS

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In recent years, tin sulphide (SnS) has attracted the attention of many researchers as a potential candidate for application as an absorber layer in heterojunction solar cells. In the present investigation, the dependence of compositional, microstructural, and photoelectrochemical properties of SnS thin films has been studied. SnS thin films with a thickness of ~ 465 nm have been deposited on different substrates such as glass, ITO (indium tin oxide) and Mo-coated substrates by thermal evaporation using a constant rate of deposition of 5 Å/s. All the layers were nearly in stoichiometric elemental composition with a Sn/S ratio of ~ 1.01. The X-ray diffraction analysis revealed that all the films were polycrystalline in nature and is composed of crystals oriented predominantly in [111] direction with Herzbergite orthorhombic structure. SEM and AFM studies revealed surface morphological profiles, which had densely packed and well adherent to the substrate surface. The Raman spectra showed bands at 64, 163, 189 and 219 cm^{-1}, which corresponds to the single phase (SnS) composition of films. *p*-type conductivity of all the deposited films has been determined by the photoresponse studies. The high photoresponse of ITO/SnS films makes them appropriate for solar cell applications.

OPTICAL AND MORPHOLOGICAL PROPERTIES OF THERMALLY VACUUM EVAPORATED CdTe THIN FILMS FOR PHOTOVOLTAIC APPLICATIONS

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We obtained p – type semiconductor CdTe thin films by thermal vacuum evaporation (TVE) on ITO coated optical glass substrates for applications in solar cells technology. The optical characterizations of CdTe thin films were made by spectroscopic ellipsometry (SE) and optical spectrophotometry. The optical constants (refractive indices and extinction coefficients) were obtained for CdTe thin films of different thicknesses. High absorption coefficients of order 10^5 cm^{-1} and suitable optical bandgaps values of 1.5 eV were obtained, confirming the role of CdTe thin films as classic p – type absorber materials for photovoltaic applications. The surface morphology of the obtained films was investigated by atomic force microscopy (AFM) and by scanning electron microscopy (SEM) techniques. The influences of different post-deposition thermal treatments applied on CdTe thin films on their morphological properties were discussed.

ULTRASONICALLY ASSISTED HYDROTHERMAL SYNTHESIS OF NANO-STRUCTURED Bi_2Te_3 SEMICONDUCTORS FOR THERMAL ENERGY RECOVERY

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Nanocrystalline powders of Bi_2Te_3 with different morphology and particle size 30-40 nm were prepared by ultrasonically assisted hydrothermal method of bismuth chloride and tellurium in aqueous solution with different concentration of $NaBH_4$ as reducing agent at 200°C for 5 hours and 80% fill degree of autoclave.

The influences of precursors and hydrothermal treatment conditions on the formation features, phase composition, particle size, morphology and properties of the products were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDAX) and atomic force microscopy (AFM).

The paper reports a comparative study regarding the precursors influence and hydrothermal condition of obtaining nano-structured thermoelectric materials. It was found that hydrothermal processing result in formation of low dimensional dispersion of Bi_2Te_3 nanostructures with desirable form and size and high degree of crystallinity.

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STABLE ZnO:Al DOPED TRANSPARENT CONDUCTIVE LAYERS, FOR PHOTOVOLTAIC APPLICATIONS

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Transparent conductive layers have been investigated for photovoltaic applications. Thin films were obtained by sputtering depostion from our own prepared targets. The AZO-type layers were obtained in an Ar environment at 220^{o}C substrate temperature for 6h. AZO layers type were obtained with controlled thickness and a uniform distribution of grains on float glass. A series of samples were annealed in H_2 environement in order to improve the electrical conductivity. Layer thickness was determined using interferometric microscopy. A Current Imaging Tunneling Spectroscopy was performed using a STM microscope. The optical transmitance in visible domain was evaluated at average 80%. The I-V characteristic was drawn and measured in order to evaluate the resistivity of the layers which is around the value of 10^{-3} Ω m. Determination of crystalline phases was performed by X-ray diffraction. Investigation by scanning electron microscopy of the surface layers of transparent conductive ZnO: Al was carried out using FESEM-FIB.

FABRICATION PROCESS FOR HIGHLY EFFICIENT WITH LOW COST SOLAR CELL

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High efficiency with low cost is one of major key in photovoltaic industry for large scale acceptability. Cost of solar cell mainly depends on the thickness of wafers; if thickness could be decreased economical benefits could be obtained. Theoretical studies show that high efficiency could be obtained by using thin monocrystalline silicon wafers ranging from 120-150µm instead of 300 µm. Our aim of this project is to fabricate monocrystalline silicon solar cell ($n^+p\ p^+$) by using phosphorus/aluminium (P/Al) technology on 150µm thick wafers obtained by Czochralski process. It is expected 18% efficiency by using this technology. At this moment we are starting up the process installation and in near future we need to optimize some parameters such as absorption and reflection of light, surface recombination velocity and life time in volume of cells to get higher efficiency. In future we will apply this technology in industrial solar cell fabrication.

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CHEMICAL WET ETCHING OF AL DOPED ZNO THIN FILMS WITH NH_4CL

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In this paper we present the results of a study on chemical wet etching with an aqueous solution of NH_4Cl on Al doped ZnO (AZO) thin films, for solar cells application. Deposition of AZO films was made by radio f**requency** (*RF*) magnetron sputtering without heating method, using a not sintered ceramic target ZnO: Al_2O_3 (98:2% weight). Post-deposition chemical wet etching was performed with a aqueous solution 5% NH_4Cl at room temperature. The influence of AZO thin films by chemical wet etching on surface texture as well as morphology structural optical and electrical properties was systematically investigated. Samples with textured surface had 80% average transmission in in the visible range and *reflection* was *reduced* with 5%. The best results after etched, was obtained for 10 minutes in NH_4Cl 5.0 % solutions with a good conductivity ($\rho = 1 \times 10^{-3}$ Ω cm), mobility (μ=12.2 cm^2/Vs) and carrier concentration (n=5.40x10^{20} cm^3).

ON THE SURFACE PROPERTIES OF TITANIUM OXIDE THIN FILMS DEPOSITED BY SPIN COATING AND SPRAY PYROLYSIS

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Dye-sensitized solar cells (DSSC) are promising candidate for relatively low cost, simple manufacture and high-performance solar cells is. Typical DSSC is composed of three elements: a sensitized monocrystalline TiO_2 film, counter electrode and electrolyte. The TiO_2 layer is considered to be most important structural unit in the device. Different deposition techniques such as evaporation, doctor-blade, dip-coating, sputtering and spray pyrolysis were developed. In this paper we present by comparison the surface morphological and optical properties of titanium oxide thin films deposited on glass and ITO substrates by spin coating and spray pyrolysis using the same starting solution. AFM and SEM analyses were carried out to investigate the surface morphology of the films. New method of crating TiO_2 films divided by ‘channels’ into ‘blocks’ was reported. Optical properties and influence of annealing of layer were examined using spectrometry and ellipsometry methods. XRD measurements were performed and phase formation from amorphous TiO_2 to anatase phase deposited on glass and ITO substrates issues were discussed. Contact angle measurements proved TiO_2 hydrophilic properties after exposure to strong UV light.

FORMATION AND ANNEALING OF INTERSTITIAL DEFECTS IN IRRADIATED SILICON AND SILICON-GERMANIUM CRYSTALS DOPED WITH BORON

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The carrier lifetime in boron-doped oxygen-rich silicon is ultimately limited by light-induced boron-oxygen-related recombination centers, limiting the efficiency of solar cells fabricated on this material [1]. Those centers are thought to consist of a substitutional boron (B_s) atom complexed with an interstitial oxygen dimer O_{2i} [1]. However this opinion was criticized in [2] where it was suggested that not B_s but interstitial boron (B_i) atoms are crucial for the formation of recombination centers. The purpose of this paper is to study in more detail some electronic properties of boron-oxygen complexes formed under irradiation and their effects on charge carrier lifetime after irradiation and annealing at elevated temperatures. We have studied devices made from silicon and silicon-germanium. The latter one is suggested to be a material with very low light-induced degradation [3].

Using DLTS measurements we have found that the single silicon self-interstitial atoms can be kept essentially immobile in p-type materials upon irradiation with alpha-particles at room temperature. The relative probabilities of Si_i reaction with carbon and boron can be controlled by electronic excitation which allows changing the formation rate of B_i after similar irradiation doses in the same crystals. Annealing of B_iO_i results in the decrease of the carrier lifetime. This observation is consistent with the model suggested in [2].

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THE INFLUENCE OF REACTION TIME ON THE FORMATION PROCESS OF TIN TELLURIDE

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Binary compound based on SnTe has been prepared using hydrothermal route at different times, in aqueous solution at 200°C for 5 hours (S1), 10 hours (S2), and 16 hours (S3). The influences of reaction time on the formation features, phase composition, particle size, and morphology of the products were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDAX) and atomic force microscopy (AFM). The structural analysis revealed that the time of synthesis has influence on the purity and crystalline structure of achieved materials.

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ADSORPTION AND PHOTOCATALYTIC DEGRADATION MECHANISMS OF HEAVY METALS AND SURFACTANTS USING TiO_2/FLY ASH WITH MODIFIED SURFACE CHANGE

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The fly ash (FA) is a low-cost waste material with predominant oxide composition, thus with significant surface charge. Therefore, FA can be a good substrate in wastewater treatment for the immobilization of heavy metal cations, (Cd^{2+}, Cu^{2+}, Zn^{2+}, Ni^{2+}, Pb^{2+}), dyes and for surfactants removal. The properties of fly ash are strongly depending on the coal-burning, type of furnace and process in the power plant. For an up-scalable wastewater treatment process, a substrate with good adsorption efficiency and with relatively constant behavior is needed and modifying the fly ashes surface can be a feasible solution for reaching both goals. The adsorption process on the FA surface is linked with the FA composition, mainly with the aluminosilicates and unborn carbon content. The use of concentrated NaOH solutions was reported, targeting the aluminosilicates for modifying the FA at room temperature or in hydrothermal processes. Still, concentrated alkaline solutions represent an environmental threat. Therefore, alternatives must develop using less concentrated NaOH solutions for surface modification. Working with industrial wastewaters proved that there is a competitive adsorption process involving more pollutants for example heavy metal cations, dyes and surfactants.

The paper presents a comparative study of the heavy metals (Cd^{2+}, and Cu^{2+}) and (1- Hexadecyl) trimetylammonium bromide (HTAB) cationic surfactant and sodium dodecyl sulfate (SDS) adsorption on FA CPH- Brasov, modified with NaOH 2n solution and TiO_2. The aqueous surfactants were prepared below CCM. As reference, experimental adsorption isotherms were obtained for HTAB on modified FA (SiO_2 -partially hydrophilic, and Al_2O_3 -hydrophilic) at room temperature.

The adsorption kinetic, the substrate capacities and the mechanisms are further discussed correlated with the surface structure XRD, SEM, FTIR and morphology (AFM). The results indicate parallel adsorptions of heavy metals and surfactants from mixed solutions. The results show that, fly ash with controlled surface properties allows the simultaneous removal of heavy metals and surfactant, resulting waters that respect the discharge regulations.

PULSED LASER DEPOSITION OF CdTe/CdS THIN-FILM SOLAR CELLS

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Structures based on cadmium telluride (CdTe) represent promising solutions for thin-film solar cells. CdS/CdTe/ZnTe thin-films were deposited by Pulsed Laser Deposition (PLD) technique using a KrF* laser source, operating at λ=248 nm with τ_{FWHM} ~25 ns. In this study we investigated the influence of electrodes and interfaces on the solar cell performances. Other transparent conductive oxides, especially Indium doped ZnO (IZO) and Al doped ZnO (AZO), were used to replace the conventional SnO_2 in combination with different back-contact films (Ag+Pd, Mo, Cu). Buffer layers with mixed composition (e.g. IZO+CdS, AZO+CdS) were deposited to increase the electrical and optical performances. The deposition conditions were optimized to improve the stability of CdS/CdTe solar cells as a function of layer thickness and laser parameters. Thus, we observed a reduction of the stress and a better adhesion of CdTe, ZnTe and CdS films deposited at moderate laser fluences. Optical properties of the deposited structures were characterized by spectroscopic (UV–VIS, Photoluminescence, Ellipsometry) methods. Morphological features were investigated by Atomic Force Microscopy and Scanning Electron Microscopy. X-ray diffraction was used to analyze the crystalline structure of the PLD films. Current density-voltage *I-V* curves were taken under both dark and white illumination and the efficiency was calculated at AM 1.5

MODELLING OF THERMO-ELASTIC STRESSES DURING SILICON DIRECTIONAL CRYSTALLISATION ACCOUNTING FOR MULTI-CRYSTALLINE ORIENTATIONS AND ELASTIC ANISOTROPY

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Directional crystallization of multi-crystalline silicon ingots with subsequent wafer sawing is still the main method for producing solar cells. Significant efforts are, however, needed to improve the process towards higher material quality and further cost reduction. In particular, it is well known that defects such as dislocations diminish the material quality by reducing the minority carrier lifetime. These defects are generated at high temperature due among other sources to thermally induced stresses and deformations. The present work presents a 3D thermo-mechanical model and its application to the analysis of thermo-elastic stresses during the casting of square multi-crystalline silicon ingots. The analysis is carried out as part of a global (heat transfer and stress-strain) furnace model including all furnace components and applied under realistic casting conditions. The mechanical model accounts for the multi-crystalline structure of the ingot as the grains structure is input to the model. Each crystal is explicitly resolved in the finite element mesh. Grain size, morphology and orientation as well as elastic anisotropy are thus all taken into account. The model is used to quantify the consequences of the ingot structure on thermo-elastic stresses by comparing multi-crystalline and mono-crystalline under several crystal orientations. The model is also use to quantify the role of the grain orientations mismatch on intra- and inter-granular stresses.

SYNTHESIS AND CHARACTERIZATION OF UNDOPED AND Al AND/OR Ho DOPED ZnO THIN FILMS

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ZnO has been widely used in organic solar cells and hybrid solar cells due to its characteristics such as low cost synthesis, non-toxicity, excellent chemical and thermal stability and specific electrical and optoelectronic properties. The synthesis of undoped and aluminium and holmium doped and co-doped zinc oxide thin films on quartz, pyrex glass and monocrystalline silicon substrates was achieved using a simple aqueous solution deposition method. The effect of the precursor solution concentration on the morphological and structural characteristics and on the optical properties of undoped ZnO thin films was also investigated. The surface AFM investigations reveal that the films deposited on the quartz substrate present higher morphological characteristics. The room-temperature photoluminescence (PL) and the optical absorption (UV-Vis) properties of the as-obtained undoped and doped ZnO thin films were investigated, as well. A strong UV emission (380 nm) was recorded for the ZnO thin films deposited on quartz substrate.

Keywords: zinc oxide, thin films, aqueous solution, optical properties

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S7

Modelling and Characterization of Advanced Materials

Invited Papers

VERTICAL GRADIENT FREEZE OF 4-INCH GALLIUM ARSENIDE IN A HEATER-MAGNET MODULE

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Presently, industrial crystal growth is focused on the improvement of the process efficiency by minimization of the production costs without loss of the quality of substrates.

The vertical gradient freeze (VGF) is the established crystal growth technique for commercial production of semiconductors such as GaAs. To improve the crystal quality, an exact control of the melt flow during the whole growth process is of crucial importance. Introduction of traveling magnetic fields (TMF) into the melt has a great potential for beneficial melt/solid interface shaping, improved melt stirring etc.

Previously gained knowledge from VGF growth of 4 inch Ge crystals in a TMF field using a heater magnet module (HMM) helped developing a new 4 inch GaAs growth process.

The difference in the material properties between Ge and GaAs, particularly in their electrical conductivity posed a challenge for the development of GaAs process.

If GaAs crystals were grown under weak TMF ($B < 1$ mT), only several weak striations were noticed. In the same time, in a benchmark GaAs crystal grown in a pure buoyancy regime, i.e. without TMF, significantly stronger pronounced striations were obtained. This was the first evidence of the lowering of micro-inhomogeneity in GaAs due to the enhanced magnetic stirring. The morphology of the interface can be controlled by TMF very precisely. Temporal change of TMF parameters is required to obtain constant interface morphology over the whole growth run. The successful GaAs crystal growth in the VGF set-up equipped by HMM was feasible only if precisely defined temperature and Lorentz force density fields in the melt were provided. Therefore, a numerous preliminary 3D CFD global simulations of the whole furnace were performed.

THERMODYNAMICAL ANALYSIS OF POLYTYPE STABILITY DURING PVT GROWTH OF SiC USING 2D NUCLEATION THEORY

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SiC is a promising material for devices operating at high temperatures, high power and high frequency. The physical vapor transport (PVT) method has been most successful and common method for growth of bulk SiC crystals. SiC is known to exist under a wide variety of polytypic forms more than 200 which have different characteristics and the requested polytype is different depending on a device. Furthermore, the several different polytypes are easily formed in a crystal during crystal growth and the mixing of the polytypes destabilizes growth and deteriorates the characteristic of SiC as an electronic material. Therefore, it is important to control the formation of desired polytype. However, reliable explanation of the polytypic control has not been reported so far. Several growth parameters such as the growth temperature, pressure in a furnace, supersaturation, vapor-phase stoichiometry, impurities and polarity of seed surface have been discussed to influence the polytype stability. Thus, the control of polytype is a complex problem. In this study, we focused attention on the nucleation stage of crystal growth and dependence of process parameters, such as temperature of a seed, pressure in a furnace and surface polarity of a substrate, on formation of specific polytypes of SiC in a process of PVT. The analysis was based on a classical thermodynamic nucleation theory reported by Fissel in conjunction with numerical results obtained from global model reported elsewhere.

We studied dependence of process parameters, such as temperature of a seed, pressure in a furnace and surface polarity of a substrate, on polytypes of SiC in a process of physical vapor transport. The analysis was based on a classical thermodynamic nucleation theory in conjunction with numerical results obtained from a global model. We investigated which polytype was more stable in the nucleation stage by a comparison of nucleation energies of each polytype. The results show that the formation of 4H-SiC was more stable than that of 6H-SiC when we used C-face SiC as a seed. Furthermore, the most stable polytype could change from 4H-SiC to 6H-SiC in a condition of higher supersaturation, with a condition of higher temperature of a seed and lower pressure in a furnace. Meanwhile, the formation of 6H-SiC was more stable than 4H-SiC when Si-face of a seed was used.

NUMERICAL STUDY OF MELT CONVECTION IN DIRECTIONAL SOLIDIFICATION METHOD OF MULTICRYSTALLINE SILICON

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Multicrystalline silicon is widely used in photovoltaic industry. A key point is to increase the ingot quality during the growth process in order to increase the solar cells efficiency. With the increase of computational power, numerical simulation has become a powerful tool in the process of optimization the crystal growth technologies. It is the aim of this contribution to look closer at the influence of the temperature gradient in melt and growth rate on the melt convection and interface shape in a pilot furnace for directional solidification of multicrystalline silicon. In order to get a basic understanding of these influences, computational code STHAMAS3D was used to compute the heat and mass transport in a local three-dimensional geometry which consists of melt and crystal. It was found that growth parameters like temperature gradients and growth rate strongly influence the melt flow and interface shape. The results indicate that both interface deflection and melt flow structure are sensitive to the variation of considered growth parameters. The regularity of the melt flow pattern increase with the increase of temperature gradient in the melt. The influence of the growth rate is somehow similar, the regularity of the flow patterns increases with the increase of the growth rate. The interface shape is slightly convex for the smaller growth rate $v_t = 5$ mm/h and concave for $v_t = 10$ mm/h and $v_t = 15$ mm/h.

The geometry of the crucible is also a factor which influences the symmetry of the flow patterns. It was found that all flow patterns studied here have a symmetry axis along the Axy direction. Also the other symmetry axes which characterize the regularity of the flow are symmetry axes for the crucible geometry.

THERMODYNAMIC AND KINETIC PROPERTIES OF NON-STOICHIOMETRIC NUCLEAR FUELS

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Thermodynamic properties of fluorite-structure materials such as CeO_{2-x}, PuO_{2-x}, UO_{2+x} strongly depend on temperature and oxygen content (deviation from stoichiometry, x). For instance, thermal conductivity significantly decreases with both temperature, T and x, while oxygen diffusivity increases with x. The change in the oxygen diffusion coefficient can be one or two orders of magnitude as x approaches 0.2. Since experimental information is scarce, or hard to directly be interpreted in terms of deviation from non-stoichiometry, atomistic models coupled with statistical methods are used to provide an improved understanding of the role of non-stoichiometry on defining thermodynamic properties such as free energy, enthalpy and entropy of fluorite oxides. A similar approach is used to develop models of oxygen diffusivity as a function of temperature and nonstoichiometry. To highlight the capabilities of our approach, we provide examples and results in a fairly good agreement with the few available experimental points used to validate the models. Moreover, the present work extends the predictions of previously published approaches, and predict properties for $300 < T < 1000$ K and $0 < x < 0.2$.

SIMULATION OF EPITAXIAL GROWTH: DENSITY FUNCTIONAL THEORY AND MONTE-CARLO METHOD

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In the first part we will present a Monte-Carlo method adapted to the layer growth of perovskites. The specific structure is taken into account. Extended density functional theory (DFT) studies have been performed for the adsorption of oxygen and water on $SrTiO_3$. The results exhibit that the adsoprtion of an oxygen molecule on a perfect surface is unlikely. but it can perfectly stick to an oxygen vacancy at the surface. Some preliminary results of kinetic Monte-Carlo simulations will be shown.

In the second part we will present a modification of the Monte-Carlo method for taking into account the elastic strain and its effect on the surface diffusion. This method has been tested for the layer growth of III-V-compounds, namely $In_{1-x}Ga_xAs$ on GaAs. We computed the critical layer thickness as a function of the In/Ga ratio of the incoming flux and found a good agreement with experimental data. Issues of island growth, wetting layer, and pyramids' shapes will be discussed.

COMPUTER SIMULATIONS OF MATERIALS UNDER EXTREME CONDITIONS: APPLICATION TO NUCLEAR REACTOR MATERIALS

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Important and intriguing phenomena occur in materials at various time and length scales, from femtoseconds to seconds or days and from angstroms to meters. In nuclear reactor materials, irradiation effects, phase transformations and deformation make the problem even more complex. A variety of ‘at-scale’ experimental, theoretical, and computational tools are available for narrow length and time intervals, as imposed by the limitations of individual techniques. In the experimental domain, these needs are fulfilled by high resolution electronic microscopy and intense gamma-ray sources that couple high-energy particle accelerators and high-power lasers. Moreover, multi-scale computer simulations can expand the investigation domain and provide information in extreme conditions that are not currently accessible to experiments. In the presentation, computer simulation results are used to illustrate trends, challenges and opportunities, as part of a proposed strategy to closely integrate experimental, theoretical and computational approaches.

THE KEY PROPERTIES IN CHALCOGENIDE SYSTEMS AND THEIR RELEVANCE ON ELECTRICAL SWITCHING

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The binary and ternary chalcogenide compositions show properties of switching that can be used in electrical memories.

It is shown that in chalcogenide systems, as a function of mean coordination number, the physical properties exhibit sharp modifications. It is discussed the chemical threshold, the topological threshold and the intermediate phase in these systems.

Careful investigations show that the position of the Intermediate Phase (IP) corresponds exactly to borderline of the glass formation domain. In this narrow domain are situated the best compositions with switching properties.

Based on these considerations it is possible to develop a method for identification of the compositions to be prepared for switching applications.

Finally, a structural explanation of the IP is given.

MAGNETO-TRANSPORT MEASUREMENTS AND MICROMAGNETIC SIMULATIONS ON SPIN-VALVE STRUCTURES FOR PLANAR HALL EFFECT SENSORS

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In this work we present magneto-transport measurements and micromagnetic simulations performed on spin valve structures used for low-field magnetic sensors based on the planar Hall effect (PHE). These sensors are interesting for field measurements and angular positioning devices due to a high magnetic sensitivity and the reduction of temperature drift by at least four orders of magnitude compared to a similar longitudinal magnetoresistive detector or even a GMR sensor. Disk shape structures like $Ni_{80}Fe_{20}$(15.5 nm), Si/SiO_2/Ta(5 nm)/$Ni_{80}Fe_{20}$(10 nm)/Cu(2.5 nm)/$Ni_{80}Fe_{20}$(6 nm)/$Fe_{50}Mn_{50}$(8nm)/Ta(5nm), Si/Ta(3 nm)/$Fe_{50}Mn_{50}$(11 nm)/Co(7 nm)/Cu(3 nm)/$Ni_{80}Fe_{20}$(10 nm)//Ta(3 nm) and Si/Ta(3 nm)/Co(30 nm)/Cu(7 nm)/$Ni_{80}Fe_{20}$(70 nm)/Ta(3 nm), 5 mm diameter, were deposited using a UHV Magnetron Sputtering ATC2200 AJA machine. The magnetization curves and magneto-transport properties of these samples were measured using a 7T Mini Cryogen Free Measurement System from Cryogenic. From planar Hall effect (PHE) measurements made on these samples we obtained sensitivities up to 0.11 $\mu V/(Am^{-1})$ for a driving current of 10 mA trough the samples. We observed that we can tune the sensitivity and field range for which the PHE is linear in response by applying biasing fields, DC or AC, with well-established values. Micromagnetic simulations were performed to explain the dependence of the field sensitivity of the PHE voltage on the biasing field. Also, the field and angular dependences of the PHE voltage were simulated. Some applications for field measurement, rotation sensor, contactless potentiometer and detection of the fields generated by super paramagnetic beads are discussed.

Oral Presentations

CALCULATIONS OF THERMALLY STRATIFIED, LORENTZ FORCE GENERATED TURBULENT FLOW IN SQUARE CRUCIBLE

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Directional solidification (DS) is widely used for the production of photovoltaic materials in crucibles, usually square-shaped, with different sizes. The quality of the ingot depends on the success of each production step, two of which – melting and solidification – include the liquid phase. Understanding and controlling of the melt flow during solidification phase is important for the reduction of incorporations and precipitation formations. Due to lack of experimental techniques for high temperature media, numerical simulations are the most powerful tool for the investigation of mass exchange processes.

Numerical research of melt motion problem in crucible implies the solution of fluid flow and heat transfer, and electromagnetic (EM) equations when EM stirring is present. However, if the flow is turbulent, a modelling approach has to be used. Two most well known approaches for turbulence modelling are Reynolds Averaged Navier-Stokes (RANS) and Large Eddy Simulation (LES), widely used also in modelling of melt flow in DS method.

This study is devoted to the numerical investigation of the turbulent melt motion in a square crucible where the flow is created by Lorentz forces generated by external AC magnetic field. As a strong vertical thermal gradient is present in DS method in melt, stratification effect takes place and motion in vertical direction is damped by buoyancy forces. This leads to anisotropic turbulence with pulsations in vertical direction being significantly weaker than in other two directions. Simulation results shows that LES with reasonable spatial discretization is able to capture this effect, however RANS results show underrated vertical temperature gradient which is important for growth rate estimation. In situations with Richardson number close to unity RANS models have problems also with correct estimation of mean velocity field. Experimental validation is also expected.

NUMERICAL SIMULATION OF DISLOCATION GENERATION: SELF-DEVELOPPED VERSUS COMMERCIAL SOFTWARE

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In the field of silicon for photovoltaic application, the crystalline quality of the wafers impacts strongly the photovoltaic properties. Among the defects responsible for the decrease in energetic conversion efficiency, dislocations have an intrinsic activity due to the effect of dangling bonds. More important, they also have a strong indirect effect associated to the formation of oxygen precipitates in their neighborhood and through the trapping of metallic impurities that both are often efficient minority carrier recombination centers.

In order to study generation of dislocation in silicon, several numerical simulations have been done by various teams and laboratories developing their own software using the finite element method. These simulations can generally only be applied to a single furnace and require an expertise in numerical modeling to be safely used. However the question of the capacity of commercial codes to handle this problem is still open.

In this work a numerical simulation is performed, based on the commercial software Comsol, in order to predict the dislocation density in Si ingots. The model is 2D-axisymmetric and considers the silicon as an isotropic material. This simulation solves the Alexander-Haasen model and uses as raw data only the geometry of the furnace and time-dependent temperature maps all along the solidification progress.

The purpose of this paper is to compare the results obtained with COMSOL to those obtained by Prof. Kakimoto's and Prof. M'Hamdi's teams in the simulation of dislocation multiplication by their own software. The input data used in their previous articles [1-2] were taken for comparison.

It is shown that the results strongly depend on the choice of the physical parameters used for the Alexander-Haasen model and on a careful description of the geometry of the problem. The implementation and optimization of the model in COMSOL is possible but long and difficult. It allows obtaining results qualitatively comparable to those obtained by the home-made codes, but reaching a semi quantitative agreement further necessitates a long adjustment of numerical parameters and procedures.

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ATOMISTIC SIMULATION OF DEFECTS IN STRAINED THIN FILMS

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We developed a Kinetic Monte Carlo (KMC) computer program (NUKIMOCS) which enables atomistic simulation of mechanical processes which occur at the nanoscale in thin films. In this paper we present the results of the KMC studies on dislocation and crack formation in a number of systems such as strained silicon and superlattices.

The code performs off-lattice KMC calculations to determine the strain in a material at the atomic scale and how this changes with time. Off lattice KMC allows atoms to occupy any position in space as in a real atomic lattice with interaction between atoms defined by interatomic potentials. Therefore, both the elastic and kinetic properties of an off-lattice KMC model are entirely defined by the interatomic potential. The underlying physics of strain-induced microstructural evolution can therefore be presented accurately and on meaningful time and length scales.

The parameters of the interatomic potentials have been determined by fitting the energy surface obtained from first principles calculations, the elastic constants and the coefficient of thermal expansion to the unit cell. Different initial and boundary conditions were applied in order to study the behaviour of defects such as vacancies and dislocations.

Our KMC modelling shows that an atomically smooth surface under tensile strain will spontaneously develop a surface roughness to minimise the energy of the system and crack nuclei can develop from the roughness profile generated. The most important relaxation processes are dislocation generation and propagation.

ANALYTICAL HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY CHARACTERIZATION OF MULTILAYERED COATINGS FOR ARTIFICIAL MULTIFERROICS

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The electrical and magnetic properties of multilayer coatings for artificial multiferroics are influenced by their structure and morphology. Analytical High-Resolution Transmission Electron Microscopy (HRTEM) characterization has been performed on multilayered $Pb(Zr_{0.2}Ti_{0.8})O_3/CoFe_2O_4/Pb(Zr_{0.2}Ti_{0.8})O_3/SrRuO_3/SrTiO_3$ structure. Thin films have been grown by pulsed laser deposition on a $SrTiO_3$ (001) single crystal substrate. Investigations of the interfaces between the layers and of the structural defects in this sequence have been performed by HRTEM and STEM (Scanning Transmission Electron Microscopy) techniques. SAED (Selected Area Electron Diffraction) patterns reveal the epitaxial growth of the layers on the $SrTiO_3$ (001) substrate. STEM – EDS (Energy Dispersive Spectroscopy) mapping has been used to show the 2-D distribution of the chemical elements in this multilayered structure. The study of strain field effects on the local structure of the thin films has been performed by advanced quantitative processing of the HRTEM images using the Geometrical Phase Method (GPM).

STRUCTURAL, ELECTRONIC, MAGNETIC AND DYNAMICAL PROPERTIES OF THE CUBIC SPINEL $LiMn_2O_4$

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We have performed an ab initio study of structural, electronic, magnetic and vibrational properties of the cubic spinel LiMn2O4. The calculations have been carried out by employing the density functional theory, a linear-response formalism, and the plane-wave pseudopotential method. The calculated structural parameters for this material compare very well with previous experimental and theoretical results. Our value of the average magnetic moment per Mn atom, 2.5 μ_B, is a bit smaller than the experimental value of 3.2 μ_B [1]. The electronic band structure of the cubic spinel LiMn2O4 shows the metallic character because there is considerable amount of overlap of bands close to the Fermi energy. Using our structural and electronic results, the phonon spectrum and phonon density of states for this material have been calculated and discussed. Our calculated zone-centre phonon frequencies accord well with previous experimental and theoretical results [2].

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INFLUENCE OF THE VIBRATIONS ON THE THICKNESSES OF BOUNDARY LAYERS DURING MATERIALS PRODUCTION FROM THE MELTS

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This paper summarizes the results of mathematical modeling of convective heat transfer and analysis of influence of the vibrations on the boundary layers of melt flow during materials production from the melts and on the shape of melt/crystal interface during crystal growth by techniques Bridgman, Czochralski and floating zone. The analysis of the influence of the vibration was done by basing on numerical calculations of Navier-Stokes and heat transfer equations and also comparison of temperature and velocity fields for cases with vibrating effects and without vibration for Space and normal gravity conditions. The calculations are carry out as for semi-conductor materials (with Prandtl number Pr<<1), and for oxides or water (Pr>1).

The numerical simulation of convective heat transfer for a cases of creation of vibrations by submerged vibrator or crystals (a position change due to vibrations under the harmonious law as $x = A\sin(2\pi\, f\, t)$) are executed. For technique of a floating zone the results of simulation in view of real shape of a liquid zone with the account and without the account of the following factors: radiations, rotation, vibrations, gravitational and Marangoni convection are simulated.

Results of numerical simulation are shown that the vibrations can intensify of heat- mass transfer and mixing of the melt, and also to reduce dynamic and temperature boundary layers.

The results of numerical simulation have shown that the vibrations can made the shape of front of crystallization flatter.

MICROMAGNETIC INVESTIGATION OF THE CURRENT INDUCED MAGNETIZATION OSCILLATIONS FOR A PERPENDICULAR POLARIZER SPINTORQUE OSCILLATOR

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Controlling the magnetization state of a small ferromagnetic element by a spin-polarized current flow rather than by an external magnetic field had attracted large interest for future high-performance spintronic products, such as microwave spintorque (ST) oscillators or magnetoresistive random access memories (MRAM). In 1996 it has been theoretically predicted [1] that in a magnetoresistive cell of type *magnetic thick layer/nonmagnetic spacer/magnetic thin layer* the conduction electrons are spin polarized by the first magnetic layer (polarizer), mostly maintains this polarization as it passes through the non-magnetic spacer and interacts with the second magnetic layer (free layer). This interaction results in a transfer of spin angular momentum from the conduction electrons to the local magnetization which is equivalent to a torque acting on the magnetization. Fast reversal of the magnetization or oscillations with frequencies in the gigahertz range can be generated in this way only through a spin-polarized current.

For ST oscillators different geometries, materials and magnetic configurations were investigated, including nanocontacts or nanopillars, spin-valves or magnetic tunnel junctions, polarizing layers having planar or perpendicular to plane magnetization, single or synthetic antiferromagnetic free layer. In this context, we perform a micromagnetic study in order to investigate the magnetization dynamics of a perpendicular to plane magnetization polarizer ST oscillator. The simulations are done by solving the Landau-Lifshitz-Gilbert (LLG) equation [2], which describes the motion of a magnetic moment under the influence of the effective field, augmented by the spin torque term [1].

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ORGANOMETALLIC COMPOUNDS AND THEIR APPLICATIONS

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Phosphorescent properties of organometallic compounds are based on Metal-to-ligand charge transfer (MLCT) and recently we propose a new compound WITH TWO TYPES of ligands (phenylpiridine and quinoline) which gives two colours (green and red). Absorption and photoluminescence spectra are studied for the neat film and films of $IrQ(ppy)_2$ –5Cl doped in 4,4′-N,N′-dicarbazolebiphenyl and polystyrene, together with the electroluminescence spectra using multi-layer light emitting devices. Emission bands are observed at 502 and 660 nm, which arise from ppy and Q ligands, respectively.

Electronic states and their energies are calculated for a mixed-ligand Ir(III) compound, $IrQ(ppy)_2$ -5Cl using time-dependent density function theory (TD-DFT) calculations and compared with experimental result. The $d\text{-}\pi_Q^*$ molecular orbital transition gives the lowest-energy triplet state absorption band. Its energy is estimated at 1.84 eV (671 nm), which is close to the absorption band position of 1.86 eV (666 nm) observed for $IrQ(ppy)_2$ -5Cl.

Mechanisms of the charge transfer between Ir 5d and C 2p orbitals were discussed, concerning the hybridizations and metallic character between Iridium and the two ligands.

Comparison between electroluminescence and photoluminescence of $IrQ(ppy)_2$ –5Cl (6%wt) dispersed in CBP were presented in a typical devices were configured as ITO/PEDOT:PSS or NPB (40 nm)/Ir-compound (6%) doped in CBP (10 nm)/ BCP(6 nm)/Alq_3 or TPBI (40 nm)/LiF (1 nm)/Al (150 nm).

The electroluminescence measurements allow us to conclude the possibility of obtaining dual emitters OLEDs with low energy consumptions due to the low voltage and currents regimes in which these devices can work.

COMPUTATIONAL PREDICTION ON DROPLETS FORMATION IN MICROCHANNELS

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The present microfluidic study is emphasizing the flow behaviour of two immiscible fluids within a Y shape microbifurcation with two inlets and one outlet. We report here a numerical investigation on the interface shape dynamics manifested at different capillary numbers (*Ca*); by a variation of the flow rate ratios (FRR) over the inlets. The immiscible fluids used for the investigation are two well characterized Newtonian fluids: mineral oil and deionised water, each being introduced in the Y-bifurcation inlets as separate fluids. An important feature from the physical characterization of the working fluids is represented by the interfacial tension, value measured with a goniometer and taken into consideration in the numerical model. The numerical results are compared with experimental investigation, performed with a special design setup based on an optical microscopic device (a CCD camera was coupled at the objective of an inverted microscope). Using this setup, direct visualizations and quantitative observations were obtained. The commercial microchannel presents a squared cross-section of 300x300 μm and was fabricated of cyclo-olefin copolymer, material with good optical property and chemically inert with the working fluids. The experimental data were compared with numerical simulations performed with commercial computational code FLUENT™ in a 3D geometry with the same dimensions as the experimental one. The flow domain has been discretized using a structured mesh containing 1,043,370 hexahedral finite elements. Being a multiphase flow, a Volume of Fluid (VOF) model has been employed. The numerical flow patterns are found to be in good agreement with the experimental manifestations. We demonstrated numerically and experimentally that the *Ca* variation has an important impact on the droplet frequency formation. Changing only the FRR of the inlets, the droplets behaviour modifies drastically. From numerical point of view the interface shape is used in characterise the manifestation of the droplet formation. Another available analysis of the droplets behaviour is the velocity and pressure distributions inside the droplet, which offers useful insights of internal flow.

Poster Presentations

MOLECULAR ORIGIN OF THE SECONDARY RELAXATIONS FROM COOPERATIVE AND NON-COOPERATIVE MOTIONS IN DIELECTRIC SPECTROSCOPY OF ALICYCLIC POLYIMIDES

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Two polyimides, poly(BOCA-ODA) and poly(BOCA-p-BAPS), obtained from an alicyclic dianhydride - bicyclo[2.2.2]oct-7-ene - 2,3,5,6 - tetracarboxylic dianhydride (BOCA) and an aromatic diamines 4,4’- oxydianiline (ODA) or 4 - (4 - ((4 -(4 - aminophenoxy) phenyl)sulfonyl)phenoxy (p-BAPS) were analysed by dielectric spectroscopy. The AC-dielectric properties, such as dielectrical constant, ε', and dielectric loss, ε'', were measured in the -200 $\div$ + 250 °C temperature and 1 Hz - 1 MHz frequency ranges. Spectrum results allowed evaluation of relaxation times, correlated with maximum frequency for dielectric relaxation, and of activation energies of γ and β relaxation. It can be concluded that the γ process corresponds to isolated motions occurring without cooperativity. Non-cooperativity confirms its origin in the rotation of lateral groups which does not lead to conformational change in the rest of the chain. This is consistent with the negligible entropy contribution and an essentially enthalpy contribution associated with the relaxation phenomenon. In contrast, the relatively high apparent activation energy of the β relaxation indicates a large entropy contribution which involves motions of segments of the main chain. The cooperativity associated with β relaxation process corresponds to the intramolecular and intermolecular contributions. These evaluations explain the influence of polyimide chemical structures on the temperature domains and on the activation energy of relaxation processes. Thus, γ and β relaxations are situated at higher and lower temperatures, respectively, and their activation energies are higher and lower values, respectively, for poly(BOCA-p-BAPS) comparatively with poly(BOCA-ODA).

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ON THE DETACHMENT OF SI INGOTS FROM SiO_2 CRUCIBLES

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In a Directional Solidification furnace, ingots of silicon are contained in a silica crucible covered with a silicon nitride (Si_3N_4) powder layer. During the crystallization of the ingot, the layer may suffer various behaviors. It can efficiently act as a mechanical shunt between the Si and the silica wall. When the filling of the crucible with silicon pieces is not done properly, it can also be scratched locally, resulting in deleterious adhesion between ingot and crucible at some particular locations. It can also be too thin, or insufficiently pyrolyzed, so that the Si interacts with the silica crucible.

A numerical study of these behaviors has been undertaken with the COMSOL Multiphysics software in a 2D-axisymmetric geometry, for isotropic GaSb attached to a SiO2 crucible and validated by comparison of experiment [1].

An extended simulation for Si has been carried out, taking into account the thermo-elastic stresses induced by the difference in thermal expansion coefficients of the crucible and ingot materials. It considers the multiplication of dislocations inside the ingot. Indeed, dislocations in a crystal are a way to reduce the stresses in the material by plastic relaxation. The Alexander and Haasen model could not be introduced because the very high stress gradient at the sticking triple line prevents numerical convergence. In order to consider plasticity, the stress is limited to σ_{CRSS} (Critical Resolved Shear Stress). Finally the model compares the remaining elastic energy to the adhesion energy between the silicon and the crucible, in order to predict the temperature at which the Si detaches from the crucible in case of sticking, and the zones where plasticity occurs in the ingot.

Various configurations are compared in terms of amount of stresses and dislocations occurring in the ingots. Moreover the visco-elasticity of the silica crucible at the melting temperature of silicon must be examined and is presently under development in the model.

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STUDY OF NANOSIZED ZINC FERRITE PREPARED BY SOFT MECHANOCHEMICAL SYNTHESIS

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The purpose of this work is a synthesis of nanosized zinc ferrite - $ZnFe_2O_4$ and to study their structural, magnetic and electrical properties. $ZnFe_2O_4$ have been prepared by a soft mechanochemical route from mixture of $Zn(OH)_2$ and $Fe(OH)_3$ powders in a planetary ball mill for varying duration. Soft mechanochemical reaction leading to formation of the $ZnFe_2O_4$ spinel phase was followed by X-ray diffraction (XRD), Raman and infrared spectroscopy (IR), scanning (SEM) and transmission microscopy (TEM). The spinel phase formation was completed after 18h of milling. The synthesized $ZnFe_2O_4$ ferrite has a nanocrystalline structure with a crystallite size of about 15nm. There are five Raman and four IR active modes. Mössbauer spectroscopy studies implied on the possible cation distribution between the tetrahedral and octahedral sites in formed spinel structure. The magnetic properties of the prepared spinel ferrite were also studied. Super paramagnetic behaviour was confirmed. The electrical DC/resistivity/conductivity of the sintered sample was measured in the temperature range 298-473K on a Source Meter Keithley 2410. Impedance measurements were carried out in the frequency range 100Hz to 1MHz on a HP-4194A impedance/gain-phase analyser using a HP-16048C test fixture in the temperature range 298-423K. For analysis of the relaxation mechanism the sintered $ZnFe_2O_4$, we were used the complex impedance spectrum of which explains what kind of dielectric relaxation exists in the frequency-dependent response of the samples.

A ONE DIMENSIONAL MODEL APPLICABLE TO FIND THE ELASTIC CONSTANTS

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The present work describes a range of theoretical and experimental studies on the binary and ternary solid elastic systems, with the aim of outlines a method for determination of elastic constants of certain materials, for example small samples which not allow individual resonance measurements. Although the spatial arrangement seems to be the same arrangement of Hopkins, there are major differences between, because if the Hopkins bar experiment implies measurements as in an acoustic tube, in our work the approach is based on a modal analysis of the embedded elastic system. The idea of the work stems from the properties of the intrinsic transfer matrix, which correlates his real eigenvalues with the frequencies of eigenmodes of the elastic system. An experimental modal analysis combined with a numerical method permits the estimation of the phase velocity and, properly, the elastic constants. Although the method can be generalized for a system build up from many materials, for practical reasons is better to use only simple systems like binary or ternary. A laborious study of the errors, shows that the errors are much smaller in the case of ternary systems and also strongly depends on the dispersion and on a better specification of the velocity in the gauge material. Method can be extended by considering also the attenuation.

GLOBAL, LOCAL AND ELECTRONIC STRUCTURE OF SUPPORTED NANOSTRUCTURED Ni-Cu ALLOYS

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The Ni-Cu nanoclusters as supported catalysts were analyzed by X-ray Diffraction (XRD) and X-ray Absorption Spectroscopy (XAS) in order to determine their global, local and electronic structure. Six samples with different Ni-Cu concentrations supported on SiO_2 were prepared by coprecipitation method at room temperature. XRD measurements evidence the Ni-Cu alloys formation with a lattice parameter varying from ~3.52 Å to ~3.58Å depending on the relative Ni-Cu concentration. The (111) peak corresponding to the investigated alloys phase with intermediate lattice parameter appear between Cu and Ni peak positions. This shifting in the (111) peak compared to nickel and copper peaks in metallic state indicates the Ni and Cu solid solution formation [1]. The average particle size, the mean squares of the microstrain, the particle size distribution and microstrain functions of the supported Ni-Cu nanoclusters were determined by XRD method using Generalized Fermi Function for the X-ray line profiles approximation. The average crystallites sizes differ from ~7 nm to ~16 nm depending on relative Ni-Cu concentration. The present study has pointed out a strong deformation of the local structure of the alloys, due to small particle size effects.

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PREPARATION AND STRUCTURAL CHARACTERIZATION OF RAPIDLY SOLIDIFIED Al-Cu ALLOYS

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Rapidly solidified Al_{100-x}-Cu_x alloys (x = 5; 10; 15; 25, 35 wt %) have been prepared and analysed. The increased cooling rate determines an expansion of cooper solubility in α-Al. In order to simulate the influence of cooling rate on the Cu solubility extension in Al, the pouring were performed in metallic die and by melt-spinning technique. The set-up of standard laboratory melt-spinning device has been modified for the present study, allowing further increases of the cooling rate. Obtained melt-spun ribbons have thicknesses range from 30 to 100µm. The value of cooling rate (v) for modified melt-spinning device was estimated based on the relation [1]: $d = A \cdot v^{-n}$, where: d- secondary dendrite arm spacing (SDAS); A and n are constants. The cooling rates provided by melt-spinning were within 10^5-10^6 K/s after the calibration of the device. The processed alloys were analyzed by OM, SEM, DSC, DIL, X-ray diffraction methods and by Vickers micro-hardness determination. The best mechanical characteristics were obtained for Al-35 wt% Cu alloy, achieving an average value of 238 HV for casting in metallic die. For the same alloy, but obtained by melt-spinning method, the hardness value increases by 45% due to the extended solubility of alloying element in the Al matrix.

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RESEARCH ON THE INFLUENCE OF THE COOLER NATURE ON THE SUPERFICIAL LAYER OF GRAY CAST IRON CASTINGS

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The structure and properties of gray iron castings is considerably influenced by the solidification cooling rate. In practice, to get gray iron castings with a hard surface layer, resistant to wear in molds of molding sand metal coolers are applied. This is the case of cleats, camshafts, drive shafts, rolling cylinders, etc.. Therefore, cooling rate, the velocity gradient at the surface of these parts, structure and properties obtained in the surface layer, the thickness of this layer depends on the thermo physical characteristics of the cooler used. Normally in practice iron coolers are used. The paper presents the results of research performed by authors on the influence of cooler nature on these parameters. The research was conducted using two methods: by computer solidification simulation and casting experimental samples. The solidification of cast samples was studied in six cases: no cooler, in the presence of cooler of iron, steel, copper, titanium and aluminum plated with steel (on the contract area with the sample). We studied the influence of cooler nature on the structure obtained in the surface layer, thickness of the hardened layer, hardness, temperature field and cooling speeds, hot spots positions. There are revealed the conditions in which the researches were done. The results are presented with a large number of micrographs, tables and graphs. There were established conclusions on this influence and the possibility to apply in the industrial practice.

ELECTRONIC AND TRANSPORT PROPERTIES OF FERROCENE DICARBOXYLIC ACID

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We present a study of the electronic transport properties of the 1,1=-ferrocene dicarboxylic acid sandwiched between Al(111) electrodes based on the density functional theory and Non-equilibrium Green functions. The transmission spectra and the current - voltage characteristics of the metalmolecule-metal system are computed for various two-terminal device models . The relation between the electronic structure of the molecule and the transport properties is thoroughly investigated. We found that the current - voltage characteristics are asymmetric, spin independent, and vary with the anchoring structure of the molecule to the electrodes. A fine-tuning of the molecular conductance can be easily achieved by applying a gate potential. Interestingly, a spin-polarized current can emerge as a consequence of the gate potential with the relative contribution of the two spin channels varying with the bias

DIELECTRIC RELAXATION IN YbF_3-DOPED AND NaF CO-DOPED CaF_2 CRYSTALS

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YbF_3 doped CaF_2 crystals are used as laser active media due to well-known good mechanical, thermal and optical properties of the fluorite host. It is known that certain fraction of Yb^{3+} ions can be reduced to divalent state by various methods [1] or directly in the as-grown crystals using high deoxidization growth conditions [2].

The goal of this work is to study the influence of Na^+ ions on the dielectric spectra of YbF_3-doped CaF_2 crystals in order to obtain informations about the impurity defect formation.

YbF_3–doped and NaF-codoped CaF_2 crystals were grown in our crystal research laboratory using the vertical Bridgman method [3]. The obtained CaF_2 crystals do not contain any undesired lead ions amount or other impurities, as results from the optical absorption spectrum. Transparent colorless crystals of about 10mm in diameter and 6-7cm in length were obtained in spectral pure graphite crucible in vacuum ($\approx 10^{-1}$ Pa) using a shaped graphite furnace [3]. The pulling rate was 4mm/h. The crystals were cooled to room temperature using an established procedure. Room temperature optical absorption spectra were recorded by Shimadzu 1650PC spectrophotometer. Capacitance (C) and dielectric loss ($D=\tan\delta$) measurements were performed on the samples using a RLC Meter type ZM2355, NF Corporation, Japan, over the temperature range of 150–320 K, with a linear heating rate of 2 K/min, at seven audio-frequencies: 1, 10, 15, 20, 40, 60 and 100kHz.

By co-doping with Na^+ ions in different Na:Yb ratios it is possible to "tailor" the type of the compensating defects in YbF_3 doped CaF_2 crystals. Dielectric relaxation in double doped (Yb, Na):CaF_2 crystals in order to study the charge compensating defects – was not reported before.

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MONTE CARLO SIMULATION OF LATTICE SPIN MODELS USING TSALLIS STATISTICS AND MULTIPROCESSOR ALGORITHMS

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Spin lattice models are useful tools to study the phase transitions which occur in magnetic systems. The Potts model is an extension of the well known Ising model, in which each spin can have one of *q* states.

Nonextensive statistical mechanics, also known as Tsallis statistics, is a generalization of the classical Boltzmann-Gibbs statistics. In the context of Tsallis statistics the entropy is generally a nonextensive function. In the last years there were established some connections between nonextensive statistics and the experimental behaviour of manganites, hence the simulation of spin models, such as the Ising or Potts model, in the framework of Tsallis statistics became of interest.

For the simulations discussed in the present work we have used a generalized variant of the Metropolis algorithm, where the classical Boltzmann-Gibbs probabilities were replaced with Tsallis acceptance ratios. The algorithm was implemented using a multiprocessor framework based on MPICH2 (a high performance and widely portable implementation of the Message Parsing Interface standard) and SPRNG (The Scalable Parallel Random Generators Library), which allows us to increase the number of Monte Carlo steps per spin in order to establish more accurately the nature of the phase transition.

Simulation results show that the Potts model exhibits a first or second order phase transition in the framework of nonextensive statistics, depending on the number of possible spin states. The order of the phase transition was established by studying the behaviour of the fourth order cumulant of energy and magnetization.

MICROSTRUCTURAL AND OPTICAL PROPERTIES OF ZnO:Al AND ZnO: Ga GROWN BY PULSED LASER DEPOSITION

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Al: ZnO thin films (AZO) and Ga:ZnO thin films (GZO) are studied in order to achieve good characteristics for optoelectronic devices. The purpose of this paper is to study the mirostructures, optical and electrical properties of ZnO: Al (1-5% wt) and ZnO: Ga (1-7% wt) thin films grown by Pulsed Laser Deposition (PLD). The ceramic target were made by mixing, pressing and sintering of pure ZnO with Al_2O_3 (1-5wt%) and Ga_2O_3 (1-7wt%). During laser ablation it was varied the temperature, the oxygen pressure and the energy fluence. The polycrystalline structure of thin films was characterized by X-ray diffraction. Microstructural characterization was made with atomic force microscope and optical characterization with UV-VIS spectrophotometer. Rezistivity measurement of thin films were made by four points method with Lake Shore Hall Effect measurement system. X-ray diffraction measurements reveal a hexagonal wurtzite crystal structure of ZnO. The optical transmittances of GZO and AZO thin films over 70%, indicates possible applications of doped ZnO thin films for optoelectronic devices.

KINETIC SIMULATION OF THE NI_4TI_3 PRECIPITATE MICROSTRUCTURE IN NITI SHAPE MEMORY ALLOYS

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Experimental characterizations of microstructural evolution in NiTi-based shape memory alloys (SMA) have shown strong coupling between Ni4Ti3 precipitate and martensitic transformations. Also, Ni4Ti3 precipitate can influence the mechanical properties of NiTi alloy. Some thermodynamic and kinetic simulations at different temperatures and compositions should be done, together with the identification of stable and metastable phases, in order to determine the right composition and heat treatment for obtaining the Ni4Ti3 precipitate.

MODEL BY BIPOLAR DIFFUSION OF THE POLARIZATION EFFECTS IN SYSTEMS WITH LIQUID CRYSTALS AND MOBILE IONS

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This work refers to the results obtained by dielectric (impedance) spectroscopy measurements on the liquid crystal systems which might contain mobile ions coming from impurities or from molecule dissociation. The spectroscopic results are influenced by the mobile ions. A model is here proposed based on the simultaneous transport of charged particles of both sign (or bipolar diffusion).

This model re-writes the well known system of the continuity equations for the mobile charge carriers and the Poisson equation of the electric potential as well by choosing new variables. Since the applied ac voltage is small enough, the equations were linearised. We suppose that the ions have different mobilities and diffussion coefficients; there is no generation-recombination process and the equilibrium concentrations of the carriers are uniform and equal each other. In addition, the electrodes are either completely blocking or blocking by adsorption-desorption processes. Conditions like those mentioning the different properties of the ions, the entire dissociation process and the neglecting of the generation-recombination of the ions are usually met in bulk liquid crystals.

Finally, one obtains an analytical expression of the system equivalent admittance, allowing a clear image of the role played by the mobile ions and by the electrode-sample interface processes on the dielectric spectra. Though the polarization appears at the electrode surface the mobile ions have a deep influence on the intrinsic bulk properties. The admittance form is simple enough to be easily used and even to be more simplified in order to extract information from experimental data. At the same time, the errors can be estimated by considering the neglected terms in the Taylor series.

STUDIES UPON MULTI-POLAR MAGNETIZATION OF THE YARNS WITH MAGNETIC PROPERTIES

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The magnetizable yarns, which can be used for niche industrial applications, protection against radiations, textronics, medical applications and many others, have basically properties which are specific to composite yarns. On a backing array made of different type yarns and with various fineness degrees obtained by diverse technologies is put a layer with magnetic properties by means of a special procedure. This layer has as components some ferro- and ferrimagnetic powders and is fixed on the backing material with a special binder.

The initiation of magnetic properties of the filiform composite is made in a permanent magnetic field, which is uniform and continuous and is produced by a device that allows a magnetization over various radial directions (multi-polar magnetization). These magnetization directions are the result of the instantaneous magnetic line paths through the yarns, which have been already impregnated after their common extrusion process. The magnetic properties of the resulted yarns refer to the followings: remanent and saturation flux density values, maximum value of the applied magnetic field, coercivity value and even maximum of magnetic energy stored per volume unit.

The paper presents in a comparative way some experimental results obtained by the magnetization of some yarn samples with a device that is similar to the toroidal transformers but with d.c. excitation against a different samples category, which have been processed with a second device (made by the authors) which uses rare earth permanent magnets under a special configuration ("auger die" configuration).

STUDIES ON THE TEXTURAL FEATURES OF SOME LAYERED DOUBLE HYDROXIDE MATRICES

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Layered double hydroxides (LDHs) porous layered matrices, in which magnesium was partially substituted by copper or nickel, were synthesized by a coprecipitation method. LDHs, known as hydrotalcite-like anionic clays, have received considerable interests in recent years owing to their applications as catalysts, catalyst precursors, adsorbents and ion exchangers. The micromorphology and structural characteristics of the substituted LDHs were characterized by field-emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD). The obtained results point out that the layered double hydroxides textural features can be tailored as a function of their composition. The possibility to shape the micromorphologic characteristics of layered double hydroxides can contribute to obtain LDHs with tailored nanoporous properties.

KINETIC MODELING OF THE WATER SOFTENING PROCESS USING CRYSTAL-RIGHT CR100 ZEOLITE

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Modern equipment for residential or industrial water softening frequently uses ion exchangers in form of polymeric resins or zeolite crystalline materials. Zeolites are mostly charged with sodium ions that will be exchanged with calcium and magnesium ions from the raw hard water passing through the crystalline structure. This process is reversible.

The ion exchange-based process referred as softening is designed to remove hardness (calcium and magnesium) from raw water. Zeolite softening will also remove other soluble cation species such as iron and manganese present in the raw water and will mechanically retain small suspended solids.

This paper presents the kinetic modelling of water softening process with a sodium aluminosilicate-based zeolite commercially available under the brand name Crystal-Right CR100. The study of parameter's influence on the ion exchange process kinetics has revealed an adsorption mechanism. It was observed that Crystal-Right CR100 zeolite presents higher affinity towards Ca^{2+} ions compared with Mg^{2+} ions.

Structural characterization of the zeolite before and after use was performed by means of polarized light microscopy, scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM-EDS) and FTIR spectroscopy.

CALCULATION OF THERMO-OPTICAL COEFFICIENT OF SILVER NANO FLUID BY A GAUSSIAN LASER BEAM AND MOIRÉ DEFLECTOMETRY METHOD

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In this paper, the magnitude of thermo-optical coefficient of Silver nano fluid is investigated theoretically and experimentally. The Moiré deflectometery technique is used for the measurement of thermal nonlinear refractive index in Ag nanoparticles suspended in the water. In this method two lasers are used, one as a pomp beam which causes nonlinear effects in sample and the second one is used as a guide beam for monitoring these nonlinear effects by Moiré deflectometry system. The magnitude of nonlinear refractive index and its sign is measured in real time by Moiré fringe deflection. Our study measures the nonlinear refractive index of colloidal Ag nanoparticles (n_2) under 47 mW diode laser pumped by Nd:YAG used as a pomp beam and a 15 mW He-Ne laser as a guide beam illumination. In this paper we introduce a new set-up with moiré deflection technique for study of temporal and spatial refractive index profile, induced by a Gaussian diode laser beam (532nm, 47mW) in a Silver nano fluid sample. Also offering a solution for the heat transfer equation governing the thermal lens effect in metal nano fluids and use the refractive index profile obtained from the Moiré deflectometry, Thermo-Optical coefficient of nano fluids obtain.

AN INVESTIGATION OF ELECTRONIC TRANSPORT PROPERTIES OF THE 2D CARRIERS IN GaInNAs/GaAs QUANTUM WELL STRUCTURE

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I present a study of the electronic transport properties of 2D carriers in n- and p-type Ga1-xInxNyAs1-y/GaAs modulation-doped quantum well structures with having different nitrogen concentrations with using photo current (PC) and I-V measurements under IR light source and at dark condition in various temperature region. From PC measurement I investigate many different behaviors on n type modulation doped quantum well structure under IR light source. From I-V measurement I observed negative differential resistant under IR light source in low electric field region. I compared the results under IR light source and at dark and also in n type and p type Ga1-xInxNyAs1-y/GaAs modulation-doped quantum well structures.

MAGNETIC PROPERTIES OF FeNiCoTi SHAPE MEMORY RIBBONS

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Ferromagnetic Shape Memory Alloys (FSMA) is a class of active materials with the potential to produce both, the large actuator strain of shape memory alloys and the rapid response of magnetostrictive materials. Besides the well known Ni2MnGa and Fe-Pd, Fe-Ni-Co-Ti based alloys [1] are promising as FSMA because of the low material cost, high productivity, high ductility and strong magnetization. For this alloy, the most important prerequisite for Shape Memory Effect (SME) is the formation in the austenitic phase of fine dispersed and coherent (Ni,Co,Fe)3Ti particles which forms the so called coherent γ' phase (FCC ordered with $L1_2$ structure), directly connected with the reversibility of the transformation.

The present work investigates the influence of Co substitution for Ni on the magnetic properties and martensitic transformation (MT) characteristics of the ferromagnetic shape memory alloy Fe52Ni29-xCo15+xTi4, (with x=0, 3 and 6, denoted P1, P2 and P3 samples) prepared as ribbons by melt spinning method and subjected to different thermal treatments. X-ray diffraction, DSC, thermomagnetic measurements and Mössbauer spectroscopy were applied for a complete structural and magnetic characterization. Corroborating the thermo-magnetic measurements with the Mössbauer data is evidenced that P1 sustain an irreversible transformation, while P2 and P3 undergo a partial reversible transformation. Also, increasing the Co content is induced an increase of the Curie temperature and martensitic transformation characteristic temperatures, but the range of MT is decreased.

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CHARACTERIZATION OF MnGeSb2:Co AND ZnSnSb2:Fe THIN FILMS DEPOSITED BY TVA

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The electronic properties of semiconductor thin films are closely related to their structure. Semiconductors that exhibit room-temperature ferromagnetism are central to the development of semiconductor spintronics. This work concerns the deposition and subsequent characterization of MnGeSb2 and ZnSnSb2 thin films that belong to the II-IV-V2 chalcopyrite family. The films doped with Co, Fe have been deposited at room temperature on silicon substrates by the thermionic vacuum arc method (TVA) from the corresponding bulk alloys. Structural and optical characterizations were done by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), Raman spectroscopy, spectroscopic ellipsometry and small-angle X-ray scattering (SAXS). The Hall effect measurements were carried out at room temperature using an Ecopia HMS-3000 Hall Measurement System that is a complete system for measuring the resistivity, carrier concentration, P/N type, and mobility of various materials including semiconductors.

MnGeSb2 exhibits a more metal-like behaviour than ZnSnSb2. A variation of the Hall coeficient with the film thickness of the TVA process is observed in both materials.

ESTABLISHMENT OF A DEFORMATION MODEL BASED ON ZENNER-HOLLOMON PARAMETER

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The stress-strain curves determined by the torsion test allowed the calculation of the Zenner–Hollomon parameter corresponding to the maximum stress. By using this parameter we could establish a set of equations which completely reproduce the stress-strain curve including the hardening area, the restoration area and the dynamic recrystalization area.

In order to determine the specific values of the micro-allied steel, of the percents belonging to the mathematic model of the curve, a set of proofs from steel microalloyed with vanadium was tried at torsion with speeds between 0.05 and 3 s^{-1}, at a temperature of 800, 900, 1000 and 1100°C Celsius degrees.

In microstructures I presented the aspects of the austenite grain size in the initial deformation state at different temperatures.

MARTENSITIC TRANSFORMATION, MAGNETIC BEHAVIOUR AND MAGNETOELASTIC EFFECT IN $Fe_{70-x}Pd_{30}Mn_x$ MELT-SPUN RIBBONS

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The disordered Fe-Pd intermetallic alloys, with around 30at. %Pd belongs to the class of Ferromagnetic Shape Memory Alloys (FSMA). The special interest for this materials is related to its huge magnetostriction (0.6%) achieved in a $Fe_{70}Pd_{30}$- single crystal. Responsible for this strain is the magnetic field induced orientation of the martensite twin variants with high magnetocrystalline anisotropy. Function of alloy composition the thermo-elastic martensitic transformation may be accompanied by a nonthermo-elastic one which weakens the shape memory effect and has to be avoided. In this respect, we report the influence of Mn substitution on the martensitic transformation in correlation with the structural, magnetic and magneto-elastic properties in $Fe_{70-x}Pd_{30}Mn_x$ (x= 0,1,2,3) ribbons prepared by melt spinning technique.

All as prepared ribbons retain the high temperature stable phase, with FCC type structure being in austenite state. The shape memory effect is enhanced after high temperature treatments; the increase of transformation enthalpy is correlated with the refine of the crystalline structure and the reduction of the quenched-in tensions evidenced by XRD.

The effect of Mn substitution on the thermo-elastic martensitic transformation temperatures has the same effect as in the polycrystalline samples [1], but the irreversible transformation was not evidenced in our ribbons. For the different Mn substitutions and thermal treatments, the magnetic behaviour and the magnetoelastic effect are discussed; a large magnetic field induced strain is obtains on x=3 ribbons after suitable thermal treatments.

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STRUCTURAL AND MAGNETIC PROPERTIES OF $GD_5SI_{2-X}GE_{2-X}BI_{2X}$ COMPOUNDS

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The structural and some magnetic properties of $Gd_5Si_{2-x}Ge_{2-x}Bi_{2x}$ (2x=0.02; 0.04; 0.06 and 0.08) compounds were investigate for small amount substitution of Bi for Ge and Si. The alloys were prepared by arc melting method and and annealed at 1250^0C for 200 min. X-ray diffraction, thermomagnetic analysis, magnetization isotherms measurements and differential scanning calorimetry (DSC) were used in order to characterize the structural and magnetic properties of compounds. The alloys have, at room temperature, a main monoclinic phase of Gd5Si2Ge2 type (S.G.: P112_1/a) with a small amount of orthorhombic Ge5Si4 type (S.G. Pnma) and traces of hexagonal structural phase of Gd5Si3 type (S.G.: P63/mcm). The Curie temperature (T_C) of monoclinic phase Gd5Si2Ge2 decreases with increasing of Bi content from ~243K, for 2x=0.02 up to 223K, for 2x=0.08. Around of T_C values, the DSC measurements show a first order structural phase transition for all compounds, with a difference of 5-10K between transitions temperatures in heating and cooling (248K and 243K for 2x=0.02, respectively 238K and 228K, for 2x=0.08). The saturation magnetization in ferromagnetic state was determined from magnetization isotherms measured in magnetic field up to 5T and these decreases also with increasing of Bi content.

THE INFLUENCE OF THE RESIDUAL COPPER ON THE PIPES STEEL'S HOT PLASTICITY

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This work's, is intended to study the cooper content influence as residual element in steel pipes, being known the fact that copper presence in steel leads to surface defects during the hot deformation.

The negative influence of copper, as a residual element of the carbon steel pipes, was proved using torsion deformation at temperatures above 1000°C. The experiments were carried, for comparation, both in the air and in the argon atmospheres.

Steels plasticity was determined by torsion test using Setaram type machine. The samples was heated and then deformed at different temperatures with a deformation speed constant. For each test, the momentum and number of torsions to fracture were wrote. The metallographic analysis of the steels which contains copper at the maximum allowed limit for 1250°C deformation showed, using SEM, copper presence at the samples surfaces of the Fe lower areas due of the oxidation when heating.

TRANSIENT HEAT TRANSFER, FLUID DYNAMICS AND CAPILLARITY MODELING IN VERTICAL BRIDGMAN SOLIDIFICATION OF InSb

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A steady model for the directional solidification of melted InSb in a silica ampoule, using a fixed grid, and solved with COMSOL Multiphysics, was presented in our previous work [1]. This time, the axisymmetric numerical simulations of temperature and velocity fields, under normal gravity, are carried out using time dependent thermal conditions. The geometry and initial boundary conditions of the models resemble those used in an induced pressure difference dewetting vertical Bridgman configuration. A slightly modified version of the method presented by Voller and Prakash [2] is used to account for solidification of the liquid phase. Our model also includes the surface tension at the gas-liquid InSb interface and the adhesive forces between the silica wall and the liquid InSb.

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NUMERICAL AND EXPERIMENTAL STUDIES ON MELT FLOW IN A MODEL EXPERIMENT FOR ELECTROMAGNETIC STIRRING

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One of the key issues in the technology development of multicrystalline silicon ingots for photovoltaic applications is to control the impurities distribution in the mould, which can influence important parameters related to the solar cell efficiency.

We have conducted numerical simulations using the STHAMAS 3D software on a rectangular crucible placed in a vertical magnetic field, through which an electrical DC current is passed. The current flows between two small diameter electrodes attached to the melt surface. In order to validate the numerical results, a model experiment in which a crucible with conductive liquid is placed inside the air gap of a C shaped electromagnet was developed. The vertical magnetic field inside the gap is uniform and stationary, and a DC current flows through two electrodes in contact with the liquid surface.

It was found both by modelling and experiment that even a small magnetic field (less than 10 mT) and an electrical current in the electrodes of a few amperes can produce a stirring effect in the melt. These results show that this idea is suitable to be applied in industrial applications because of the low values of the magnetic field and electrical current.

S8

Advances in Surface Science and Engineering

Invited Papers

CHARGE SENSITIVE X-RAY PHOTOELECTRON SPECTROSCOPY FOR PROBING MOLECULAR NATURE OF ELECTRET FORMATION

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Electrets are materials that develop a permanent electrostatic potential or a permanent dipole moment that have been well-known and utilized. However, unlike metallic or semiconducting systems, the nature of this “electrification“ at the atomic and molecular scale is poorly understood. Kelvin Probe Atomic Force Microscopy (KP-AFM) has been the most advanced analytical tool for probing, mapping, and quantifying the development of charge at submicron length-scales. However, as in most electrical-based measurements it lacks chemical specificity. On the other hand, spectroscopic techniques like IR, Raman, NMR, or variants of them coupled with scanning probe techniques like NSOM, etc. have excellent chemical specificity, but are not sensitive to charge and/or electrostatic potentials developed. Unlike common optical spectroscopic techniques, X-Ray Photoelectron Spectroscopy (XPS) is a charged particle-based chemical analysis technique, and is also extremely sensitive to the presence of electrical potentials on the analyzed surfaces. Besides, the photoelectron emission process itself leads to creation of positive electrical potentials in nonconductive samples due to uncompensated charges, and elaborate charge compensation methods have been developed using low energy electron and/or ion guns to eliminate sample charging. However, complete removal, i.e. achieving the point of zero-charge (PZC) is only an ideal. Besides, measurement of the sign and the extent of the potentials developed as a result of the charging/discharging processes can reveal significant information related with material properties. In this contribution, we will describe a contactless analysis technique to investigate the nature of the charging process of polymer surfaces at the molecular level, using XPS, where films cast from solutions of poly(methyl methacrylate) (PMMA), polystyrene (PS), as well as their blends have been analyzed either in their pristine state or deliberately charged using a flood gun as an external electron source, and by applying external bias to control the extent of charging resulting from a combination of the photoemission process and the compensating electrons from the flood gun. In addition, application of the technique for analysis of various semiconducting materials and devices will be given and discussed.

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Oral Presentations

SYNTHESIS OF WO_3 FILMS BY SURFACTANT ASSISTED SPRAY PYROLYSIS FOR ELECTROCHROMIC APPLICATIONS

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In applications such as electrochromic devices reproducibility in terms of thickness, porosity, structure and morphology largely influences the long term durability and reversibility.

In this study, thin films of WO_3 were prepared by surfactant assisted spray pyrolysis on FTO conductive glass by using cetyltrimethylammonium bromide (CTAB) and PEG400:CTAB mixtures in the spraying solution as growth controlling agents. Surface tension of the spraying solutions was measured and correlated with the film nucleation and growth processes. The effect of surfactant addition on the structure (XRD), surface morphology (AFM) and composition (XPS, FTIR), and on the electrical (impedance spectroscopy) and electrochromic properties was investigated.

The surfactant was found to stabilize the precursor solution due to interactions with the ions in solution which favored the growth of very smooth and homogenous films with improved crystallinity. Significant changes in the morphology and thus in the hydrophilic character of the samples were observed at surfactants' concentrations below and above CMC proving the beneficial role of surfactants in tuning surface properties. The electrochromic activity decreases with increasing crystallinity although electrolyte-layer interaction seems to be favored according to contact angle measurements. Better results in terms of cycling stability were obtained for the samples containing PEG400:CTAB which is probably due to improved adhesion of the layers to the substrate.

Keywords: WO_3; surfactant mediated spray pyrolysis; electrochromic properties

PLASMA TREATMENT OF SOME QUATERNIZED POLYSULFONE FILMS FOR SURFACE AND ADHESION IMPROVEMENT

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The new quaternized polysulfone prepared by quaternization of chloromethylated polysulfone with N-dimethyloctylamine was investigated to obtain information on its surface tension parameters and surface morphology. The results were correlated with the hydrophilic/hydrophobic properties of the polymers. The corresponding studied films were prepared by a dry-cast process in DMF/MeOH and DMF/water, and the composition of the solvent/nonsolvent mixtures were selected as a function of polymer solubility. The type of nonsolvents significantly influenced the surface tension parameters, yet maintaining the surfaces hydrophobic characteristics. Also, a low plasma treatment, besides cleaning the surface, decreases hydrophobicity, which is a reversible phenomenon.

The AFM images showed that the morphology of the films changed because of the nonsolvents used and the solvent/nonsolvent composition.

Low pressure plasma treatment causes significant changes of the polysulfone, inducing several kinds of reactions, the mechanism of which is still unknown, such as breakage of the covalent bonds along the chain, crosslinking, grafting, interaction of surface free radicals, alteration of the existing functional groups and/or incorporation of the chemical groups originated in plasma. However, polymer etching, modification of surface chemistry and deposition of plasma polymer, are the three main groups of processes which should be considered for analysis.

Investigations show that the polar components of surface tensions significantly increase after the plasma treatment. Thus, the relatively hydrophobic surface (higher contact angle for water) is converted into a more hydrophilic one (lower contact angle for water), by the plasma treatment of all studied samples. In addition, paper shows that the values of the polar component to total surface tension ratio increase and the morphology of films significantly changed after plasma treatment.

THERMALLY GROWN OXIDES ON Si(100) INVESTIGATED BY CAVITY RING-DOWN SPECTROSCOPY

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The use of Silicon oxide (SiO_2 and SiO_x) films as protective layers and antireflective coatings in electronics and optics has induced investigations regarding their properties. The growth of oxides on a Silicon (Si) surface can be a particularly tedious process, since the growth must be uniform and pure. For commonly Si electronic applications the thickness of SiO_2 layer is in the range (50÷500)Å, which can take long time for growth process and must be done on a large scale on wafers. The procedure exposed in this work is performed in air flow at 1 atm pressure at the temperature of 250^0C. The contribution of SiO_2/Si interface to optical absorption was studied by measuring the ring-down time on Si oxide film as an optical cavity. Like most of new investigation methods, Cavity Ring-Down Spectroscopy (CRDS) grew out from an advance in technology as a sensitive optical spectroscopic technique that enables measurements of absolute optical extinction by samples that scatter and absorb light. In CRDS a laser pulse is trapped in a highly reflective (R>99.9%) resonant cavity. The intensity of light within the cavity is determined by an exponential decay rate. The decay constant τ_{RD} is called ring-down time and is dependent on the loss mechanisms within the cavity as a measure of its inner characteristics. Our experiments on very thin Si oxide films were performed at a laser wavelength λ=564 nm and put into evidence an absorption coefficient α in the range (10÷15) cm^{-1} in agreement with the general trend from literature data. The ring-down time τ_{RD} has a general decay feature starting from the cavity including a chemical etched Si probe to a cavity including SiO_x/Si probe obtained after exposure time from 1 to 8 hours at 250^0C constant temperature. Our experiment shows that as the oxide layer thickness grows with the treatment time, in the same time τ_{RD} decays, and related to this tendency, the absorption coefficient value α depends on the evolution of $d \bullet \tau_{RD}$ product, where d is the thickness of Si oxides layer grown on Si(100).

ADSORPTION PROPERTIES ON WATER AND ISOPROPYL ALCOHOL MOLECULES AT THE WO_3 SURFACE

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The material WO_3 is well-known as an important photo-catalyst which can produce oxygen from water and also a promising material to decompose many kinds of harmful organic compounds by visible light irradiation. Aiming to obtain the clue to elucidate the origin of the excellent photo-catalytic properties, detailed fundamental properties of WO_3 about the surface relaxation, molecular dynamical properties and adsorption features on water and isopropyl alcohol at the surface and the related electronic structures have been investigated by first principles approach.

The surface structure relaxation and the molecular dynamics on the surface were investigated by Car-Parrinello molecular dynamics simulation using a super cell (2~4 nm^2 × ~3 nm) including WO_3 slab (~1.0 nm or more in thickness) with keeping a stoichiometric charge neutral condition at room temperature (300 K) using a velocity rescaling algorithm. Two kinds of surface models were investigated. One exposes only five-fold oxygen coordinated W (5c-W) at the surface and the other exposes 3c-W at an initial stage before relaxation although the bulk crystal is normally composed of only WO_6 octahedra.

Main results are as follows. (1) In case that just one water molecule is on the surface, the system tends to indicate non-dissociative water molecule adsorption to the 5c-W site. (2) However, in case with bulk water on the slab, some water molecules are dissociated at the surficial 5c-W or 3c-W site with the help of hydrogen bond not with the WO_3 slab but with the bulk water. (3) It is guessed that a R-OH type organic molecule should be unstable and can be dissociated when it comes in proximity of the imperfectly coordinated surficial W site and this has been demonstrated by the molecular dynamics simulation on the isopropyl alcohol at the 3c-W site. (4) Normally, the so-called surface level can be considered to be one of the dominant factors to suppress the oxidization (i.e. O_2 generation from water) efficiency in photo-catalysis in metal oxides, however, in the WO_3 systems, it has been understood that the surface level does not always suppress the oxidization process of adsorbed water by detailed electronic structure investigation.

STRUCTURE AND PROPERTIES OF Mo COATING DEPOSITED ON STEEL SUBSTRATE

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In this paper we have analyzed thermal deposition in electric arc. In order to improve the machine elements durability, we proposed deposit on steel type 16MnCr5 a coating of Mo. After we have realized the samples for mechanical and tribological analysis we have realized the coatings by electric arc deposition. In order to characterize the systems of materials obtained by thermal deposition, we have made SEM and XRD analysis to determine the quality of them. To determine mechanical and tribological properties we used Bending Device, Vickers Hardness Device and Fretting Facility. After we accomplished all those tests we found out that they have superior properties to basic steels.

In SEM analysis, it was found that the coating is homogeneous but it has some cracks due to the deposition processes, when it should be heated the substrate before its coverage deposition.

The X Ray Diffractogram showed us the phases, constituents and Miller indices from the coating. The material had coated the steel surface without any diffusion between their atoms.

The Mo coating is porous and dense but has some cracks. The maximum bending stress which resisted of is about 870 N. The crack appeared between the grains and its distribution was transversal to the layer but it didn’t propagate until the substrate.

On the fretting test, the material was subjected to a sliding fretting in a dry contact. Both the sample and the counter sample were coated with Mo. After $1.1 \cdot 10^6$ cycles, the volume of material lost by friction has been measured with was measured with μScan Profilometer and the wear resulted was almost nonexistent.

EVALUATION OF MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF CARBON STEEL ELECTROPHORETIC DEPOSITED BY ALUMINA-ZIRCONIA COMPOSITE COATINGS APPLYING IN SEMI-PASSIVE DEVICES FOR SEISMIC PROTECTION

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The electrophoretic deposition (EPD) technique was recently applied in semi-passive devices for seismic protection to coat carbon steel sheets with alumina-stabilized zirconia composite powders. The coated specimens were characterized by scanning electron microscopy (SEM), metallography and X-ray diffraction (XRD) techniques in our laboratory. Moreover, these samples were evaluated for microstructural and mechanical behaviours based on load-displacement curves and the results were compared to alumina inserts applying in semi-passive devices for seismic protection. Then, the results of micro-hardness and surface toughness of coated samples were discussed in comparison with alumina inserts after sintering.

Keywords: Alumina-zirconia; Ceramic coating; Electrophoretic deposition; Seismic protection; Carbon steel.

MULTIFUNCTIONAL ADVANCED COATINGS BASED ON ZNO/M OBTAINED BY NANOCASTING METHOD

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Nano-coating has attracted a lot of attention as a simple and cost-effective method of enhancing coating properties by the addition of a small amount of properly designed and dispersed nanometer fillers.

In this research, we applied a nanocasting route for synthesis of ZnO nanoparticles on the mesoporous silicate supports. In the first step, mesoporous silica (SBA-15 structure) was prepared using a soft template and in the second step, this material was used for impregnation of ZnO nanoparticles. The obtained nano-composites were morphologically and structurally characterized by XRD diffraction, SEM, IR and UV-Vis spectroscopy.

The new obtained material was investigated as an additive in two coating systems: acrylic and polyurethane.

With the goal to see how the new material improves the film characteristics of the coatings, different amounts of ZnO/M composite were introduced by ultrasound shaking for 2 hours in the acrylic and polyurethane coating system.

SEM and AFM were used to investigate dispersion of ZnO/M nanocomposite and the changes in the surface behavior of the modified coatings.

The results showed an improvement of the optical and mechanical properties, UV resistance of the new coatings at lower concentration (0.1% by wt), indicating the positive effect of addition of ZnO/M nano-composite in the coatings. These composites will be investigated as an additive for improve weathering resistance of the coatings.

FIRST PRINCIPLES STUDY OF STRUCTURAL, ELECTRONIC AND DYNAMICAL PROPERTIES OF THE ZrC(001) SURFACE

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We presented an ab initio pseudopotential study within the generalized gradient density functional approximation of the structural and electronic properties of the ZrC(001) surface. The calculated structural parameters for the ZrC(001) surface accord very well with previous experimental findings [1]. We have identified surface electronic states on the ZrC(001) surface is metallic, because at least one surface state crosses the Fermi level. Using our atomic and electronic structures, surface phonon dispersion curves are calculated by employing a linear response approach. The calculated phonon spectrum compares very well with the experimental one [2]. Energy locations and polarisation characteristics of Rayleigh, Love, Wallis, Lucas, and Fuchs-Kliewer modes have been determined. Finally, we have compared the phonon density of states of ZrC(001) with the bulk ZrC and find a strong peak with the frequency of 50 meV due to the surface phonon modes in the acoustical-optical gap region.

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CARBURIZATION OF NICKEL SUBSTRATES USING GLUCOSE AS A CARBURIZING MEDIUM

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Carburizing of Nickel using glucose as a carburizing medium was examined in this study. The purpose of this study was to improve the properties of Nickel by modifying its surface. In order to determine the parameters of the carburizing, thermal properties of glucose along with the thermochemical behavior were examined by Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA); thus an estimation of the degradation temperature of glucose was achieved supplying information about the suitable starting point of carburizing. The carburizing process of nickel samples was carried out under vacuum conditions at 380°C and 650°C for 6 hours. The microstructure of carburized nickel samples was characterized by Scanning Electron Microscopy (SEM) and the phases formed were analyzed by X-Ray diffraction (XRD). Electrochemical corrosion tests were also carried out. It was found that samples which were treated at temperatures lower than 400°C showed a better behavior towards corrosion during the electrochemical tests. It was observed that the polarization curves for these samples were shifted to lower corrosion current densities. Consequently, lower corrosion rates were achieved for these samples preventing the formation of extensive pitting corrosion over their surfaces comparing with pure Ni and carburized Nickel substrates at 650°C.

NANOSTRUCTURED COPPER OXIDES OBTAINED VIA ELECTRODEPOSITION FOR GAS SENSORS APPLICATIONS

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Nowadays, interesting research is focusing on the use of copper oxide thin films as multifunctional material. Its chemical stability, optical, electrical and magnetic properties makes it suitable for different applications as electrochromic display devices, solar selective coatings and gas sensors. Tailoring the surface properties and the porosity of the material using self-assembly within the crystallites/films is an innovative way to develop efficient sensor materials.

The paper reports on the surface properties of nanostructured copper oxides deposited on copper substrate. The thin films were obtained via template assisted electrochemical deposition, from copper and sodium acetate electrolyte solutions. Sodium dodecylsulfate (SDS) was added in the precursor solution as surface morphology controlling agent. The effect of the surfactant and its critical micellar concentration (CMC) on the formation of copper oxide has been evaluated. Moreover, the electrical-induced conformational transitions of the SDS in the precursors' solution are studied and correlated with the nucleation and growth mechanisms of the thin films.

To evaluate the wetting behaviour and the morphology, contact angle measurements were performed and correlated with the XRD and AFM/SEM results.

The optimized material with increased specific surface will be used in the construction of a gas-flow-meter. By using nano-structured copper and copper based materials, the value of the heat transfer surface is increased, thus the sensitivity of temperature measurements.

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RAMAN SURFACE CHARACTERIZATION OF CADMIUM TELLURIDE USING 325 NM AND VISIBLE LASER LINES

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Telluride compounds have been extensively studied by micro Raman spectroscopy during the last years. Vibracional modes observed in the range of 90 – 130 cm-1 are associated with tellurium inclusions or aggregates due to the lack of stoichiometry. In previous works we showed Raman peaks of the crystalline tellurium phase and the intensity enhancement behavior with the energy density of the laser spot with energies above the CdTe bandgap. This behavior suggests the formation of crystalline tellurium aggregations on the CdTe surface due to laser irradiation during the acquisition of the spectra.

In this work, the changes on surface morphology and surface composition of Cadmium Telluride (CdTe) single crystals grown by vertical Bridgman process and CdTe thin films surface grown by metalorganic chemical vapor deposition (MOCVD) on GaAs substrate, are studied by Raman Spectroscopy and complemented by Atomic Force Microscopy (AFM) and Energy Dispersive x-ray Spectroscopy (EDS) techniques.

The goal of this work is the study of the appearance of two Raman peaks at around 600-800 cm-1 in CdTe surface when the samples are excited with 325 nm HeCd laser at the room temperature and to clarify the origin of those peaks.

Poster Presentations

AFM OBSERVATIONS ON THE FORMATION OF LEAD PHOSPHATE ON THE LEAD ELECTRODE WITH INSOLUBLE SULPHATES ADDED

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Electrochemical phenomena and processes, conversion of matter's chemical energy into electricity, and/or electricity in various substances, are essential elements in many research fields such as metallurgy, interface interactions, reaction mechanisms, etc. The paper present the in-situ observations of the electrode/electrolyte interface, of the lead sulphate crystal nucleation, growth and reducing, by Electrochemical Atomic Force Microscopy (EC-AFM) combined with Cyclic Voltammetry (CV). An experimental stand was built for this study, consisting of an AFM, a potentiostat and an electrochemical cell. The potentiostat ensures a controlled variation of the electrode potential. The AFM records in-situ images of the crystallisation/dissolution on the electrode surface during oxidation and reduction reactions. The electrochemical cell was built in such a way to ensure the correct electrical connection between Working Electrode, Reference Electrode and Counter Electrode. Crystals such as calcium sulphate, bismuth sulphate, europium sulphate and dysprosium sulphate were placed and pressed onto the lead electrode (the negative electrode). These crystals were chosen because they are insoluble in sulphuric acid and they have a crystalline structure similar with the lead sulphate. The aim was to observe if they could become crystallisation seeds for the formation of the lead sulphate crystals. It was found that when calcium sulphate was added on the lead electrode, the lead sulphate crystals grew in width and much less in height. Using bismuth sulphate as additive, no lead sulphate crystals were formed on the lead electrode. With rare earths sulphates as additives, it was found that they do not participate to oxidation/reduction process.

THE CHANGING OF THE WELDED SURFACES APPEARANCE AND OF THE PHYSICAL – MECHANICAL CHARACTERISTICS UNDER THE ACTION OF VIBRATIONS

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The paperwork presents aspects regarding the influence of sinusoidal mechanical oscillations of low frequency on the surfaces's appearance achieved by welding-loading operations, and on the microstructure, density and logarithmic decrement. The results obtained and presented can be used, for the development of welding-loading technologies, both construction steels with improved weldability and alloy steels or higher carbon content steels, which are not currently welded.

Under the action of vibration applied during welding process, there are made surfaces with fine and uniform scales, showing max. 0.2 mm dimples. It also increases the density of the deposited material, reduce tensions and internal friction of base material.

PREPARATION AND CHARACTERIZATION OF TANTALUM NITRIDE AND TANTALUM OXIDE THIN FILMS: THE EFFECT OF STRUCTURAL FEATURES ON THE DECORATIVE PROPERTIES

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This study aims to investigate structural and decorative properties of tantalum nitride/oxide films deposited onto silicon, stainless steel, high-speed steel and glass substrates by reactive magnetron sputtering. One set of samples (tantalum nitride) were deposited varying the nitrogen flow from grounded condition up to 17.5 sccm, with a bias voltage of -50 V and deposition time of 3600 s keeps constants. The other set of samples were deposited with a variation of the time from 3600 s up to 7200 s and the oxygen flow from 15 up to 20 sccm. The obtained films were optically characterized by reflectance spectroscopy, in the visible region. The colour of the films was expressed in the CIE 1976 L*a*b* colour space. Colour measurements in the tantalum nitride films revealed a change from silver at low reactive gas flows to metallic for the films prepared at the highest flows. Moreover, for tantalum oxide different colours were obtained, from silver to interference. All these kinds of films (tantalum nitride/oxide) are directly correlated with the deposition conditions and, as consequence, with the structural properties. The structure was assessed using X-Ray diffraction. The results reveal for tantalum nitride a crystalline structure with strong peaks of TaN and with a (100) preferential growth. At the same time, tantalum oxide showed weak peak intensities which correspond to (001) and (101) planes of that hexagonal TaO type structure and for the samples prepared with the highest values of oxygen flows the XRD pattern showed a significant loss of crystallinity due to the increase of the number of defects and made to appears the amorphization.

Keywords: tantalum nitride**,** tantalum oxide, sputtering, structure, optical and decorative properties.

TRIBOLOGICAL INVESTIGATIONS OF HIGH-PERFORMANCE COATINGS FOR INTERNAL COMBUSTION ENGINES USED IN MOTOR VEHICLES

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The goal of this study is to investigate the tribological properties of a high-performance coating, intended to be used for internal combustion engine components, in comparison to conventional materials. By applying the performance material, the manufacturer's objective is to extend the lifetime of the specific component of the engine. In order to highlight the benefits of using performance coatings, a direct comparison between the conventional material used in the engine (named reference material) and the performance material, was necessary. The manufacturer provided four different types of performance material, and the reference material.

The study was structured in several investigation phases. First, all investigated materials underwent an optical investigation of the surface, by using a scanning electron-microscope (SEM). In the second phase, the mechanical properties were determined, by using a roughness-meter and hardness tester. In the third phase, the friction properties of the materials were studied on a rotating tribometer, using two lubrication types. In the last phase, by replacing the rotating tribometer with an oscillating tribometer, the friction properties of the materials, in the case of boundary lubrication, were investigated. The final objective of this study was to determine the best performance material, among the four provided by the manufacturer and to quantify the benefits of using advanced materials in comparison to classic, conventional materials.

Keywords: high performance coatings, internal combustion engines, tribology, lubrication.

ARC WELDING IN INERT GAS ENVIRONMENT (WIG, MIG) OF TITANIUM AND ITS ALLOYS

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Inert gas welding is the most common welding method (using Ar, He as inert gases – the purity of Argon used for welding Titanium will be 99.99%). The easiest assessment of gas protection is observing the color of the stitching. In the case of insufficient gas protection the welding quality decreases. In descending order of the quality of stitching due to inefficient protection of inert gas, we can notice the following colors: light silver, light straw yellow, dark straw yellow, light blue, dark blue, gray blue, ash white. The color criteria is not accepted as a standard for quality check, but can serve as quality estimation (silvery white stitching and light straw yellow or dark straw yellow can be generally considered adequate). The assessment of stitching is based upon mechanical tests.

INFLUENCE OF LASER HEAT TREATMENT ON METAL COATING LAYERS

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The paper presents a research concerning the influence of laser heat treatment on welded coating layers. The research was made with four types of electrodes for welding coating. Evaluation of results was made by researching the microhardness, the microstructure, SEM and EDAX. Results reveal a good effect of laser heat treatment in case of coating with electrodes having low carbon and moderate chrome content.

Usually, the hardness obtained after coating depends only on the filler material characteristics. The goal of this research is to demonstrate the increasing of hardness after the laser heat treatment of the welded coating layer. The welding coating process enables the recovery by means of reworking of some parts or machine components that reached the wear limit. In this situation some examples are: dies, crankshafts, different machinery axles, camshaft which can be brought back to the nominal quota.

After the analysis of the laser-hardened and remelted shaft under different power conditions, the conclusion was that the laser beam heating produces two kinds of regions inside the laser tracks. One region is composed predominately of martensite; the other region consists of unchanged proeutectoid ferrite, martensite and some pearlite.

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EFFECTS OF LASER SHOCK PROCESSING ON 316L STAINLESS STEEL WELDS

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This paper presents an experimental study on the effect of laser shock processing (LSP) on mechanical properties of 316 L stainless steel welds. An Q-switched Nd: YAG laser with a 1064 nm wavelength and maximum power of 3.3 kW was used for welding. The LSP energy 2.8 J/ pulse was produced by the same laser, having a spot diameter of 1.5 mm and 10 Hz frequency.

We present the results for LSP surface treatments applied with two different pulse densities 900 pulse/cm^2 and 1600 pulse/cm^2. The purpose of the work is to compare the effects on mechanical properties of untreated weld and LSP treated with both densities. Also the effect on microstructure was investigated by optical microscopy. The results are discussed and plotted.

The results show the advantages of LSP surface treatment.

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A COMPARISON STUDY BETWEEN BORONIZING AND CARBURIZING PROCESS ON IRON- BASED AUSTENITIC STEELS AT THE SAME TEMPERATURE TREATMENT

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In this study, mechanical properties of borided and carburized Iron- Based Austenitic Steels (IBAS) 0.24 wt% C were investigated. Boronizing was performed by pack cementation in Ekabor II powders and carburization was carried out under vacuum conditions using glucose as a carburizing medium at the same temperature treatment at 900^{0}C for 3h, respectively. X-ray diffraction analysis, Optical observations, SEM-EDX measurements and micro-Vickers hardness tester were conducted. Borided layers with a single phase layer, exclusively Fe_2B strongly “toothed”, exhibited better hardness values upon carbides formation from carburizing process. Potentiodynamic polarization curves were used to determine such electrochemical characteristics as the corrosion potential (mV), corrosion current (μA/cm2) and corrosion rate (μm/year).

RESEARCH CONCERNING THE CORROSION BEHAVIOUR OF THERMAL TREATED HYBRID COMPOSITES WITH ALUMINIUM ALLOY MATRIX REINFORCED WITH SiC AND GRAPHITE

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This paper presents results of studies on the corrosion behaviour of AlMCH composites with different concentrations of the reinforcement elements (10% SiC and 1.25% and 3% graphite coated with copper) in various processing stages.

Composites with the aluminium alloy matrix AlSi7Mg03 reinforced with SiC particles and graphite coated copper were obtained by applying the Vortex method.

The behaviour to corrosion was evaluated by immersion tests, electrochemical measurements and optical microscopy analysis. The immersion tests were executed in a -5% NaCl solution with different exposure times and facilitated through gravimetric measurements the determination of the kinetics of the process and the growth law of film on the analyzed composites in the initial state and thermal treated T6. In accordance with conventional corrosion resistance scale the analyzed materials are included in the very stable group, with a coefficient of stability 3-4. For materials composite with 3% graphite, the mass loss is greater but the variation curve over time has the same character.

Composites with a metallic matrix (MMCH) have wide application in the aerospace, automotive and naval industries. Reinforcement of aluminium alloys with SiC and graphite led to a new generation of engineering materials.

Specialised literature presents research focused on comparative analysis of the behaviour of monolithic and composite alloys with a single element of reinforcement in various concentrations.

Many studies have focused on MMC composites with different manufacturing technology, with different casting and powder metallurgy. This paper aims to analyze the corrosion behaviour of the aluminium alloy matrix, reinforced with two elements, SiC and graphite coated with copper, which provides physical, mechanical characteristics and superior service.

DEPOSITION OF METALLIC RESISTANT COATINGS BY PACK CEMENTATION FOR THE PROTECTION OF COPPER COMPONENTS

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There are several metallic coating elements, such as zinc, chromium, magnesium, and aluminum which are proved to protect against different corrosion types. Copper is widely applied in high strength and thermal conductivity applications at elevated temperatures such as cooling of high-temperature metallurgical reactors. It is also used in integrated circuits due to its excellent electric conductivity features. However, it is susceptible when exposed in high temperature aggressive environments as the oxidation products easily delaminate and cannot protect the underlying material. Up to now, the previously referred protective coatings, were mainly applied and tested, for the protection of ferrous components.

In this work the feasibility of aluminum and magnesium oxidation resistant coatings, on copper substrates is investigated. The deposition was carried out by pack cementation process which is a novel and simple diffusion coating process. The experiments were undertaken at temperatures from 500 to $600^{o}C$, while the duration of the process varied from 30min to 3hours. The samples were examined with X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). It was found that both parameters, temperature and duration of the deposition, had only affected the thickness of both intermatellic coatings and not the structure and morphology.

In the cross sectional SEM micrographs of the aluminum deposited copper substrate, it was revealed that it was composed by a single layer corresponding to Al_4Cu_9. The magnesium coated substrates were found to be composed of two distinct layers with different Mg content. Phase identification analysis revealed that these phases are $MgCu_2$ and Mg_2Cu. The second phase corresponds to the outer layer and the first to the inner one (in contact with the substrate). The oxidation performance of the samples was evaluated by exposing the coated coupons in a high temperature air environment together with a bare copper component. The results showed that the coated samples have increased oxidation resistance while copper samples had undergone severe degradation. The resistance of the coated samples is mostly attributed to the formation of resistant oxides of the coating materials.

THE EFFECT OF HEAT TREATMENT OF THERMAL SPRAYED Al/NiCrBSiFe COATINGS ON LOW CARBON STEEL SUBSTRATES

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Multilayer coatings are reported to be a promising method for the protection of ferrous substrates in order to combine the protection offered by, more than one materials and reserve the enhanced mechanical properties of the substrate even under aggressive environments. Aluminium coatings offer a combination of attractive properties such as low density, and good oxidation resistance. Nickel-based alloyed coatings are widely applied because of their proved good wear resistance and workability at high temperatures. Nickel aluminides have been claimed to possess high-temperature mechanical strength and oxidation resistance which increases with temperature. A simple and economic method for applying such coatings is thermal spray which is a method with high production rate and high versatility. In this work a double layered coating was formed by a two stage thermal spray deposition process. An initial Ni-Cr layer was primarily deposited followed by an Al containing layer in the next stage. Selective specimens were also heat treated in inert atmosphere in order to form nickel aluminides compounds as a result of the diffusion between the two layers. The donor feedstock material was NiCrBSiFe and Al metallic powders (-120 +325 mesh).

The aim of this research is to reveal the morphological and microstructural characteristics of the as deposited and heat treated flame sprayed coatings. Moreover, an assessment of the durability of the samples was made, after their exposure in a high temperature air environment and in a simulated marine atmosphere. The examination of the samples was performed using a Scanning electron microscopy and X-ray diffraction analysis. The as sprayed coatings were found to contain two distinct layers of Al and Ni-Cr which were also characterized by elevated porosity. In the structure of the annealed samples several island like areas were traced in the Al layer which were mainly composed of nickel and aluminum. Coatings treated at 700^{o}C were completely transformed in Ni-Al compound. The examination of the oxidized and corroded coupons showed that the annealed samples have significantly higher resistance than the as sprayed ones. Furthermore, the annealed samples have better performance in marine environment which is mainly due to the presence of Ni-Al and Al oxide compounds on the coating surface.

STRUCTURAL, OPTICAL AND MECHANICAL PROPERTIES OF TANTALUM NITRIDE THIN FILMS PREPARED BY REACTIVE-MAGNETRON SPUTTERING

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This work reports on the study of structural, optical and mechanical properties of tantalum nitride films produced by reactive magnetron sputtering. The thin films were deposited onto silicon, stainless steel, high-speed steel and glass substrates from a pure Ta target. The nitride samples were deposited varying the nitrogen flow from 4.5 up to 17.5 sccm, with a constant bias voltage equal to -50 V, applied to the substrate. The obtained films were optically characterized by reflectance spectroscopy, in the visible region. The colour of the films was expressed in the CIE 1976 L*a*b* colour space. Different colours were obtained, from silver at low reactive gas flows to metallic for the films prepared at the highest flows, and they are directly correlated with the deposition conditions and, as consequence, with the structure. The structure of the deposited coatings was analyzed by X-ray diffraction. The structural results showed the presence of strong peaks of TaN with a (100) preferential orientation. Nano-indentation measurements showed that the hardness and elastic modulus value of the tantalum nitride thin films with nitrogen flow of 4.5 sccm were higher than with other deposition conditions. A correlation of the reactive magnetron sputtering deposition parameters with the structural and mechanical properties of tantalum nitride thin films was discussed in this paper.

THE WEAR RESISTANCE OF HEAT TREATABLE STEELS, SURFACE HARDENED WITH CONCENTRATED ENERGY SOURCES

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Carbon and alloyed heat treatable steels have been subjected to some surface treatments using the laser radiation; the surface treatment was preceded by the improvement one. The attempts to wear effectuated on special samples have revealed the different behavior depending on the steel quality and the applied heat treatment.

PREPARATION AND CHARACTERIZATION OF NON-SULFIDED CoMo CATALYST FOR HYDROCRACKING OF FISCHER-TROPSCH WAX

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Fischer-Tropsch synthesis could be a promising process for production of biocombustible materials from biomass gasification if the large fraction of long chain paraffins obtained could be cracked to lighter hydrocarbons belonging to diesel fraction. Hydrocracking of Fischer-Tropsch wax can yield high quality diesel with lower content sulfur and aromatics and higher cetane number comparative with conventional commercial fuels.

Non-sulfided CoMo supported on commercial Al_2O_3 and SiO_2-Al_2O_3 were prepared by non-dry successive impregnation method: first, a solution of ammonium heptamolybdate with a pH of 6.5 was impregnated onto support, and then Co was deposited on the calcined Mo/support precursor, using aqueous solution of cobalt nitrate. The samples had the same composition of Co and Mo, corresponding to an atomic ratio Co/(Co+Mo) equal to 3. The samples were characterized and compared using N_2-adsorbtion for determination of specific surface area, pore volume and average pore diameters, AAS, XRD, SEM and AFM analysis in order to establish the morphology and the dispersion of Co and Mo species on simple Al2O3 or SiO2-Al2O3.

THE INFLUENCE OF PIGMENT VOLUME CONCENTRATION (PVC) ON CORROSION BEHAVIOR OF ZINC-RICH EPOXY COATINGS

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In this work, the effect of the ratio of the pigment volume concentration (PVC) to the critical pigment volume concentration (CPVC), denoted hereafter by Λ, on the corrosion behavior of the zinc-rich epoxy coatings has been investigated. Paints based on epoxy resin and polyamide hardener were prepared at different Λ values (0.80 0.90, 0.96, 1.05,1.14 and 1.23) and applied on degreased and sandblasted mild steel sheets at 60 microns dry film thickness. In order to investigate the corrosion behavior of the zinc-rich epoxy coatings, the painted mild steel electrodes were studied during exposure to 5%NaCl solution for up to 90 days and electrochemical impedance spectroscopy test (EIS) has been taken after 7and 90 days exposure. In parallel, continuous salt spray test in accordance with ASTM B117 has been done for 2000 hours. The results showed that, corrosion resistance can be affected greatly by the PVC/CPVC ratio and conclusions offered each of the techniques used are quite similar. It has been proved that coatings with PVC=59% orΛ=1.14 have better corrosion resistance in comparison with other ratios.

Keywords: Corrosion protection, zinc-rich paints, Electrochemical Impedance Spectroscopy

MEASUREMENTS OF CONTACT ANGLE OF ROUGH AND FUNCTIONALIZED SURFACES

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Contact angle is an important characteristic since it is a sensitive indicator of changes in the surface energy and changes in the chemical and supermolecular structure of the surface(s) under modification. Knowledge of the contact angle allows estimation of the interaction between surfaces and liquids and has practical applications.

This contribution presents measurements of contact angle performed by the sessile drop method which analyses the shape of a liquid drop put on the surface to be examined. However, the surfaces investigated are far for being flat, smooth and homogeneous. Textile surfaces are rough to a degree; depending on the structure of the interlace of yarn strands, the fibre arrangement in the fabrics, woven, knitted and needled nonwoven ones. The application of the model algorithm for these surfaces requires a specific setting of the base line, establishing the contact point of the three-phases on the base line and differentiating artefacts on the liquid drop shape.

Wetting of fabric surfaces are processes complicated not only by surface roughness, but also the heterogenity, diffusion of liquid into the fibre, and the capillary action of the fibre assembly: therefore the experimentally measured contact angle is in fact an apparent one and can differ considerably from the true value.

Fabrics made from cotton, polyester and/or linen, either as such or after functionalization with organic/inorganic agents were investigated. The contact angle values of water onto the fabrics speak about their chemical modifications.

HELIUM ATOM DIFFRACTION STUDY OF PENTACENE MULTILAYERS AND MONOLAYERS ON VICINAL Ag(111) AND FLAT Ag(111) PREPARED BY SUPERSONIC MOLECULAR BEAM DEPOSITION

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Pentacene thin films are still being studied heavily due to their potential electronic device applications and being a model system for organic semiconductor film studies [1-3]. Here we present a structural study of pentacene multilayer and monolayer thin films grown with diffrent carrier gases He and Kr on vicinal Ag(111) and flat Ag(111) surfaces by He atom diffraction measurements. Our He atom diffraction results suggest initial adsorption at the step edges evidenced by initial slow specular reflection intensity decay rate as a function of Pentacene deposition time.

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HELIUM ATOM DIFFRACTION STUDY OF DECANETHIOL SELF-ASSEMBLED MONOLAYERS GROWN ON Au(111) PREPARED BY SUPERSONIC MOLECULAR BEAM DEPOSITION

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Thiol self-assembled monolayers (SAMs) on gold surfaces have a wide range of applications including surface patterning and metal contact – organic semiconductor interface modification in organic electronic devices. Here we will present our studies about the low coverage phases of decanethiol ($CH_3(CH_2)_9SH$) SAMs on Au(111) surface[1-3], grown in vacuum by supersonic molecular beam deposition. Crystals structures, phase behaviors and desorption energies of these films determined by helium atom diffraction technique will be discussed.

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COMPARISON OF GAS SENSING PROPERTIES OF ZnO NANOFLOWERS AND 2D ZnO THIN FILMS GROWN BY SPRAY PYROLYSIS

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This paper studies synthesis and characterization of quasi one dimensional zinc oxide semiconductor nanostructures with the purpose of comparing them with thin film structures in oxygen gas sensing. These nanostructures were synthesized by spray pyrolysis method.

The growth process is controlled based by different growth condition.

The structure of developed samples was studied using field emission scanning electron microscope (FESEM). The SEM images show the formation of ZnO nanoflowers with single and multiple bar structures, length in order of 1 micron and 80 nm diameter. One of the most important tests for ensuring formation of crystal phases is X-Ray diffraction pattern (XRD). The pattern indicates formation of wurtzite ZnO crystal phase (due to the formation of main ZnO peaks) in synthesized samples.

The results of gas detection tests have been also discussed, and suitable structures for fabrication of gas sensors are specified. According to the measurement results of various samples of alumina and silicon substrates, it can be concluded that low temperatures are the optimal sensing temperatures and Alumina is the better desirable substrate. This study attempts to compare the nanostructure sample with thin layer which were fabricated using same method, but under different development conditionsThe sensitivity of the sample nanostructure compared to Oxygen in different temperatures and gas flows. The results show sensitivity in these nanostructures is more than their thin layer samples and indicate that it is possible to fabricate desired gas sensors with wide applicability in different fields of industry, such as oil, automotive, mines and etc.

DEPOSITION AND OXIDATION STUDIES OF Mg_2Si COATINGS ON SI SUBSTRATES BY PACK CEMENTATION

D. Stathokostopoulos, D. Chaliampalias, E. Stefanaki, E. Pavlidou, G. Stergioudis, E. Hatzikraniotis,
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Research on new thermoelectric materials is one of the main scopes of the recent experimental studies in order to develop new materials which have similar performance and additionally low environmental impact and low fabrication cost. One of the candidate materials, which have attracted a lot scientific interest, is the intermetallic Mg_2Si compound. Its main advantages are that is formed from inexpensive materials, it is lightweight, non-toxic and has high electron mobility. In several researches Mg_2Si has been used as matrix for doping with metallic elements, such as Al, Bi, Sb as n-type dopants and Ag as p-type, in order to improve the electric properties and enhance the thermoelectric figure of merit ZT. The easy oxidation and volatilisation of magnesium are the two main obstacles to obtain high-quality crystalline Mg_2Si. Magnesium silicide can be applied on thermoelectric devices which work under high temperature aggressive environments. However, there are few published studies which examine the oxidation resistance at such conditions.

In this work the feasibility of depositing Mg_2Si coatings on Si substrates, is investigated, by pack cementation which is a novel and simple method in comparison with the already used deposition processes. Moreover, the oxidation performance of the samples was evaluated by subjecting the coated coupon in high temperature air environment. The samples were examined with X-ray diffraction analysis (XRD), Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The deposition experiments were undertaken for different deposition durations varying from 15min to 300min and temperatures from 450°C to 650°C. It was found that both time and temperature parameters only affected the coating thickness while cross sectional examination revealed that the coatings are composed of pure Mg_2Si without any impurities that are present in coatings deposited with other processes. From the structural examination of the oxidized samples it was found that the oxidation products were MgO and Si.

NANOINDENTATION ANALYSIS OF DLC/Si HARD COATINGS

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The attractive chemical and mechanical properties of diamond like carbon (a-c:H) proves itself as a versatile candidate from automotive to micro-electronics industry and particularly for cutting tools applications. The mechanical properties of Dlc/Si hard films deposited by physical vapour deposition (PVD) at 180ºC have been analysed by nanoindenter. The thickness (2µm) of the coating was measured by cross-sectional SEM image and also compositional microanalysis confirmed by EDS. The mechanism after the indentation process were discussed and interpreted. Hardness and elastic modulus values were tabulated for the coatings and the substrate. The performance of the coating/substrate system was also studied by taking into account the H/E ratio.

LASER SPECKLE PATTERN TECHNIQUES APPLIED TO CORROSION MONITORING

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We present an experimental study on corrosion monitoring by using speckle pattern techniques. The corrosion is a surface degradation process and the optical methods based on coherent light are sensitive techniques to detect surface alterations. Our setup includes a three-electrode corrosion cell connected to a PGstat device as commonly used for studying corrosion by standard electrochemical methods. The corrosion cell has a special design to support a digital microscope, so that we can inspect the probe surface. The probe is illuminated by a laser diode. A speckle pattern is superposed onto the image of the probe surface. The speckle pattern has statistical properties and we are studying how they are related to the corrosion state of the probe, which undergoes a corrosion process at a rate controlled by the PGstat.

We use dynamic speckle procedures in the frequency domain in order to compute some statistical quantities of the speckle pattern, such as average brightness and average speckle size and we present a fast algorithm for processing the speckle image. Generally, the information about the average speckle pattern is found in the very low frequency range of the Fourier spectrum, around the central zero-frequency peak. A commonly used fast Fourier transform procedure can determine this quantity with a poor resolution. We have made a substantial improvement by using a type of scaled convolution, which allows zooming into the frequency domain and the average speckle size can be determined much better. The probes consist of copper and some copper alloys and we study their corrosion behaviour in seawater.

EVALUATION OF MECHANICAL PROPERTIES BY INSTRUMENTED INDENTATION OF DUPLEX TREATED STEELS

Ramona Monica Dobra, Rui Vilar, and Ion Mitelea
Politehnica University of Timişoara, Technical University of Lisbon, Politehnica University of Timişoara

The continuous deterioration, due to corrosion and wear, of metallic surfaces used in major industrial applications leads to loss of efficiency and in some cases to eliminations of these parts. The surface properties of such mechanical parts may be improved through Duplex treatments. A Duplex treatment, as the name implies, is the application of two treatments, combining their advantages and leading to better surface properties.

A Duplex treatment consisting in a gas carburizing prior to surface induction quenching have been proposed as a solution for improving the properties of EN 16MnCr5 alloyed steel. This papers report the results obtained for hardness and bulk modulus on treated and untreated specimens. These specimens were examined using a Dynamic Ultra Micro Hardness (DUH) tester under a set of maximum loads ranging from 100 to 1000 mN. Each specimen was subjected to load-unload cycle under the same amount of maximum load and the loads vs. penetration depth curves were plotted.

Keywords: Duplex treatments, carburizing, bulk modulus, hardness

NOVEL APPROACH REGARDING ZETA POTENTIAL VARIATION AS A CONSEQUENCE OF ANTIMICROBIAL FABRICS WASHING TREATMENT

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Police uniform made of polyester fibres, should present a high level of antimicrobial protection, given the environmental conditions they are worn in. Taking into consideration this aspect, the present research has been conducted on polyester fibre materials, previously antimicrobial treated with zinc oxide in various concentrations. The main parameter used to study the time stability of the antimicrobial treatment is the Zeta Potential and its variation. The stability of the antimicrobial treatment during the period of uniform exploitation was simulated through repeated washes in the laboratory and the measurement of the Zeta Potential. Zeta Potential values belonging to specimens treated with zinc oxide suspensions with different concentrations, before and after washing decreased with the augmentation of zinc oxide concentration, but stability maintenance is noticeable.

MECHANICAL AND TRIBOLOGICAL EVALUATION OF WC COATING

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In this paper we have realized coatings by Electro-Spark Deposition using WC electrode obtaining a coating of 200 μm thickness. The systems of materials obtained by thermal coating have been analyzed by SEM and XRD methods in order to determine their quality. Bending facility has been used, in order to establish the admissible bending stress of WC coating. For determination of friction coefficient, we have been made the fretting test, and to evaluate the WC coating wear we have done roughness measurements in contact area. To estimate coating hardness properties we have done measurements by Vickers method.

In SEM analysis, it was found that the coating is homogeneous but it has some imperfections due to the deposition processes. In chemical composition of WC coating obtained by ESD it was found iron as an alloying element which shows that there was an impregnation, diffusion between the atoms of the substrate and of the electrode used. At bending test the material resisted to a force of 1107N, but when the first crack appeared, the sample had destroyed very fast, because the crack's propagation has been transversal directly to the substrate. Fretting test has been made with amplitude of 75 μm, a frequency of 10 Hz and a sliding force of 50 N, applying a normal load of 200 N for more than one million cycles in dry contact. The 3D roughness profile in the contact area is the volumetric analysis of the material lost by friction, and it showed that after more than one million cycles the wear is insignificant and could be neglected.

The material's microstructure influences the mechanical and tribological properties, proved experimentally, when, at bending test, WC coating behaved bad, while at fretting test, it behaved very well.

THE ROBUSTNESS OF THE ELECTRON'S PHASE MEASUREMENTS IN ISOSPECTRAL 2D NANOSTRUCTURES

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Recently, C.R. Moon et al. [Science 319, 782 (2008)] have built pairs of isospectral shapes - i.e. non-congruent polygons which share the same eigenspectrum of the Schrodinger equation - by individually planting CO molecules on a Cu surface, with the use of an STM tip. The focus was to measure the phase of the electron's eigenfunctions, in a non-interferometric way, for the first time. Indeed, the set-up allowed them to extract not only the amplitudes distributions of the eigenmodes (which are directly measurable with the STM tip), but also their phase distributions, which was possible to obtain because of a supplementary information, called transplantation, relating the eigenmodes of the isospectral pairs. The purpose of the present paper is to theoretically investigate the robustness of the isospectrality-basted phase extraction against inherent "imperfect" experimental conditions. One should expect that surface disorder, edge roughness or electron-electron interaction may compromise the isospectrality-based phase extraction in a similar way the interferometry-based phase extraction is compromised by inelastic scattering or environmental-induced dephasing. Our systematic numerical investigations lead to the conclusion that the isospectrality-based phase extraction presents a remarkable robustness. In terms of wave functions misfit, it should be less than 5% after the transplantation procedure, in order for the measured phase to coincide –or differ negligibly from- the phase of the disorder-free system. Another legitimate question that we shall address is the reversed problem, namely how to extract information on the surfaces disorder, based on the phase extraction procedure.

THE USING OF LASER RADIATION AT SURFACE HARDENING OF IMPROVEMENT STEELS

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The paper aims the influence of the laser radiation parameters on superficial layers of carbon and alloy improvement steels. Geometry and depth of penetration of radiation effects are studied by optical and electronic microscopy. It aims also the distribution of components on the affected film thickness by laser energy, analyzed by EDAX and variation of mechanical properties.

OPTICAL AND ELECTRICAL PROPERTIES OF Si_xGe_{1-x} (0<x<1) THIN FILMS OBTAINED BY COMBINATORIAL PULSED LASER DEPOSITION

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During the last decade, great efforts have been focused to discover and develop new materials with improved optical, electronic and physical properties. Extraordinary progresses have been made in the synthesis of novel compounds with remarkable features. However, the conventional "step by step" investigations are time consuming and expensive due to the requirements and the complexity of the materials used for the prospective technologies. Recently, the combinatorial approach has offered a comprehensive manner to overcome this barrier. Combinatorial approach implies the rapid synthesis of a large number of different but structurally related compounds. A collection of these synthesized compounds is referred as a combinatorial library. SiGe combinatorial films were deposited at room temperature by simultaneous ablation of two targets (pure silicon and germanium) on glass and Al_2O_3 substrates. Morphological and topological profiles were determined by microscopy techniques (AFM, SEM) while the profile of composition were obtained by energy dispersive spectroscopy (EDS). Optical properties (band gaps, T%) and thickness profiles of the deposited structures were characterized by spectroscopic (UV–VIS and Ellipsometry) methods. We measured the corresponding electrical properties (resistivity, carrier concentration and mobility) for different Si_xGe_{1-x} compositions.

NANOMECHANICAL STUDIES OF EPITAXIAL GaN/InGaN HETERO.STRUCTURES

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Wide-band-gap gallium nitride (GaN) and related III-nitride materials are technologically important for their applications in optical and electronic devices. Studies of the processes controlling contact damage, wear, and cracking of semiconductor layers have significant technological importance. Nanoindentation-induced mechanical deformation behaviour of GaN/InGaN multiple quantum well (MQW) structure grown on (0001) sapphire was investigated and analyzed using atomic force microscopy (AFM)

A single discontinuity ('pop-in') in the load-indentation depth curve was observed for both the films, the physical mechanism being the interaction of the deformed regions produced by the indenter. AFM studies on the residual indentation impression revealed pile-up and sink-in behaviour which can be related to the plasticity nature of the samples. Hardness and elastic modulus of the films were evaluated from the load–displacement data obtained. The Oliver and Pharr method was followed to determine the hardness and Elastic modulus from the load-displacement curves.

Addenda

Invited paper

Application of Carbon–Tungsten, Carbon-Berillium And Carbon-Aluminium Nanostructures in Divertors Coatings from Fusion Reactor

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In the current ITER design, the tiles made of carbon fiber composites (CFCs) are foreseen for the strike point zone and tungsten (W) and beryllium for other parts of the divertor region. This choice is a compromise based mainly on experience with individual materials in many different tokamaks. Also Carbon-Aluminum composites (C-Al) are the candidate material for the First Wall in ITER. The goal of this paper is to exchange, the C-Be thin films, with equivalent C-Al thin films. In this respect, it is necessary a solution for material synthesis and also find correct concentrations between carbon and aluminum. The Thermionic Vacuum Arch (TVA) method is suitable for the synthesis of wide range of materials, and more important is a low cost method that allows variation of concentration in one shot deposition.

Keywords: divertor, C-W, C-Be, C-Al composites, TVA method.

Poster presentation

MODELLING OF VACANCY DIFFUSION IN CRYSTASTALLINE SILICON

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The quality of silicon single crystals, and the resulting electronic devices, depends on the distribution of microdefects formed by silicon vacancies and interstitials and by impurities, therefore it is important to study the dynamics of these defects. Molecular dynamics and Monte Carlo methods are important tools for studying the thermodynamic properties of processes like defect formation, and defect aggregation. In this paper we present a preliminary study on the diffusion of vacancy defects in Si crystals using the radial distribution function. The model we use is a molecular dynamics code based on the Stillinger-Weber potential for Si atoms. By comparing the radial distribution function (RDF) values for each atom with the average value, we were able to track the movement of the vacancy through the crystal and find its diffusion coefficient, for various temperatures. We have compared our results for the diffusion coefficient with other results from the literature.

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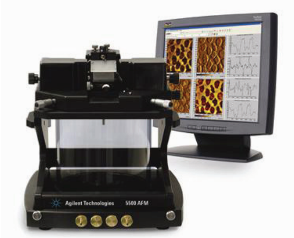

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