3RD BRAZIL MRS MEETING

October 10-13, 2004

SYMPOSIUM D:
NANO-SCALE STRUCTURAL CHARACTERIZATIONS OF MATERIALS

Symposium Organizers:

Guillermo Solórzano (DCMM/PUC RJ)

Joachim Mayer (Central Facility for Electron Micrsocopy, Aachen University)

SYMPOSIUM D

ATOMIC/NANOSCALE STRUCTURAL CHARACTERIZATION OF MATERIALS

Monday, October 11

Session Chair: Guillermo Solórzano (PUC-Rio)

8:20 Opening Remarks

8:30 Invited THE BEHAVIOUR OF GLASS IN CERAMIC MATERIALS
C. Barry Carter and Shelley R.Gillis, Dept. of Ch. Eng. & Materials Science, University of Minnesota, Minneapolis, MN, USA (D- I 12)

9:00 APLICATIONS OF EBSD TO TEXTURE DETERMINATION IN Cd Te THIN FILMS
A Pinto, and L.R. Cruz, Materials and Mechanical Engineering, Military Institute of Engineering- IME, Rio de Janeiro, Brazil (D – O 20)

9:15 TRANSMISSION ELECTRON MICROSCOPY OF IRON SILICID THIN FILMS
Karla Balzuweit, R.R. Andrare, D.R. Miquita, B.P. Moreira, W.N. Rodrigues and A.G. de Oliveira, Depto de Física - ICEX – UFMG, Belo Horizonte, MG , Brazil (D-O 16)

9:30 Invited ATOMIC STRUCTURE OF NOVEL OXIDE / SEMICONDUCTOR INTERFACES
Susanne Stemmer, Dimitri O. Klenov, Materials Department, University of California, Santa Barbara; Darrel Schlom and Lisa Edge, Penn State, USA (D-I 4)

10:00 COFFEE - BREAK

Session Chair: Joachin Mayer (Aachen)

10:30 Invited IMAGING ELECTROSTATIC AND MAGNETIC FIELDS USING ELECTRON HOLOGRAPHY
M. R. McCartney, Center for Solid State Science, Arizona State University, Tempe, AZ 85287 – 1704, USA (D-I 8)

11:00 Invited REAL TIME HRTEM OF ATOMIC-SIZE METAL NANOWIRES
Daniel Mario Ugarte, Depto de Física Aplicada, IFGW-Unicamp, and LNLS, CP 6192, 13084-971, Campinas, SP, Brazil (D-I 3)

11:30 NANOPARTICLE SELF-ASSEMBLY BY DEWETTING OF CHARGED POLYMER SOLUTIONS

C. A. Rezende, F. Galembeck, Instituto de Química, UNICAMP, Cidade Universitária Zeferino Vaz, s.n, CP 6154,13083-970, L.T. Lee, Laboratory Leon Brillouin (D-O 1)

11:45 EVIDENCE OF SiC BONDS FORMATION DURING GROWTH OF InGaP AND GaAs SEMICONDUCTORS
J. Bettini, M.M.G. de Carvalho, Laboratório Nacional de Luz Síncroton, Unicamp, Rua Giuseppe Máximo Scolfaro 10000, CP 6192, Campinas, SP, Brazil (D-O 13)

12:00 LUNCH

Session Chair: Fernando Galembeck (Unicamp)

14:00 Invited APPLICATION OF VALENCE ELECTRON ENERGY- LOSS SPECTROSCOPY AND PLASMON ENERGY MAPPING FOR DETERMINING MATERIAL PROPERTIES AT THE NANOSCALE
J.M. Howe and V.P. Oleshko; Dept. of Materials Science & Engineering, University of Virginia, Charlottesville, VA 22904 USA (D- I 7)

14:30 Invited SOLVING MATERIAL SCIENCE PROBLEMS BY ENERGY – FILTERED ELECTRON DIFFRACTION
C. T. Koch, Max Planck Institute for Metals Research, Heisenbergstr.3, 70569, Stuttgart, Germany (D-I 2)

15:00 FORMATION OF Sn EPITAXIAL ISLANDS AT SiO/ Si INTERFACES OF Sn^+ INPLANTED FILMS
P.F.P Fichtner, J.M.S. Lopes, F.C. Zawislak, Department of Physics, UFRGS, Porto Alegre, Brazil (D- O 19)

15:15 Invited ON GROWTH AND FORM OF LAYERED NANOSTRUCTURES: THE ROLE OF CURVATURE
H. Terrones, Advanced Materials Department, IPICYT, Camino a la Presa San José 2055, Col. Lomas 4. Seccion, San Luis Potos 70216, México (D-I 9

15:45 TRANSITION AND NOBLE METAL FILMS DEPOSITED ON METAL SUBSTRATES
S. S. Maluf, L.P. Pinheiro, P.I. Paulin-Filho, P.A.P. Nascente, UFSCar/DEMa, Via Washington Luiz km 235 CP 676, 13565-905, São Carlos, SP, Brazil; A.L.Gobbi, LNLS, Campinas; M.C.A. Fantini, USP/IF, Brazil (D- O 5)

16:00 COFFEE-BREAK AND POSTER SESSION

Wednesday, October 13

Session Chair: Guillermo Solórzano (PUC-Rio)

8:30 Invited NANOSCALE TEM CHARACTERIZATION OF DEFECTS IN STRONTIUM TITANATE
Wilfried Sigle, Zaoli Zhang, Sung Bo Lee, Behnaz Rahmati, Manfred Rühle Max-Planck-Institut für Metallforschung, Stuttgart (Germany)

9:00 Invited ELECTRON HOLOGRAPHY FOR ANSWERING NANO-QUESTIONS IN MATERIALS SCIENCE
Hannes Lichte, Center for Electron Microscopy de Dresden, Germany (D- I 13)

9:45 MAGNETIC PROPERTIES OF THIN FILMS PREPARED BY SOL - GEL PROCESS
L.M, Seara, N.D.S. Mohallen; Laboratório de Materiais Nanoestruturados, Departamento de Química, UFMG, Av. Antônio Carlos 6627, Belo Horizonte, MG, and M. Novak , UFRJ (D-O 17)

10:00 COFFEE - BREAK

Session Chair: Paulo F. Fitchner (UFRGS)

10:30 Invited BULK VERSUS THIN FILM – WHAT IS THE DIFFERENCE?
G. Van Tendeloo, EMAT, Groenenborgerlaan 171, B-2020, University of Antwerp, Belgium (D-I 5)

11:00 MICROSTRUCTURAL EVOLUTION UNDER HIGH STRAIN RATE PROCESSING OF Ti-ALLOYS
A. J. Ramirez, LNLS, CP 6192, 13084-971, Campinas, SP, Brazil; M.C.Juhas, OSU (D-O 9)

11:15 FATIGUE FREE BEHAVIOUR OF STRONTIUM BISMUTH TITANATE OBTAINED BY THE POLYMERIC PRECURSOR METHOD
G. Biasotto, A.Z. Simões, M.A. Zaghete and J.A. Varela, Unesp; Rua Professor Francisco Degni, s/n, CP 355, Araraquara, SP, Brazil (D-O 11)

11:30 Invited ATOMIC-SCALE CHARACTERIZATION OF SEMICONDUCTOR HETEROSTRUCTURES AND NANOSTRUCTURES
David J. Smith, Center for Solid State Science and Department of Physics & Astronomy, Arizona State University, Tempe, AZ 85257-1704, USA (D-I 11)

12:00 LUNCH

Session Chairs: Paula M. Jardim (PUC-Rio) and André Pinto (IME)

14:00 Invited SIGNIFICANCE OF NANOSCALE LAYERS IN WEAR AND CORROSION RESISTANCE OF STEEL
Joachin Mayer, Central Facility for Electron Microscopy, Aachen University, 52074, Aachen, Germany and Ernst Ruska, Centre for Microscopy and Spectroscopy with Electrons, Research Center Jülich, 52425, Jülich, Germany (D-I 10)

14:30 STRUCTURAL ANALYSIS OF A TiN COATING ON Cu-Ti-Cr ALLOY: STUDY OF THE INTERFACE
C. Carrasco, C. Villegas, M. López, C. Camurri, Departamento Ingeniería de Materiales, Universidad de Concepción, Edmundo Larenas 270, Concepción, Chile; N. Mingolo, CNEA, Argentina (D-O 12)

14:45 LOCALIZATION OF Mg+2 IN YEAST SUPPORTED ON BRAZILIAN CHRYSOTILE BY X-RAY MICROANALYSIS
F. Cassiola, M. Solveiora and P.K. Kiyohara, Laboratório de Biologia Celular e Molecular, ICB-USP, Av. Prof. Lineu Prestes, 1524, Ed. 1, 05508-900, São Paulo, Brazil (D-O 6)

15:00 Invited NEW DIRECTIONS IN CARBON NANOTUBE SCIENCE: CONTROLLED SYNTHESIS, ELETRONIC PROPERTIES AND NOVEL DEVICES USING B- AND N- DOPED SYSTEMS
Mauricio Terrones, Advanced Materials Department, IPICYT, Camino a la Presa San José 2055, 78216 San Luis Potosí, SLP, México (D-I 6)

15:30 STATISTICAL ANALYSIS OF TOPOGRAPHIC IMAGES OF NANOPOROUS SILICON AND MODEL SURFACES
J. B. da Silva Jr., E.A. de Vasconcelos, B.E.C.A dos Santos, E. F. da Silva Jr, W.M. de Azevedo, Grupo de Física de Semicondutores e Novos Materiais, Departamento de Física, UFPE, 50670-901, Recife, PE, and J.A.K. Ferire,UFC, Brazil (D-O 10)

15:45 OPTICAL ANALYSIS OF GaInP NANO/MICRO-WIRES
M. K. K. Nakaema, IFGW, Unicamp, CP 6165, 13083-970; M. Sacilotti, C.Dumas, P.Viste, CNR,Dijon; J.Decobert, H. Sik, G.Post, Alactel-Cit, Maracoussis, France, (D-O 18)

16:00 COFFEE-BREAK AND POSTER SESSION

INVITED PRESENTATIONS

D-I 2 SOLVING MATERIAL SCIENCE PROBLEMS BY ENERGY-FILTERED ELECTRON DIFFRACTION

C. T. Koch, Max Planck Institute for Metals Research, Heisenbergstr. 3, 70569 Stuttgart, Germany

The trend of modern material science to explore the “room at the bottom”, i.e. increasingly small length scales, requires new techniques and methods of characterization. Transmission electron microscopy (TEM) techniques, being able to probe sub-nanometer volumes for their structure, composition, optical, magnetic, as well as other properties may soon be the only choice for the characterization of tomorrow\'s materials. Despite the development of aberration correctors the spatial resolution of TEM imaging techniques is still well below the limit imposed by the scattering process of the fast electrons with the material. The resolution limit of electron microscopy could therefore be drastically enhanced if the problem of interpreting electron diffraction data directly could be solved. In this talk I will discuss different approaches to solve the dynamic as well as the kinematic phase problem and their application to solve material science problems: for example methods to obtain the structure of crystalline nano-volumes as well as nanometer thick amorphous inter-granular films.

D-I 3 REAL TIME HRTEM OF ATOMIC-SIZE METAL NANOWIRES

Daniel Mario Ugarte, : IFGW-UNICAMP and LNLS Depto. de Física Aplicada, Inst. Física Gleb Wataghin, UNICAMP, Cx. Postal 6165, 13083-970 Campinas SP and Laboratório Nacional de Luz Síncrotron, Brazil

Electrical properties of metal nanowires (NWs) attract great attention due to their quantum conductance behavior. NWs can be generated by stretching metal contacts; during the elongation and just before rupture, the conductance shows flat plateaus and abrupt jumps of approximately a conductance quantum. As both the NW atomic arrangement and conductance change simultaneously, it is difficult to discriminate electronic and structural effects. In this work, we have determined the NW structure was studied by time-resolved in situ experiments in a high resolution transmission electron microscope. Independent experiments based on a UHV mechanically controllable break junction (MCBJ) were performed to measure the conductance properties. Real-time HRTEM data showed that metal (Au, Ag, Pt, etc.) junctions generated by tensile deformation are crystalline and free of defects. The correlation between the observed structural and transport properties of NW points out that the quantum conductance behavior is defined by preferred atomic arrangement at the narrowest constriction. We have also carefully analyzed the formation of one-atom thick wires. Using HRTEM, we have observed that chains of atoms show very long interatomic distances (0.30-0.36 nm). An extensive study of gold atom chain structure reveals that even if the presence of contaminant atoms is assumed to be true, clean metal-metal bonds as long as 0.33 nm can still be derived from the available experimental data.

D-I 4 ATOMIC STRUCTURE OF NOVEL OXIDE/SEMICONDUCTOR INTERFACES

Susanne Stemmer, : D. O. Klenov, D. Schlom, L. Edge, Materials Department, University of California, Santa Barbara

Novel metal oxide films are currently being developed for future generations of Si based field-effect transistors as the SiO2 gate dielectric is reaching scaling limits. Device properties are controlled by the interface between the oxide and the semiconductor. Ultimately, interfaces between Si and these new gate dielectrics need to be atomically abrupt and epitaxial interfaces are attractive to study the fundamental properties of direct interfaces. In this presentation, we report atomic resolution observations of single-crystalline Si/LaAlO3 and Si/Sc2O3 interfaces. The atomic structure of the epitaxial interface between (001) LaAlO3 and Si directly from atomic resolution high-angle annular dark-field images obtained in scanning transmission electron microscopy. An unusual 3×1 interface reconstruction, in which every third La column is removed from the interface plane, is observed. The interface atomic structure is discussed in the context of electrically favorable interfacial bonding between the ionic oxide and Si. Epitaxial Sc2O3 with the cubic bixbyite structure on (111) Si show a high density of misfit dislocations, which completely relieve the lattice mismatch. A high-density of defects, likely antiphase boundaries, formed during growth as films nucleate with no unique arrangement relative to the Si lattice. The implications of these defects on the device properties are also discussed.

D-I 5 BULK VERSUS THIN FILM - WHAT IS THE DIFFERENCE ?

Van Tendeloo, EMAT, Groenenborgerlaan 171, B-2020 Antwerp University of Antwerp, Belgium

When a film is deposited epitaxially on a substrate, there will unavoidably be a misfit between substrate and film. This misfit can be positive or negative. Different structural possibilities to adapt to the misfit will be covered. In general, for very thin films the material will adapt itself to the structure imposed by the substrate. For larger thickness however, the influence of the substrate diminishes and above a critical thickness the material may adopt the structure of the bulk, when grown under similar conditions. Such a discontinuity in the film growth has been clearly observed when growing LCMO on a STO substrate. Apart from the classical misfit dislocations other ways of adapting to the misfit have been observed. Examples are periodic twinning, phase transition or oxygen non stoichiometry. We will show different examples where transmission electron microscopy (TEM) plays a crucial role in determining the structure and microstructure of a thin film. Structural changes will obviously induce changes in the properties of the thin films. This is e.g well illustrated for superconducting (La-Sr)CuO4 thin films grown on different substrates. The superconducting temperature varies from 0K to 50K, reaching values as high as the double of the Tc in bulk material. Other examples of perovskite based materials will also be presented.

D-I 6 NEW DIRECTIONS IN CARBON NANOTUBE SCIENCE: CONTROLLED SYNTHESIS, ELECTRONIC PROPERTIES AND NOVEL DEVICES USING B- AND N-DOPED SYSTEMS

M. Terrones, Advanced Materials Department, IPICyT, Camino a la Presa San José 2055, 78216 San Luis Potosí, SLP, México

The latest advances in the production and state-of-the art characterization of B- and N-doped carbon nanotubes and nanofibers is discussed. Particular attention will be focused on efficient self-assembly pyrolytic routes to large arrays (< 2.5 cm2) of aligned C, CNx and BxCyNz nanotubes (15-80 nm od and < 100 microns length). In general, these nanofibres do not easily break upon bending and may behave as shock absorbing fillers in the fabrication of robust composites. The electronic properties and the density of states (DOS) of CNx, BCx and BCN nanotubes using scanning tunneling spectroscopy (STS) will be presented. Using tight-binding and ab-inito calculations, we demonstrate that the presence of N and B are responsible for introducing donor and acceptor states near the Fermi Level. Novel applications of these doped materials will also be discussed in areas of chemistry (as chemical sensors), biology (as protein immobilizers) and electronics (metallic and semiconducting nanowires). It is clear that these materials possess outstanding properties when compared to pure carbon nanotubes, and it is foreseen that these systems will certainly revolutionize some aspects of nanotube science and technology, thus opening a vast field of experimental and theoretical research.

D-I 7 APPLICATION OF VALENCE ELECTRON ENERGY-LOSS SPECTROSCOPY AND PLASMON ENERGY MAPPING FOR DETERMINING MATERIAL PROPERTIES AT THE NANOSCALE

J. M. Howe, V. P. Oleshko, Department of Materials Science & Engineering, University of Virginia, Charlottesville, VA 22904-4745, USA

Measuring material properties at the nanoscale is critical to understanding the behavior of nanostructured materials. In this paper, we demonstrate a novel technique that allows direct determination and imaging of physical properties of individual nanoprecipitates and nanoparticles using energy-filtering transmission electron microscopy (EFTEM) combined with valence electron energy-loss spectroscopy (VEELS). We show that strong scaling correlations exist between the plasmon energy and elastic properties, hardness, valence electron density and cohesive energy. We apply these scaling relationships to characterize the elastic properties of metastable nanoprecipitates in a Ti-based structural alloy and the hardness of diesel-engine soot particles. We also discuss additional factors that need to be considered when using plasmons as a quantitative tool for nanoscale property measurement. The results show that VEELS has the potential to determine multiple solid-state properties of materials at the nanoscale, establishing a new capability for analytical electron microscopy. This research was supported by DOE.

D-I 8 IMAGING ELECTROSTATIC AND MAGNETIC FIELDS USING ELECTRON HOLOGRAPHY
M. R. McCartney, Center for Solid State Science, Arizona State University, Tempe, AZ 85287-1704, USA

Electron holography allows quantitative, high-resolution measurement of magnetic and electrostatic potentials for a wide variety of fascinating and technologically relevant systems. As semiconductor device parameters continue to decrease into the deep sub-micron range, electron holography can provide a unique imaging tool with its inherent two-dimensional, high-spatial resolution capabilities. Opportunities are available for in-situ modification of electrostatic and magnetic potentials. Application of magnetic fields using small excitations of the objective lens allows imaging of magnetic structure during hysteresis cycles for both thin films and patterned nanostructures. Examples to be discussed will include imaging of magnetic fields in magnetotactic bacteria and patterned magnetic nanostructures for magnetic random access memory elements. Electrostatic applications will include dopant profiling of source/drain regions in transistors as well as measurement of electrostatic polarization fields in an AlGaN/InGaN/AlGaN heterojunction diode.

D-I 9 ON GROWTH AND FORM OF LAYERED NANOSTRUCTURES: THE ROLE OF CURVATURE
H. Terrones, Advanced Materials Department, IPICYT, Camino a la Presa San José 2055, Col. Lomas 4. Seccion, San Luis Potos 70216, México

D-I 10 SIGNIFICANCE OF NANOSCALE LAYERS IN WEAR AND ORROSION RESISTANCE OF STEEL
Joachin Mayer, Central Facility for Electron Microscopy, Aachen University, 52074, Aachen, Germany and Ernst Ruska, Centre for Microscopy and Spectroscopy with Electrons, Research Center Jülich, 52425, Jülich, Germany

D-I 11 ATOMIC-SCALE CHARACTERIZATION OF SEMICONDUCTOR HETEROSTRUCTURES AND NANOSTRUCTURES
David J. Smith, Center for Solid State Science and Department of Physics & Astronomy, Arizona State University, Tempe, AZ 85257- 704, USA

D-I 12 THE BEHAVIOUR OF GLASS IN CERAMIC MATERIALS
C. Barry Carter and Shelley R.Gillis, Dept. of Ch. Eng. & Materials Science, University of Minnesota, Minneapolis, MN, USA

ORAL PRESENTATIONS

D O1 NANOPARTICLE SELF-ASSEMBLY BY DEWETTING OF CHARGED POLYMER SOLUTIONS
C. A. Rezende, F. Galembeck, Instituto de Química, UNICAMP, Cidade Universitária Zeferino Vaz, s.n, CP 6154,13083-970, L.T. Lee, Laboratory Leon Brillouin

D O2 CHEMICAL PROPERTIES AND STABILITY OF ALUMINUM OXYNITRIDE FILMS ON Si UNDER VACUUM AND O_2 ANNEALING
K. P. Bastos, G. V. Soares, L. Miotti, C. Driemeier, J. R. Baumvol, UFRGS, Instituto de Física, Laboratório de Superfícies e Interfaces Sólidas, Av. Bento Gonçalves, 9500, Bairro Agronomia, Porto Alegre-RS, CEP 91509-900 Brazil

Aluminum oxynitride films on silicon are candidates to replace the gate dielectric in the next generations of CMOS devices. In this work the effects of post-deposition rapid thermal annealing in vacuum and in dry O2 atmospheres on the stability of remote plasma-assisted nitrided aluminum oxide films on silicon are investigated. The areal densities of Al, O and N were determined by nuclear reaction analysis and their concentration versus depth distributions by narrow nuclear reaction resonance profiling, with subnanometric depth resolution. The chemical environment of N in nitrided aluminum oxide films on Si(001) was investigated by angle-resolved X-ray photoelectron spectroscopy. Two different bonding configurations were identified, namely N-Al and N-O-Al, suggesting the formation of the AlN and AlO2N compounds. Oxygen from the gas phase was incorporated in the AlON films in exchange for O and N previously existing therein, as well as in the near-interface region of the Si substrate, leading to oxynitridation of the substrate. The mobile N is partly lost by desorption from the surface and partly fixed by reacting with the network to form AlO2N. Low-energy ion scattering analyses revealed reduction in the migration of Si atoms from the substrate across the films as compared to non-nitrided Al2O3 films confirming that the presence of nitrogen improves the thermal stability characteristics of the AlON/Si structures in comparison with non-nitrided Al2O3/Si

D O3 ATOMIC TRANSPORT IN $LaALO_3$ GATE DIELECTRIC ON SI DURING THERMAL ANNEALING

L. Miotti, IF-UFRGS, Av. Bento Gonçalves, 9500 Porto Alegre

Lanthanum aluminum oxide is a high-k material candidate to replace SiO2 as the gate dielectric material in the next generation of CMOS devices. The thermodynamic stability of the interface LaAlO3/Si was investigated by ion beam analysis and X-ray photoelectron spectroscopy. Thermal annealing at 450-600oC in oxygen atmosphere promoted exchange of oxygen from the gas phase for oxygen previously in the film with no apparent diffusion of any other atomic species through the film. Rapid thermal annealing typical to dopant activation (1000 ^{o}C for 20 s) promoted Si diffusion into the film which was greatly increased with oxygen pressure. The presence of oxygen in the rapid thermal annealing ambient promoted film growth and La diffusion into this new grown layer. C-V characteristics of RuO2/LaAlO3/Si structure were investigated before and after thermal processing described above.

D O4 TEM MICROSTRUCTURE AND PHASE COMPOSITION STUDY OF AG:(BI,PB)2SR2CA2CU3OX COMPOSITE PRECURSOR POWDERS OBTAINED BY SPRAY PYROLYSIS

Composite particles of Ag:(Bi,Pb)-2223 stoichiometry were synthesized by spray pyrolysis from a mixed nitrate solution. This kind of composite powder has a great potential utilization as the precursor powder in technologies for obtaining superconducting tapes, bulks or foams. The preparation of a high quality precursor powder is the first critical step in the development of high-temperature superconducting devices. Aerosol decomposition routes, such as spray-pyrolysis, possess several advantages (chemically homogeneity, uniform submicron particles, etc.) over conventional solid-state techniques. The as-prepared and heat-treated (750oC /1 h and 780oC/3 h) powders were characterized by X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM) and Magnetic Susceptibility measurement. XRD quantitative phase analysis was carried out using the Rietveld method. For TEM analysis the samples were prepared by ultramicrotomy in order to enable the study of the particles internal structure. The as-prepared powder exhibited polycrystalline and multiphasic particles, containing the phases (Bi,Pb)-2212, Bi-2201, cuprate 14:24, CuO and Ag. The sample preparation by ultramicrotomy allowed the observation that some of these particles are hollow. After heat treatment the particles become manly Bi-2212 single-crystals. The magnetic susceptibility measurements showed that, altho ugh not detected by XRD and TEM analysis, traces of the phase Bi-2223 are already present after heat treatment.

D O5 TRANSITION AND NOBLE METAL FILMS DEPOSITED ON METAL SUBSTRATES

S.S. Maluf, L.M.P. Pinheiro, A.L. Gobbi, P.I. Paulin-Filho, M.C.A. Fantini, P.A.P. Nascente, UFSCar/DEMa, Via Washington Luiz km 235, Sao Carlos, SP, Brazil, Caixa Postal 676, CEP 13565-905.

Thin films of transition and noble metals deposited on metal substrates present interesting catalytic, electronic, and magnetic properties. In this work, thin films (thickness of 10 nm) of Ni, Cu, Pd, Ag, and Au were deposited on polycrystalline substrates of the same metals by sputtering. Atomic force microscopy (AFM), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS) were employed to characterize the morphology, structure, and composition of the samples. One of the main objectives was to check the possibility (or not) to form alloys. The combined AFM and XPS results showed that the Ni, Cu, and Ag films were oxidized, while the Pd and Au ones were in the metallic form. XRD results showed that the thin films of Cu on both Pd and Au substrates, Ni on Pd, and Pd on Au were amorphous. The XPS results indicated the possibility to form alloys in the Au/Cu, Pd/Cu, and Pd/Ag samples, in which the films are crystalline, as detected by XRD.

D O6 LOCALIZATION OF Mg^{+2} IN YEAST SUPPORTED ON BRAZILIAN CHRYSOTILE BY X-RAY MICROANALYSIS

F. Cassiola, G. M. M. Santelli, M. Silveira, P. K. Kiyohara, ICB-USP, Laboratório de Biologia Celular e Molecular, Av.Prof. Lineu Prestes, 1524, Ed. 1, São Paulo-SP, Brasil, CEP 05508-900

Yeast supported on Brazilian chrysotile has been described as an excellent biocatalyst for ethanol production, rendering an ethanol yield up to 26% higher than the free cells. Chrysotile is a fibrillar magnesium silicate (diameter around 50nm) abundant in Brazil. The fibrils were molded to the cells resulting in entrapment, until full cell surface coverage. Transmission Electron Microscopy investigation results showed that the fibrils are adhered only to the external cellular wall layer, and that no damage is caused to the cell wall structure. The entrapped yeast shows increased thermotolerance, and is active for at least 3 years of storage time in the absence of nutrients. Chrysotile flexibility and the geometry of the systems are responsible for the entrapment. Once entrapped, the cells enter in latency, in which they may use Mg+2 from the fibrils as a single life-supporting nutrient in extreme conditions. We are using TEM, combined with energy-dispersive X-ray microanalysis, to localize Mg ions, inside the cells, after their selective precipitation with K-pyroantimonate. Electron micrographs and X-ray spectra showed that fine precipitates of Mg+2 (diameter<100nm) were localized in cytoplasmic granules, generally near cell wall, and inside the vacuoles of the fibril entrapped cells. Soluble Mg ions supplied in the fermentation media have no similar effect on the uptake of Mg+2 by yeast. These results suggest that chrysotile would be serving as an important store for Mg+2.

D O7 MELTING BEHAVIOR OF THE SYSTEM LIF-GD0.5LU0.5F3

I. M. Ranieri, A. H. A. Bressiani, A. V. P. de Castro, S.L. Baldochi, S.P. Morato, N.D. Vieira Jr, IPEN, Center for Laser and Aplication, Av. Prof. Lineu Prestes 2242, São Paulo, SP, Brazil, CP11049, CEP 05422-970.

The main objective to propose the construction of the phase diagram of the system LiF-GdF3-LuF3 is to obtain crystals with congruent melting behavior. To determine the lutetium concentration that makes the system congruent, it was constructed a section of the phase diagram, where the mixture compositions were fixed at 50 mol% LiF: 50 mol% Gd1-xLuxF3 and 0<x<1. This composition was determined to be 50 mol% LiF: 50 mol% Gd0.5Lu0.5F3 at 800 0C. Afterwards the phase diagram of the system LiF-Gd0.5Lu0.5F3 was constructed. Fluoride powders were used to prepare the samples; the compounds were weighted and mixed in a mortar prior the measurements. The samples weighing around 50 mg were placed in open platinum crucibles without a reference material. The measurements were performed under a flux of purified helium, with heating rates of 10 and40 0C/min. To determine the phases present in each two-phase regions of the phase diagrams, two samples of each composition were melted u

nder a flux of hydrogen fluoride gas to obtain the phase equilibria. Powder X-ray diffraction and scanning electron microscopy were used to determine the phases present and to observe respective the microstructures, preliminary results will be presented.

D O8 MN K-EDGE X-RAY ABSORPTION SPECTROSCOPY STUDIES ON THE ELECTRONIC AND STRUCTURAL PROPERTIES OF LA1-XSRXMNO3 PEROVSKITES

F. M. M. Borges, R. Canha, D. M. A. Melo, C. N .Silva Jr., P. M. Pimentel, A. E. Martinelli, UFRN, Universidade Federal do Rio Grande do Norte, CCET, Departamento de Química, CP 1662, 59078-970, Natal/RN-Brasil

This work reports preliminary results on the electronic and structural characteristics of La1-xSrxMnO3 compounds (x = 0; 0.2; 0.4; 0.6) obtained by Mn K-edge X-ray absorption spectroscopy. The Fourier transform of EXAFS signal for all samples present the same radial distribution (RD) in the range of 1-7 Å, indicating that the local structure surrounding the Mn atoms must have been similar. The first peak, around 1.6 Å, corresponds to the nearest neighbor oxygen backscatter (first shell coordination). The intensity of this peak increased with the substitution of La by Sr. This fact can be attributed to a reduction of the Jahn-Teller (JT) distortion of the MnO6 octahedra upon Sr introduction. The smaller peak between 2.5-3.0 Å could be attributed to Mn – A (A = La / Sr) interactions. This peak shifted to higher R-values and intensities with increasing Sr content. These local effects could be explained in terms of the differences in the Sr and La ionic sizes (1.36 Å for La3+ and 1.44 Å for Sr2+) as well as to the larger structural disorder in the cation sublattice at lower Sr concentrations. The third peak, around 3 – 4 Å, was attributed to the Mn– O – Mn interactions. This peak showed a peculiar feature, extremely high amplitude for the Sr containing compounds corroborating to a reduction on the JT distortion. The Mn K-edge XANES spectra shifted to higher threshold energies with the introduction of Sr implying an increase on the Mn oxidation state upon doping.

D O9 MICROSTRUCTURAL EVOLUTION UNDER HIGH STRAIN RATE PROCESSING OF TI-ALLOYS

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Friction stir welding-FSW is a solid state process where a cylindrical tool with an extended pin is rotated and gradually plunged into the joint. The non-consumable, rotating tool, is translated along the joint, producing a weld. The combination of frictional heat and intense deformation produces a local forging/stirring effect, which develops a high integrity joint. FSW is now a commerciall joining technology for Al alloys. The possibility of producing solid state joints in plates has driven the expansion of this technology to higher temperature processing materials such as Fe, Ni and Ti alloys. The metallurgical and economic advantages associated with FSW of Ti have directed this study towards the relationships between the process and the resultant microstructure and properties. Mill and beta annealed 6 mm thick plates of Ti-6Al-4V alloy were FSW using a W-based tool. A detailed microstructure characterization was performed using SEM, TEM, STEM and XEDS. In both material conditions, the stir zone-SZ presented a colony structure with a refined prior beta grain size that developed during the cooling from above beta-transus-BT. The SZ beta grain size was controlled by extensive local deformation and short dwell time above BT. The TMAZ contained large amount of fine eq-alpha, which formed by recrystallization, and secondary alpha, which was identified by STEM. These results show how FSW can tailor modify the microstructure and properties of Ti alloys.

D O10 STATISTICAL ANALYSIS OF TOPOGRAPHIC IMAGES OF NANOPOROUS SILICON AND MODEL SURFACES

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Light emission from silicon nanostructures has been an extremely active research area field in the past decade, mainly due to its potential to integrate different technologies leading to the development of silicon-based optoelectronic devices. Silicon porosification is one of the possible techniques to obtain light emission from silicon. However, porous silicon morphology is notoriously difficult to describe quantitatively. In this work, we investigate improved methods to describe quantitatively nanoporous silicon morphology and its correlation with electrical and optical properties. We performed first-order (surface height distribution, 2nd, 3rd and 4th-order moments), as well as second-order statistical analysis (height-height correlation function) of AFM images of nanoporous silicon fabricated by two different methods: (1) reaction-induced vapor-phase stain etch and (2) electrochemical. We also simulated AFM images by generation of various model surfaces. From the height-height correlation plots, we were able to identify the growth method and to observe the onset of the self-affine character. The analysis of the model surfaces reveals that the AFM images cannot be adequately described by simple algorithms. Linear combination and smoothing methods are necessary to reproduce the self-affine character of the surfaces.

D O11 FATIGUE FREE BEHAVIOUR OF STRONTIUM BISMUTH TITANATE OBTAINED BY THE POLYMERIC PRECURSOR METHOD

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In recent years ferroelectric materials have generated a lot of interest from an application as well as from an academic point of view. Due to fast switching speed and low operating voltage, ferroelectric thin films have been integrated into silicon integrated circuits to provide high speed, density, write/read endurance and hardened nonvolatile ferroelectric randon access memories (FeRAMs). Bismuth layered perovskites belonging to Aurivillius family have received a lot of attention as new ferroelectric materials with excellent fatigue-resistant properties. These materials are constructed by stacking n perovskite units between layers. The films were obtained by the polymeric precursor method and deposited by a spin-coating on a platinum coated silicon substrates. They were characterized by x-ray diffraction, atomic force microscopy (AFM), scanning electron microscopy (SEM) and electric properties. It was noted that ferroelectric strontium bismuth titanate thin films with a well developed hysteresis loop were produced by a chemical solution deposition method. Although the films present a fatigue free behaviour, it´s remanent polarization should be increased aiming to application in nonvolatile ferroelectric random acess memories.

D O12 STRUCTURAL ANALYSIS OF A TiN COATING ON Cu-Ti-Cr ALLOY: STUDY OF THE INTERFACE

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D O13 EVIDENCE OF SIC BONDS FORMATION DURING GROWTH OF IN GAP AND GAAS SEMICONDUCTORS

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InGaP layers, lattice-matched, to GaAs have shown interesting characteristics from the point of view of device applications. InGaP has similar values of band gap energy and higher valence band discontinuity. This makes InGaP a very suitable material for laser and Heterojunction Bipolar Transistor (HBT) fabrication. In order to achieve this goal, InGaP layers with good crystal quality and morphology are necessary. Doping these layers is also an important issue when device applications are considered. The presence of dopant atoms may affect the growth, inducing modifications of the crystal quality.

Here, we have studied the effect of high Si doping on the crystal quality of InGaP and GaAs layers grown by CBE. Electrical properties have been derived from Hall Effect measurements, Scanning Electron Microscopy (SEM) has been used for morphologic characterization and, Transmission Electron Microscopy(TEM), X-Ray diffraction and Photoluminescence at 77K were used to estimate the crystal quality.

Our results suggest that during the growth of heavily Si-doped InGaP (or GaAs) layers, residual C is also incorporated as an acceptor. This leads to the formation of SiC bonds or nanocrystals, what causes large density of defects in these films.

D O15 IGH RESOLUTION ELECTRON MICROSCOPY OF POLYMER/FILLER DERIVED CERAMIC

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Ceramic materials obtained from the pyrolysis of a polymer precursor have gained growing interest due to their unique combination of low temperature processing, versatile shaping and the formation of a novel material class like nano structured or molecular composite. During pyrolysis polissiloxane precursor releases gaseous products and is converted gruadually to a amorphous silicon oxycarbide (SiOC) phase and an excess of free carbon. In this work high resolution electron microscopy was used to study the nanostructure of a ceramic material obtained from the pyrolysis of polissiloxane/active filler mixture.

MICROSCOPIA ELETRÔNICA DE TRANSMISSÃO EM FILMES FINOS DE SILICETO DE FERRO

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Embora o silício seja a principal escolha para dispositivos aplicados à microeletrônica, o desenvolvimento de dspositivos opto-eletrônicos integrados baseados neste elemento é impedido pelo fato do silício ser um pobre emissor de luz, decorrente de um bandgap indireto do silício. Este problema motivou e ainda motiva numerosas tentativas de desenvolver estruturas baseadas em silício com boas características de emissão de luz. Nos últimos 12 anos foram investigados principalmente as propriedades estruturais, elétricas e ópticas do FeSi2. Esses trabalhos destacaram os silicetos como promissores candidatos para fabricação de dispositivos opto-eletrônicos. Em particular a fase semicondutora (beta-FeSi2) despertou maior interesse devido às suas características: ?gap? de natureza direta com Eg ~ 0,85 eV (1,46 ?m), próximo ao mínimo de absorção das fibras ótica atuais; o fato de ser não tóxico e composto por dois dos elementos mais abundantes no planeta e apresentar um coeficiente de absorção óptico 50 vezes maior que o do Si cristalino na faixa do infravermelho próximo. Foram obtidos filmes finos de beta-FeSi2 através da técnica de RDE(Reactive Deposition Epitaxy). Estes filmes estão sendo investigados quanto aos mecanismos de crescimento e morfologia através de Microscopia Eletrônica de Transmissão e Difração de Raio-X, entre outras técnicas. Serão apresentados resultados preliminares em relação à morfologia e estrutura destes filmes.

D O17 MAGNETIC PROPERTIES OF THIN FILMS PREPARED BY SOL-GEL PROCESS

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Nanostructured particles have been researched due to their high surface/volume ration, which gives them unique properties diverse from those of similar polycrystalline materials. The use of an inorganic matrix as a host for this nanocrystalline particles has been demonstrated to be a form of getting uniform size distribution. Silica has been used as a matrix material due to its high thermal and chemical stability. In this work, cobalt ferrite thin films and magnetic composite thin films formed by cobalt ferrites disperse in a silica matrix were prepared by sol-gel process using metallic nitrates as a precursor of the ferrite and tetraethylorthosilicate (TEOS) as a precursor of silica. The films were prepared and deposited on glass and quartz plates using the dip-coating process and they were adherent, transparent, homogeneous and free of microcracks. Film thickness and processes parameters such as ferrite concentration, dipping velocity, solution viscosity and heat treatment temperature were correlated and associate with the coating morphology and magnetic properties. The morphology was studied by atomic force microscopy and the magnetic behavior by a sample vibrant magnetometer. Porosity and refraction index were estimated by spectroscopy UV/visible.

D O18 OPTICAL ANALYSIS OF GAINP NANO/MICRO-WIRES

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Semiconductor nano/microwires is an interesting system to develop novel and more efficient optical quantum devices. Sceptre like nano/micrometer sized structures composed of GaInP rods with a metallic balloon at the top (mostly In) were grown on polycrystalline InP substrates by MOCVD technique with TMGa as the only source. Optical techniques were used to analyze these sceptre like structures. We analyzed several rods including the top balloon. Raman scattering measurements performed along the rod length present the typical TO and LO GaInP alloy features. Considering strain free rod structures, we inferred from the TO GaP-like peak position that the alloy composition varied along the rod length in a large range of Ga content (~ 50 to 80%). The Ga concentration is usually larger near the balloon and shows a tendency to decrease, as we get closer to the rod basis. The rods present a knot-like structure adjacent to the balloon where the Raman signal is much stronger than the signal from the rod below it. We speculate that this enhancement could be associated to the effect of surface enhancement of Raman scattering related to the presence of a metal around this region. We also observed a small signal attributed to GaInP alloy on some balloons. The Raman spectra from the surface also present GaInP alloy and InP substrate vibration modes.

POSTER PRESENTATIONS

DP 1 THE SOLID STATE NMR EVALUATION OF POLYPROPYLENE NANOCOMPOSITE

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The solid state nuclear magnetic resonance spectroscopy (NMR) is one of the best methodology to analyse polymeric nano material in nanometric scale. Different NMR techniques can be applied o the material to investigate the molecular mobility, sample hetrogeneity and components interaction. In this context the present work have chosen solid state NMR techniques to evaluate the nanocomposite formed by polypropylene and organophilic brazilian montmorillonite. The techniques used were magic angle spinning (MAS)- to evaluate the foramtion of a mobile region; cross polarization magic angle spinning (CPMAS)- was used to analyse the sample homogeneity and the proton spin-lattice relaxation time in the rotating frame (T1Hrho) was measured to determine the interaction between both nanocomposite components. From the results it was evaluated that the mobile region was formed by polypropylene chains, because the polymer matrix is very big. The CPMAS results showed that the sample componnents present some homogeneity. The proton spin-lattice relaxation parameter showed that an interaction between both nanocomposite componnents was determined. Analysing these result we can conclude that solid state NMR spectroscopy is a powerful methodology to be used to study the polymeric nanocomposites in details.

DP 2 MAGNETIC PROPERTIES OF ND-FE-RU-B INTERMETALLICS

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Although high coercivities are observed in sintered carbides, an opposite behavior is observed for the borides. The first studies of rare-earth-transition-metal carbides were reported about two decades ago. In view of the fact that the carbides are less stable
than the borides, usually they are not found in as-cast alloys. A full description of the role of different substituents is crucial to understand the physical origin of the intrinsic magnetic properties in these compounds. It has been pointed out before that, among the six inequivalent crystallographic Fe sites, i.e., 16k1, 16k2, 8j1, 8j2, 4e and 4c, as a whole, the k-sites are preferred by the substituent for transition metals, and a preference of Fe for the j-sites was found, which favors larger anisotropy field values. For cobalt, as a substitution for Fe, a deviation from random occupation has already been reported. In this work we study the effect of the substitution of Ru atoms for Fe on the magnetic properties of the tetragonal 2:14:1 form of Nd-Fe-Ru-B alloys. The
Mossbauer parameters derived from fitting the six sextets assigned to the six different iron sites can give information about preferential site occupation of solute atoms. The cell volume decreases with the substitution and the shrink of the lattice is greater in the c direction of the tetragonal unit cell.

DP 4 LOW-FIELD MAGNETIZATION STUDIES OF ND-FE-CO-RU-B INTERMETALLICS

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Since the development of the new generation of supermagnets based on the R-T (rare earth-transition metal) intermetallic compounds few decades ago a large effort has been dispensed to improve their magnetic properties. As an attempt to enhance the intrinsic magnetic properties of borides various substitutions for Fe have been investigated. Among them the 3d metals have been intensively studied in order to clarify the role of the 3d sublattice in the magnetic behavior of these compounds. The Co substitution increases the Curie temperature of alloys for $0 < x < 1$. However, the spontaneous magnetization decreases, owing to the lower value of the cobalt moment. The anisotropy energy which originates from the transition metal sublattice is of the same order of magnitude in these compounds. The cobalt substitution also leads to a significant decrease in the annealing time needed to harden magnetically the ingots of Nd-Fe-C carbides. The combined addition of high-melting and low-melting 3d metals to the intergranular region has been observed to enhance the coercivity of Nd-Fe-B magnets. The addition of ruthenium reduces the Curie temperature, the anisotropy field and the magnetization. In this work we investigate the composition dependence of the
intrinsic magnetic properties of Nd-Fe-Co-Ru-B alloys, to obtain a description of the contribution of the transition metals sublattices to the overall magnetization in these compounds. This can contribute to the understanding.

DP 5 PRECIPITATION OF NiBe IN THE GRAIN BOUNDARY OF SUPERMARTENSITIC STAINLESS STEELS CONTAINING BERYLLIUM

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Supermartensitic stainless steels (SMSS) are a new class of stainless steels showing better mechanical strength, corrosion resistance and weldability when compared to the conventional martensitic stainless steels.The SMSS steels is based in the system Fe-Cr-Ni-Mo, containing low of carbon, nitrogen and sulphur. In the present work we report on the Beryllium alloying effect on the microstructure of SMSS, for as-rolled condition. Microestrutural characterizations was accomplished using optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to evaluate products of austenite transformation under cooling rates simulating water quenching, air quenching and verylow cooling. The formation precipitate interphasic of NiBe (dimension of 10nm), at grains boundary of the original austenite after tempering heat treatment. The final microstructure obtained after heat treatment, showing martensite matrix. Mechanical properties were evaluated by of hardness, microhardness test, tension and impact tests. Results show that studied stainless steels grades can be fit in the standard specifications of the SMSS.

DP 6 STRUCTURAL CHARACTERIZAZION OF KAOLINITE/ POLY(VINYLPYRROLIDONE) NANOCOMPOSITE

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Manufactures fill polymers with particles in order to improve the stiffiness and toughness of the materials, to enhance their barrier properties, their resistence to fire and ignition. Nanocomposite are new class of composites, that are particle filled polymers for which at least one dimension of the dispersed particles is in the nanometer range. In this work, kaolinite/ poly(vinylpyrrolidone) nanocomposite was obtained by direct intercalation of PVP into kaolinite by a displacement method (using DMSO as intermediate). Kaolinite was submitted to SEM, XRD, XRF, BET, Malvern, TGA/DSC and FTIR. The kaolinite-dimethylsulfoxide (K-DMSO) intermediate compound and kaolinite- poly(vinylpyrrolidone) nanocomposite (K-PVP) were characterized by SEM, XRD and FTIR. SEM analysis showed varing degrees of imperfection, rounded edges and corners of the kaolinite cristalite.

DP 7 MICROSTRUCTURAL CHARACTERIZATION AND CORROSION RESISTANCE OF AISI 420 MARTENSITIC STAINLESS STEEL LASER SUPERFICIAL TREATED

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Surgical instruments have been made of stainless steel due to its high corrosion resistance. However, corrosion problems can cause damage in these instruments. Some theories indicate that body fluids and chemical products used in the cleanliness is the cause of corrosion. In present study, samples of the AISI 420 martensitic stainless steel, used in fabrication of surgical instruments, were submitted to laser superficial treatments applying different processing parameters, in order to analyse the phases transformations during rapid solidification proportionate by treatment. The microstructures obtained were characterized by Optic Microscopy, Scanning Electron Microscopy, X-Ray Diffraction and Vickers Microhardness. After characterization, the samples were submitted to electrochemical tests using enzymatic detergents as electrolytes in order to analyse the corrosion resistance of AISI 420 steel after laser treatments.

DP 8 CHARACTERIZATION OF NANOCRYSTALLINE BA3CO0.9CU1.1FE24O41

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Z-type barium hexaferrite is promising for application up 100 MHz in high frequency electronic devices, which require high initial permeability, great resistivity, low magnetic and dielectric losses and good thermal stability.
In this work, the nanocrystalline Ba3Co0.9Cu1.1Fe24O41 was obtained by citrate sol-gel process under inert atmosphere. Cu was incorporated into the structure of Ba3Co2Fe24O41 and consequently, a relation between magnetic properties and low temperature sintering was achieved.
X-ray diffraction (XRD), X-ray fluorescence (XRF), TGA/DTA and scanning electron microscopy (SEM) were used to characterize the synthesized material. Magnetic properties were evaluated by using vibrating sample magnetometer

DP 10 STRUCTURAL STUDY OF COPPER ALLOYS CONTAINING MAGNETIC NANO PARTICLES

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In recent years, a number of studies have been carried out with different techniques on the properties of magnetic and nanostructured alloys. In particular, the phenomenon of giant magnetoresistance (GMR), given its promising capabilities for information storage systems and sensor technology. In the present study, the precipitation behavior of Cu-1% Co, Cu-2% Co and Cu-4% Co alloys under isothermal annealing was investigated by means of TEM and SEM. The microstructure of the polycrystalline alloy was found to be equiaxial. Coherent Co particles of 10 nm in size were randomly formed since the early stage of the precipitation processes. Upon isothermal aging, it was observed the developed of colonies of discontinuous precipitation products. The coexistence of nano-scale coherent precipitates together with micro-scale incoherent precipitates was observed after long aging time, forming well developed faceted crystals. Depending upon the thermal conditions, the generations of domains in the order of 100 nm, within the matrix grains of 100 mm in average size was also observed. The substructure, however, requires thorough interpretation. Further investigations are directed towards the relation between GMR and nanoscopic phases in Cu-Co alloys

D P11 STRUCTURAL ANALYSIS OF THE CO7-XNIXSB2O12 COMPOUNDS USING RIETVELD

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Crystal structure of the single-phase of Co7-xNixSb2O12 (x = 0,1, 2, 3 and 4) powders synthesized by polymeric precursor method and heat-treated at 1100oC were characterized by Rietveld refinement with X-ray diffraction data. For these refinements, the cations distribution was kept constant, where it was supposed that the Ni2+ ions substitute Co2+ ions only in the octahedral sites. It is not possible to determine the cation distributions because Ni and Co is almost identical for the copper radiation used in this experiment. The structural analysis revealed that cell parameter a decrease linearly as a function of the amount of nickel introduced in the structure (x) with correlation factor R2 =0.960, denoting that the dependence of the lattice parameter with x obeys Vegard's law. The oxygen positional parameters, u, for Co7-xNixSb2O12 phases, also obtained in the refinement, show a linear dependence in relation to x with a correlation factor R2 equal to 0.998. The increased in u values induced by cobalt substitution with nickel, can be interpreted in terms of the ionic radii of the cations. From the unit cell parameter a and the oxygen position parameter u, the average interatomic metal-oxygen distances in the tetrahedra, Dt(Co-O) and octahedra, Do(Co-O) were esteemed and for the spinel phase Co7Sb2O12, are slight different of those reported by Poix.

P12 STRUCTURAL ANALYSIS OF A TIN COATING ON CU-TI-CR ALLOY: STUDY OF THE INTERFACE TI-NON FERROUS ALLOY

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The crystalline structure of a TiN thin film deposited on a ternary copper alloy by means of DC planar magnetron sputtering was analyzed. Between the TiN coating and the substrate, a thin pure titanium film was deposited to obtain a greater adhesion among them. The samples were analyzed by means of transmission electron microscopy to determine the structure of each one of the components as well as the lattice distortion in the Cu-Ti interface. The obtained results are complemented with previous analyses of X-rays diffraction, where the lattice parameters of each one of the studied components were determined plus the respective residual stresses. Analyzing the obtained data, this study shows that there is an agreement between the lattice distortion and the measured residual stresses, and relates in addition the theoretical crystalline coherence with the experimentally calculated misalignment, by means of electron diffraction, in the interface titanium - non ferrous substrate.

D P13 SYNTHESIS AND CHARACTERIZATION OF $CaBi_4Ti_4O_{15}$ THIN FILMS OBTAINED BY THE SOFT CHEMICAL METHOD

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CaBi4Ti4O15 (CBTi144) thin films were deposited on Pt/Ti/SiO2/Si substrates by the soft chemical method. The 700°C annealed thin film was a single phase of layer-structured perovskite and showed random orientation. The dielectric constant and loss factor were 360 and 0.037, respectively, at 1 MHz. The thin film exhibited a P–E hysteresis loop with remanent polarization (Pr) and coercive electric field (Ec) equal to 12 mC/cm2 and 77 kV/cm, respectively. All the capacitors showed good polarization fatigue characteristics at least up to 1x1010 bipolar pulse cycles and excellent retention properties up to 1x104 s.

D P14 THE ELECTRON MICROSCOPY FACILITY AT THE LNLS

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The Electron Microscopy Laboratory (LME) is one of the facilities of the Lab. Nacional de Luz Síncrotron (LNLS). Equipment installed at the LME: a) Low Vacuum Scanning Electron Microscope (SEM) with microanalysis and crystallographic mapping capabilities; b) Field Emission SEM (FEG-SEM); c) 300 kV High Resolution Transmission Electron Microscope (HRTEM, 1.7 Å Point Res.) with TV Camera, CCD Camera and X-ray Si(Li) detector; and d) Complete sample preparation laboratory. A simple procedure allows access to the LME instruments, firstly a research project must be submitted for evaluation of viability and relevance; subsequently the training microscope sessions are scheduled. It is important to remark that EM is a routine characterization tool and the researchers have to operate the microscope by themselves. For this purpose, a training period may be necessary, which may vary from 1-2 weeks for a SEM to 2-4 months for the HRTEM. Our staff addresses a great effort to the formation of human resources in order to allow inexperienced Users to become capable of acquiring and interpreting data for their research. Since its installation in 1999, the LME has trained more than 300 Users. In 2003, the number of research projects was 36 in the HRTEM, 16 in the FEG-SEM and 48 in the LV-SEM. An expansion of the LME is at present being implemented, by the addition of a 200 kV FEG-TEM oriented for nano-analysis and Electron Energy Loss Spectroscopy (instrument funded by a FAPESP grant).

D P15 THE EFFECT OF TUNGSTEN ADDITION IN THE STRUCTURAL AND MICROSCTRUCTURAL PROPERTIES OF SrBi2(Nb0,5Ta0,5)2O9 OBTAINED BY THE SOFT CHEMICAL METHOD.

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Pure and tungsten doped SrBi2(Nb0,5Ta0,5)2O9 (SBTN) powders and thin films were prepared by the soft chemical method. The Rietveld analyses of the SBTN powders shown that the addition of tungsten reduces the crystallization temperature of the system. Besides that, it was found a larger amount of SBTN phase in the system doped with tungsten. The thin films were deposited on Pt/TiO2/SiO2/Si substrate and annealed at 700ºC for 2 hours. The microstrucuture of pure and doped SBTN films was investigated by SEM and AFM techniques. It was found that the films are homogeneous and free of defects. The AFM analysis shown grains with spherical form for the SBTN doped with tungsten with size of about 170 nm.

D P16 MERCURIC IODIDE THIN FILMS DEPOSITED BY SPRAY PYROLYSIS

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Mercuric iodide (HgI2) crystals have been used in the past to construct detectors for X- and γ-rays. More recently, attention has been focused on the production of thin films aimed for the development of digital imagers for medical applications. This material has a melting point at 237ºC, a boiling point at 351ºC, and a phase transition at 127ºC. Bellow 127ºC its structure is tetragonal (called α-HgI2) and its color is red. Above 127ºC the material presents an orthorhombic structure (called β-HgI2), and its predominant color is yellow, thus being called also yellowHT in the literature. The aim of this work is the investigation of the production of HgI2 thin films using the spray pyrolysis technique. The salt and its solvent (water or ethanol) are directed towards the heated substrates (glass) by a carrying gas (N2). When the solution reaches the heater substrate, the solvent evaporates and a thin film is formed. We observed that the yellow phase can be obtained even below 127ºC if the deposition occurs too fast. This material is called yellowM in the literature (M stands for metha-stable). We also managed to obtain yellowish and reddish thin films depending on the solvent, and the substrate temperature. The work discusses the final structure of the obtained thin films and its correlation with the fabrication conditions using X-ray diffraction, Scanning Electron Microscopy and Energy Disperse Spectroscopy. This work was funded by CAPES, CNPq and FAPESP (01/08221-9).

D P18 ON THE ORDERING PROCESSES IN SUPERALLOY 59 (NI-CR-MO) UPON AGING

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Nickel base alloy 59(59%Ni, 16%Mo,23%Cr and 1%Fe) was designed in the nineties to be highly resistant to corrosion. Indeed its high content guarantees the alloy applicability under oxidizing atmospheres while molybdenum plays the same role under reducing environments. The alloy has been successfully applied in pollution control equipment and in chemical and petrochemical industries. However, problems related to pit corrosion within the heat affected zone of welded joints were observed. Measurements of electrical resistance have revealed that this localized corrosion is associated with a considerable reduction in the electrical resistivity of the alloy. To investigate this phenomenon, isothermal aging treatments were carried out at 700°C and 900°C for 1, 10 and 100 hours. Micro and nanostructural evolution was studied by means of optical, scanning and transmission electron microscopy, making use of EDS and EBSD techniques. Electrical resistance and microhardness measurements were also performed. The results indicate that an ordering process, which takes place during aging, is responsible for the large variations in the electrical resistivity and microhardness of the material. An SRO (short range ordered) state was identified by the presence of {1,1/2,0} reflections, typical of atomic rearrangements on the {4,2,0}f.c.c planes in the Ni-Cr-Mo system. The presence of LRO (long range ordered) precipitates was also detected.

RETROGRESSION AND REAGING OF ALLOY 8090 (AL-LI-CU-MG-ZR)

D P19

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Alloy 8090 (Al-Li-Cu-Mg-Zr) has been used for aeronautic and aerospace applications, in virtue of its high specific stiffness. The purpose of this work is to evaluate the microstructural stability of the alloy when submitted to heat treatments of retrogression and reaging at different temperatures and different time intervals. Characterization of the morphology and stability of the second phases was carried out by scanning electron microscopy (SEM), making use of the electron backscattering diffraction (EBSD) technique. Transmission Electron Microscopy (TEM) was also used for this purpose in virtue of the nanometric size of the second phases precipitated in the alloy. It was noted that the alloy exhibits a remarkable stability, not only in regard to its polycrystalline composition but also to its microstructure. The deformation texture introduced in the alloy due to its fabrication process was found to persist after the retrogression treatment. In addition, the evolution of precipitation stages did not very considerably until peak aging was reached. The main phases observed in the alloy were the phases δ' (Al3Li), β' (Al3Zr) and T1 (Al2CuLi). The tensile results indicated the occurrence of Portevin-Le Chatelier effect for the alloy in the as-received and short time reaged conditions. This dynamic effect results from the interaction of dislocations with solute atoms as well as second phases particles.

D P20 CHARACTERIZATION BY X-RAY DIFFRACTION OF THE ZINC OXIDE OBTAINED BY PECHINI METHOD

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Zinc oxide is a semiconductor of the family II-VI. It shows interesting eletric properties and for this reason it's used in eletronic industries to manufacture of varistors. The advantages of zinc oxide varistors are your crystalline grains with homogeneous and controled microstructure. This control is obtained by correct processing of ceramic powders. The tradicional method of attainment of ceramics by mecanic blending and/or simultaneous milling of oxides not always leads to a intimate mixture of components. Other significant aspect is the precursor powders generally have reduzed superficial area, involving a low reactivity, requiring high temperature of sinterization. The goal of this article is the production of zinc oxide, from the Pechini Method, in different temperatures of calcinations. The product of the reactions was analised by DRX to estimate the formation of the desired phase, size of crystallite, degree of crystallinity and the parameter of lattice. The results showed the attainment of nanometric powders of the zinc oxide monophase and the parameters of lattice were compatible with the values found in literature. The medium size of the crystallite obtained by Scherrer formula and the relative degree of the crystallinity of the specimens showed solid subjection with the temperature of calcination. The variation in temperature of calcination was showed like an interesting mean in the management of the characteristics of the powders obtained by Pechini Method.

D P21 SURFACE MODIFICATION OF BIOMATERIALS IMPLANTS BY USING ND:YAG LASER BEAM IRRADIATION IN DIFERENTS ENVIRONMENT

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Titanium has been used in biomedical and dental implants. In the recent years, interest has been shown to modify the structure and composition of its surface in order to improve the osseointegration. Surface modifications of mettalic compounds through laser irradiation is a interesting technique due to its high spatial resolution, velocity and reproducibility, despite of being a clear process. However, for these applications it is important to establish the correct relationship between the laser beam parameters and surface morphology and composition. The aim of this work is to study Ticp surface modification by a Nd:YAG laser beam in O2, N2, air and argone atmosphere. The surface treatments were performed with a Digilaser DML-100 - Violino 10 (l = 1064nm) laser system operating in pulsed mode at 35 kHz repetition rate. The samples were investigated by a scanning electron microscopy and by X-ray diffractometry analyses. High surface area materials were obtained and the presence of nanoparticles on its surface was observed. The quantitative phases analyses indicated the presence of Ti (substrate), TiN, Ti2N and TiO2 (rutile and anatase) on the samples. These surface phases are important to improve the wetness property. In this study better conditions were established in order to achieve appropriate roughness and wetness properties leading to an increase of surface energy and contact area. These characteristics are important to improve the osseointegration mechanism.

D P22 THE EFFECT OF LANTHANUM ADDITION ON THE STRUCTURAL, MORPHOLOGIAL AND ELECTRICAL PROPERTIES OF THE BISMUTH TITANATE CERAMICS

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Bi4Ti3O12 (BIT) is a ferroelectric material with great potential in the development of technological devices, exhibiting piezoelectric, pyroelectric and electrooptic properties. Pure and lanthanum doped ceramics were prepared by the polymeric precursor method in the following stoichiometry, Bi4-xLaxTi3O12 with x ranging from 0 to 0.75. The powders were calcinated at 850 °C for 4 hours and milled in isopropilic alcohol media. Thermogravimetric and differential scanning calorimetric analyses were used to verify the decomposition temperature of organic material and the formation of the crystalline phase. It was noticed by Rietveld analyses that the addition of lanthanum leads to a change in crystal structure from orthorrombic to tetragonal. The powders were isostatically pressed at 200 MPa and sintered in a conventional furnace with heating rate of 10°C/min up to 1000°C. Scanning electron microscopy was used to verify the surface of the sintered pellets where it was noted a plate-like morphology, which is typical for this system.

D P23 THE EFFECT OF THERMAL TREATMENT ON THE PROPERTIES OF SOL-GEL RuSn-SILICA CATALYSTS

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This work intends to evaluate RuSn-doped silica glasses obtained by sol-gel method as a heterogeneous catalyst for hydrogenodechlorination reactions. The humid gel obtained from a starting solution containing tetraethylorthosilicate and RuSn chloride or a RuSn organometallic compound was thermally treated at 110°C to obtain the xerogel and dried at various temperatures in various kind of atmosphere such as air, He and N2. The structural, textural and morphological changes were studied by powder X-ray diffraction, scanning electronic microscopy, nitrogen adsorption, Mössbauer spectroscopy and thermal analyses. The composites heated at 500°C showed high surface area with strong mesoporous contribution. These samples showed high activity for various catalytic reactions like as hydrogenodechlorination of CCl4, hydrogenation of olefine among others.

D P24 POLARON PROPAGATION DUE TO DOPED CONJUGATED POLYMERS

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Polarons are charge carriers generated by photo excitation or doping in conjugated polymers. The purpose of the present work is to study the polaron propagation when there is doping in conjugated polymers. The dynamics of structural polaron-type defects in conjugated polymers is analysed using a modified tight-biding extended to include the effects of an external electric field and a type site impurity (by radical parameter terms in Hamiltonian). The electric field is used to accelerate charged polarons in polymers and it is introduced in terms of a time-dependent vector potential. We use the Pariser-Parr-Pople (PPP) model combined with the Su-Schieffer-Heeger (SSH) model of electron-lattice. The dynamics is analyzed by numerical resolution of the equations of motion within the unrestricted Hartree-Fock approximation. Polarons on impurity and polaron-impurity collisions are considered. When the polaron-structure is around the impurity site a charge oscillation can be observed. Nevertheless when there are collisions between the polaron and the impurity, the polaron-structure can pass, be trapped or be reflected. These effects are determined by straint of the radical parameter and by the electric field intensity. Then the polaron effects can be analyzed by an effective potential. Therefore the effective potential determine the polaron behavior for each case, if the polaron pass, be trapped or be reflected by an impurity.

D P25 CHARACTERIZATION OF A TiO_2 NANOPOWDER BY MEANS OF TRANSMISSION ELECTRON MICROSCOPY AND IMAGE PROCESSING ANALYSIS

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Interface phenomena, in general, and grain boundary diffusion, in particular, are of paramount importance in nano-scale polycrystalline materials as a whole. Aiming at studying transport properties in consolidated nanostructured oxides, we have, initially characterized TiO2 nanoparticles by means of diffraction contrast transmission electron microscopy, based on both bright field (BF) and centered dark field (CDF) micrographs, as well as High Resolution Electron Microscopy (HREM) images. The BF images documented at 600000 magnifications showed agglomerations of nanoparticles. These micrographs were used to analyze the particles size repartition with an average size of 23.2nm. The HREM pictures showed that the powder consists in almost defect free single crystalline particles. A Radial Distribution Function (RDF) method is proposed to compute atomic lattice characteristics from HREM images, it calculates the histogram of the distances between all the atoms in a limited neighborhood. The Lattice Parameter Analyzer (LPA), an image processing program in Matlab, was developed using this technique. From a good quality HREM image, the LPA gives the value of the first neighbors' distances and presents the results as a nanometer scaled plot showing peaks corresponding to those distances. The LPA is a powerful tool since it gives statistical results and may be used for any crystalline material HREM image.

D P26 STUDY OF THE INFLUENCE OF TEMPERATURE ON THE STRUCTURE OF A SILICA-DISPERSED COBALT FERRITE NANOCOMPOSITE BY MICROANALYSIS

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Composites of cobalt ferrite particles dispersed in a silica matrix ($CoFe_2O_4/SiO_2$) were prepared by the sol-gel process. Wet $CoFe_2O_4/SiO_2$ samples were prepared
in monolithic shape, dried at 110°C, and treated at various temperatures between 110 and 1100°C. Ferrite phase was identified by X-ray diffraction. The samples were morphologically analyzed by scanning electron microscopy and chemically mapped by energy dispersive spectroscopy. The backscattered electron image of the composite treated between 300 at 7000C presented defined white regions distributed throughout the sample, whose EDS analyses detected mostly the presence of cobalt clusters.
In the black region, Si, Fe, O and traces of Co were detected. As the calcination temperature increased, the clusters disappeared, and the cobalt diffused into the composite, binding to iron to form the ferrite. At temperatures above 900°C, EDS analyses detected a homogeneous distribution of Co and Fe in the composite.

D P27 THE ROLE OF VANADIUM OXIDE ON THE PROPERTIES OF THE BARIUM ZIRCONATE TITANATE CERAMICS

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The structural, morphological and electrical properties of pure and vanadium doped ($BaTi_{1-x}Zr_x)O_3$ ceramics with $x = 0.05$ were evaluated by using the mixed oxide method. By XRD a single perovskite phase was verified while by SEM the addition of vanadium leaded to a decrease in the mean grain size. It can be noted that the addition of vanadium decrease the maxima sinterization temperature due to the formation of a liquid phase. The study of dielectric properties suggests that the addition of vanadium decrease the dielectric constant due to the reduction of the grain size. The remnant polarization behavior are also studied for different samples and the saturated hysteresis loops were observed for pure BZT ceramics. On the other hand, the addition of vanadium causes a strong decrease in the grain size affecting the domain walls and therefore the switching characteristcs of the ceramics.

D P28 STUDY OF NEW AND USED AUTOMOTIVE CATALYSTS BY SCANNING ELECTRON MICROSOPY AND ENERGY DISPERSIVE SPECTROSCOPY

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In this work, we discuss the deactivation of automotive catalyst by inorganic contaminants originating in engine oil and fuel, and by high temperatures. These catalytic converters are formed by noble metals such as platinum, rhodium, palladium, and molybdenum supported in ceramic materials. These metals convert the pollutant gases in others no-pollutant like carbon dioxide, water and nitrogenous. Scanning electron microscopy images and energy dispersive spectroscopy analysis were used to characterize the variation of contaminant relative concentration and the changes in the catalyst morphology with the operation temperature. Other complementary techniques such as X-ray diffractometry and gas adsorption were also used to analyze the structural and textural changes in the material.

D P29 α-UPS AND VS SILANES AS ADHESION PROMOTERS AND CORROSION INHIBITOR COATINGS ON HOT DIP GALVANIZED SURFACES (HDG) AND ON ZN/FE, ZN/CO ALLOYS ELECTRODEPOSITIONS

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Zinc´s excellent resistance in corrosive environments and great capability to protect steel has made hot dip galvanized steel (HDG) and Zn electrodepositions (ED) usual materials in industry. However, they must be protected from corrosive environments using organic coatings (paint). Zinc/paint system´s weak adhesion makes the Zinc substrate surface to receive a relatively effective pretreatment. Nevertheless, it is toxic to human being and environment. Organofunctional silanes, free from chromium (Cr+6), are an alternative to substrate pretreatment, because they are bifunctional molecules, which can act as adhesion promoters or reticulation agents. The adhesion promotion between organic and inorganic materials is their main advantage. Thanks to this they can be used in paints for metallic substrates. In this work Silane film depositions on HDG and Zn ED were made. Electrochemical Impedance Spectroscopy (EIS) was used. When polarization resistance of several silane-paint systems, calculated through EIS using an equivalent circuit, is observed as a function of time in a saline solution, we note that some treatments are more stable than others. In this sense, EIS is useful for silane selection. In this context, tests were made with important variables: silane solution pH and concentration and type of substrate. Partial results were obtained at 2% concentration. On the basis of these results the ideal conditions were determined. SEM and EDS were used to ascertain quality films.

D P30 IMAGING AND CHARACTERIZATION OF NANOTUBE MICROBUNDLES

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A number of different carbon nanostructures including carbon nanotubes CNTs is observed in the products collected from different parts in an arc-discharge chamber. There are many facts that influence the yield and morphology of the CNTs obtained during the growth process such as: stability of the electric arc, atmosphere, pressures, gas composition, state, structure and distance between electrodes, etc. Besides,characterizing the physical properties of individual nanostructures such as carbon nanotubes (CNTs) ends up in a challenging situation due to the difficulty in manipulating these nanometer size objects. This job will use TEM, STM and AFM as techniques to determine purity and homogeneity of bundles of carbon CNTs produced with the arc discharge method through image comparison. TEM imaging will be carried out under diffraction contrast and phase contrast modes . The former in order to reveal the overall size and shape distribution together with elastic constraints.The later aiming at reaching atomic scale resolution. Electron diffraction studies are also to be explored.

D P31 MORPHOLOGICAL AND THERMAL CHARACTERIZATIONS OF POLYCRYSTALLINE CO7-XNIXSB2O12 SPINELS

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Polycrystalline Co7-xNixSb2O12 (0 ≤ x ≤ 4) powders were obtained by polymeric precursor method and heat-treated at temperatures ranging from 600 to 1100 oC. They were characterized by X-ray diffraction (XRD), thermal analysis (TG/DSC), specific surface area (BET) and scanning electron microscopy (SEM). Analysis of the XRD data indicate that most of the samples are single phase, only the phase with x = 4 presents two additional phases (NiO and NiSb2O6). TG/DSC measurements revealed that until 1200 oC all compounds showed thermal stability and that nickel occupation seems decrease the temperature of spinel phase formation. Surface area data for all phases revealed a linear reduction in the specific area (SBET) as the heat treatment temperature were increased. Such analysis showed that when sintered at 900 oC, the powders of all phases are nanometric. The nickel addition in the structure seems to promote an enlargement of the surface area, which results in a lower diameter of the grains with the increase of the Ni content even for the samples heat-treated at higher temperatures. This result suggested that the introduction of Ni in the Co7-xNixSb2O12 structure seems to change the diffusion process into the crystalline lattice and have promoted an inhibition of the particles growth. But it could be also related to an agglomeration of the grains in such compounds, as observed by means of SEM images.

D P32 ANELASTIC SPECTROSCOPY IN A0,7CA0,3MNO3 (A = LA, ND, PR, Y) CMR COMPOUNDS

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During the last decade, several works studied the oxide manganites with perovskite structure due to their rich magneto-structural phase diagram and the occurrence of colossal magnetoresistence properties (CMR). Some of these studies used anelastic spectroscopy techniques to precise determine the frequency jumps of an atomic species, such as vacancies or interstitial atomic specimens. If different types of atomic jumps are possible, they can be select discriminated. In this work, anelastic spectroscopy measurements in CMR samples are presented using an inverted torsion pendulum, operating with oscillation frequency between 1 and 40 Hz, temperature range of 88 and 700 K, heating rate of 1 K/min and vacuum better than 10−5 mBar. The samples prepared by Sol-Gel routes were previously characterized by x-ray diffraction and magnetic susceptibility. Anelastic relaxation results we observed a relaxation process in the high temperature region attributed to the ordering of the interstitial oxygen atoms in the crystalline lattice. (work supported by: FAPESP).

D P33 INFLUENCE OF CONCENTRATION, THICKNESS AND GROWING RATE IN THE OPTICAL QUALITY OF CuBr DOPED KBr FILMS

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The production of materials of CuBr doped KBr films by the resistive evaporation method, makes feasible to obtain high Cu+ ions concentrations(1021 atoms/cm3), opening the possibility of applying these films as UV optical filters, with the maximum in 278nm.The usual concentration found in single doped crystals is very limited and there are always Cu+ ions in agglomerate states and the formation of crystalline CuBr.The main objective of the present work is to report the effect of increasing Cu+ concentration on the optical properties of KBr film, trying to optimize its transparence in the visible and infrared regions and trying to get a high absorption coefficient in the UV region. The films were prepared by co-evaporation of KBr and CuBr powders on SiO2 substrates. The Cu+ concentration, as determined by EDX, for 1 - 10%CuBr. Structural and optical properties were investigated through SEM, X-ray diffraction and transmittance. The films are polycrystalline, and the grain size decreases with increasing Cu+ impurity concentration, yielding an increase of visible transmittance.These films show a 6.601Å lattice parameter with a fcc structure.When the Cu+ concentration is increased, the UV band position remains the same and no clusters are detected even with the high 10%CuBr concentration investigated, which differs significantly from single crystals samples. We observed that for smaller thickness and larger growing rate, the films present better optical characteristics