3RD BRAZIL MRS MEETING

October 10-13, 2004

SYMPOSIUM B:

NUCLEATION AND GROWTH PROCESSES IN SOLIDS: FUNDAMENTALS AND APPLICATIONS

Symposium Organizers:

Aldo Craievich (IF-USP)

Guillermo Solórzano (DCEM - PUC-Rio)

SYMPOSIUM B

NUCLEATION AND GROWTH PROCESSES IN SOLIDS: FUNDAMENTALS AND APPLICATIONS

Tuesday, October 12

Session Chair: Guillermo Solórzano (PUC- Rio)

08:00 Invited KINETICS AND MORPHOLOGIES RESULTING FROM NUCLEATION AND GROWTH TRANSFORMATIONS IN TEMPORALLY AND SPATIALLY INHOMOGENEOUS FINITE SPECIMENS
J. W. Cahn. NIST. Gaithersburg, MD 20899-8555, USA.

08:40 Invited NUCLEATION AND GROWTH DURING RECRYSTALLIZATION
A.F. Padilha. EPUSP. Departamento de Engenharia Metalúrgica e de Materiais. Av. Prof. Mello Moraes, 2463. 05508-900, São Paulo, Brasil. F. Siciliano Jr. CBMM. P. R. Rios. UFF.

09:20 Invited TRANS-INTERFACE DIFFUSION-CONTROLLED COARSENING
A. J. Ardell, V. Ozolins. Department of Materials Science and Engineering. University of California at Los Angeles.Los Angeles, CA 90095-1595, USA

10:10 COFFEE-BREAK

Session Chair: Fernando Lázaro Freire Jr. (Depto. de Física, PUC-Rio)

10:30 Invited CHEMICAL MIXING AND GROWTH BEHAVIOR OF Ag AND Au NANOCRYSTAL MIXTURES
J. M. Howe. Department of Materials Science & Engineering, University of Virginia, Charlottesville, VA 22904-4745, USA T. M. Murray. Department of Materials Science & Engineering, University of Washington, Seattle, WA 98195, USA.

11:10 Invited TRIPLE JUNCTION CONTRIBUTIONS TO THE PHENOMENON OF GRAIN GROWTH?
Alex King. School of Materials Engineering. Purdue University. 501 Northwestern Avenue West Lafayette IN 47907-2044. USA.

11:50 Invited HOMOGENEOUS NUCLEATION IN DEEP UNDERCOOLED METALLIC, POLYMER AND INORGANIC LIQUIDS - A 50 YEAR PERSPECTIVE
E. D. Zanotto. Vitreous Materials Laboratory, LaMaV. Department of Materials Engineering, DEMa. Federal University of São Carlos, 13565-905, São Carlos-SP.

12:25 LUNCH

Wednesday, October 13

Session Chair: Aldo Craievich (Instituto de Física, USP)

08:00 Invited INFLUENCE OF STRAIN, SURFACE DIFFUSION AND OSTWALD RIPENING ON THE EVOLUTION OF NANOSTRUCTURES ON Si SURFACES
R. J. Nemanich, W.l Yang, L. Fitting, M. C. Zeman. Department of Physics, North Carolina State University, Raleigh NC 27695-8202. USA.

08:40 Invited NUCLEATION AND GROWTH AND SPINODAL DEWETTING PROCESSES IN THIN POLYMER FILMS
P. F. Green, Texas Materials Institute and Department of Chemical Engineering. The University of Texas at Austin, Austin TX 78712. USA

09:20 Invited A METALLIC GLASS THAT IS NOT A FROZEN MELT: DISCOVERY AND IMPLICATIONS OF METALLIC GLASS FORMATION BY A NUCLEATION AND ROWTH MECHANISM.
J. W. Cahn, L. A. Bendersky. NIST. Gaithersburg, MD 20899-8555, USA.

10:10 COFFEE-BREAK

Session Chair: Angelo F. Padilha (Escola Politécnica, USP)

10:30 ACTIVATION ENERGIES FOR NUCLEATION, GROWTH AND DIFFUSION IN THE Cu-2%Al ALLOY WITH Ag ADDITIONS
A. T. Adorno, R. A. G. Silva. UNESP - Instituto de Química. Caixa Postal 355, 14801-970 Araraquara-SP.

10:50 NUMERICAL SIMULATION OF DENDRITIC GROWTH DURING THE PROCESS OF SOLIDIFICATION IN UNDERCOOLED Fe-C LIQUID SYSTEM
A. F. Ferreira, J. A. Castro, A. J. Silva. UFF. Rua: N, 164, Jardim Amalia I, CEP 27250-840, Volta Redonda, Rio de Janeiro.

11:10 CRYSTALLIZATION OF A NIOBIUM PHOSPHATE GLASS
L. Ghussn. CAB/IPEN. División Materiales Nucleares, Av. Bustilo, km 9.5, Bariloche, Río Negro, Argentina, CP 8400. M. O. Prado, D. O. Russo. CAB. J. R. Martinelli. IPEN.

11:30 NUMERICAL SIMULATION OF THE PURE IRON SOLIDIFICATION VARYING THE PARAMETERS OF THE PROCESS
A. C. Mossi, M. M. Pariona. UEPG. Campus Uvaranas, Bloco CIPP, Laboratório LIMAC, Ponta Grossa, Pr.

11:50 MAGNETO-VOLUME EFFECT IN ULTRATHIN FexNi1-x FILMS ON CU90Au10(100)
W.A.A. Macedo, P. L. Gastelois, M. D. Martins, L. C. M. Belo, R. Landers. CDTNLaboratório de Física Aplicada, Centro de Desenvolvimento da Tecnologia Nuclear, CP 941, 30123-970 Belo Horizonte.

12:25 LUNCH

Session Chair: Waldemar A. Macedo (CDTN, Belo Horizonte)

14:00 STRAIN PROFILE AND CHEMICAL COMPOSITION OF SiGe:Si(001) DOMES
M. S. Leite, G. Medeiros-Ribeiro. LNLS. PO Box 6192, Campinas, SP 13084-971. A. Malachias. UFMG. R. Magalhães-Paniago. UFMG/LNLS. T. I. Kamins, R. S. Williams. HP Labs.

14:20 THE EFFECTS OF H+ AND He+ IMPLANTATION AND ANNEALING PARAMETERS ON BLISTERING AND ION-CUT PROCESS IN Si.
A. A. D. de Mattos. ULBRA , PGMICRO-UFRGS. Caixa Postal 15051, 91501-970 Porto Alegre RS, S. Reboh. PGCIMAT-UFRGS. T. L. Marcondes. PGMICRO-UFRGS. P. F. P. Fichtner. DEMET-UFRGS.

14:40 XANES CHARACTERIZATION IN PECVD-SiOxNy FILMS
D. Criado, M.I.Alayo, I. Pereyra. POLI-USP. Av. Professor Luciano Gualberto, 158 trav. S São Paulo. M.C.A. Fantini. IF-USP.

15:00 INFLUENCE OF THE NANO-CRYSTALS IN THE ION DYNAMICS OF Pb–Cd FLUORGERMANATE GLASS-CERAMICS INVESTIGATED BY CONDUCTIVITY AND NMR
J.P. Donoso, C.C. Tambelli, C.J. Magon. IFSC-USP. C.P. 369, CEP 13560-970 São Carlos, SP. S.J.L. Ribeiro, Y Messaddeq. UNESP-Araraquara. L.A. Bueno. UNOESC.

15:55 COFFEE-BREAK

16:00 POSTER SESSION

INVITED PRESENTATIONS

B-I001 KINETICS AND MORPHOLOGIES RESULTING FROM NUCLEATION AND GROWTH TRANSFORMATIONS IN TEMPORALLY AND SPATIALLY INHOMOGENEOUS FINITE SPECIMENS

J. W. Cahn. NIST. Gaithersburg, MD 20899-8555, USA.

Under a set of very restrictive assumptions, the well-known Kolmogorov-Johnson-Mehl-Avrami (JMAK) theory for nucleation and growth reactions gives exact statistical solutions for the time dependence of the expected fraction transformed X, the interfacial areas S, and other stereological quantities. Many f these restrictive assumptions, that the specimen is infinite, that nucleation is random in the untransformed volume and that growth after nucleation is constant and radial ntil there is hard impingement, are introduced to facilitate the use of statistics. The \"Avrami\" equation, X(t) = X( )(1-exp(btn)] is widely used to fit rate data even where the JMAK assumptions are not met. The fitting parameters, b and n, are often used to interpret mechanisms. Better theories exist for specific conditions, among them the \"time-cone\" theory in which some of the JMAK assumptions can be relaxed and exact solutions can still be obtained, and various theories of diffusional transformations. The predictions of these theories can show for how the Avrami equation fails quantitatively, and lets us assess the risk of misinterpretation of mechanisms.

B-I002 NUCLEATION AND GROWTH DURING RECRYSTALLIZATION

A. F. Padilha. EPUSP. Departamento de Engenharia Metalúrgica e de Materiais. Av. Prof. Mello Moraes, 2463. 05508-900, São Paulo, Brasil. F. Siciliano Jr. CBMM. P. R. Rios. UFF.

Recrystallization is an irreversible, thermally activated heterogeneous transformation that takes place by nucleation and growth. It has some specific characteristics which are described as follows: a) its driving force or thermodynamic potential, the stored energy of deformation, is normally low as compared to those of phase transformations. It is also independent of the temperature; b) classical nucleation theory is normally considered unsatisfactory to explain the nucleation of recrystallization; c) nucleation is therefore expected to take place from preexisting nuclei; d) there are evidences of at least three different nucleation mechanisms; e) nucleation is highly heterogeneous and predominantly takes place at the heterogeneities of deformation; f) a relatively small number of nuclei is formed as compared to most of the phase transformations; g) after the initial nucleation stage only growth takes place, that is, site-saturation is often observed in recrystallization; h) the growth velocity normally decreases with time due to the concurrent recovery processes which decrease the driving force and deformation gradients; i) recrystallized grain sizes are quite sensitive to the amount of previous deformation and not much sensitive to the annealing temperature. In this work, a short historical survey of recrystallization is presented, followed by the main nucleation mechanisms and finally the growth of the recrystallized regions is discussed.

B-I003 TRANS-INTERFACE DIFFUSION-CONTROLLED COARSENING

A. J. Ardell, V. Ozolins.Department of Materials Science and Engineering. University of California at Los Angeles.Los Angeles, CA 90095-1595, USA

We present a model of coarsening controlled by diffusion through a coherent interface between an ordered precipitate and a disordered matrix. Since chemical diffusion in ordered phases is far slower than in disordered phases, the interface is a diffusion bottleneck. The new theory predicts that when the condition ?$D_m >> rD_I$ is fulfilled (? is the width of the interface, r is the radius of the precipitate and D_m and D_I are chemical diffusion coefficients in the matrix and interface, respectively: 1. The average radius squared increases linearly with time; 2. Solute is depleted linearly as the inverse square-root of time; 3. A broad scaled particle size distribution (PSD) is found; 4. The coarsening rates are completely independent of volume fraction. Published data on the coarsening of ?' (Ni_3Al) precipitates agree quite well with the new theory, which moreover explains the independence of the rate constant for coarsening on volume fraction, f, at large values of f in several Ni-base alloys. Additionally, the theory also predicts that coarsening of disordered ? (Ni-Al solid solution) precipitates in a ?' matrix (the inverse alloy), should obey traditional LSW kinetics and be strongly dependent on f, as observed experimentally, since chemical diffusion in the ordered matrix is slower than diffusion through the interface and the interface is therefore no longer a diffusion bottleneck. No other theory is consistent with the totality of these experimental results.

B-I004 CHEMICAL MIXING AND GROWTH BEHAVIOR OF Ag AND Au NANOCRYSTAL MIXTURES

J. M. Howe. Department of Materials Science & Engineering, University of Virginia, Charlottesville, VA 22904-4745, USA T. M. Murray. Department of Materials Science & Engineering, University of Washington, Seattle, WA 98195, USA.

The evolution of mixtures of supported nanoparticles during heating is not well understood either theoretically or experimentally. This is a fundamental problem relevant to important technological systems such as catalysts. In this investigation, mixtures of elemental Ag and Au nanocrystals on an amorphous C support containing various proportions of Ag and Au with particle sizes in the range of ~2-12 nm were heated for times of 5-45 min. at 400°C. The particle size distributions and compositions were followed using quantitative image and spectroscopic analyses in the TEM. Results from these studies show that the rate of particle growth is slower for Ag-Au nanocrystal mixtures compared to pure Ag or Au, particularly when small Au particles are present. In addition, Au particles act as a template for the final particle size distributions in Ag-Au mixtures, due to the strong tendency for the system to alloy and the low diffusion rate of Au compared to Ag. Compositional analyses of the Ag and Au nanocrystals versus time shows that chemical mixing plays a major role in determining the growth behavior of the nanocrystals and that the rate of composition change is proportional to the deviation of the nanoparticles from the average mixture composition. These and other factors involved in the behavior of supported nanoparticle mixtures are discussed. This research was supported by NSF and DOE.

B-I005 TRIPLE JUNCTION CONTRIBUTIONS TO THE PHENOMENON OF GRAIN GROWTH?

Alex King. School of Materials Engineering. Purdue University. 501 Northwestern Avenue
West Lafayette IN 47907-2044.

Interface-driven phenomena have been an important theme in the study of materials since the middle of the 20th Century, and the phenomenon of grain growth is a particularly well-studied example. In most grain-growth theories, triple junctions are assumed to have negligible impact, and the growth rate depends directly upon the grain boundary curvature. In this paper, we consider the effects that triple junctions may make in driving the phenomenon of grain growth. We discuss the requirements for reliable measurements of triple junction energy and present an experimental estimate of its magnitude in gold. We consider the characteristics of grain growth under conditions where triple junction energy is significant but grain boundary energy is negligible, and show that the behavior differs in many respects from the grain growth model of Von Neumann and Mullins, leading to unusual behaviors when the contributions from the triple junctions and the grain boundaries are comparable. This work is supported by the US Department of Energy, under Contract # DE-FG02-01ER45940.

B-I006 HOMOGENEOUS NUCLEATION IN DEEP UNDERCOOLED METALLIC, POLYMER AND INORGANIC LIQUIDS - A 50 YEAR PERSPECTIVE

E. D. Zanotto .Vitreous Materials Laboratory, LaMaV.Department of Materials Engineering, DEMa. Federal University of São Carlos, 13565-905, São Carlos-SP, Brasil

Even in the absence of catalyzers, which are usually employed in the manufacture of commercial glass-ceramics, a few glass-forming systems display spontaneous internal (homogeneous) nucleation in addition to surface (heterogeneous) nucleation. Hence these glasses are, in principle, good model materials to test nucleation, growth and overall crystallization theories. However, numerous tests of the Classical Nucleation Theory (CNT), which have been carried out in the last 50 years with deep undercooled metallic, polymer and inorganic liquids and glasses, demonstrate that the theory describes the temperature dependence, but fails to quantitatively predict the magnitudes of crystal nucleation rates. Possible explanations for this failure, such as the possible breakdown of the Stokes-Einstein equation (that is used to access the effective diffusion coefficients), the early precipitation of metastable phases, a possible size and temperature dependence of the nucleus-liquid surface energy, and the precipitation of mutant nuclei are tested and discussed. We conclude that despite all the above-mentioned problems, some intriguing empirical trends regarding nucleation in silicate glasses reveal a limited but important range of applicability of CNT.

B-I007 INFLUENCE OF STRAIN, SURFACE DIFFUSION AND OSTWALD RIPENING ON THE EVOLUTION OF NANOSTRUCTURES ON Si SURFACES

R. J. Nemanich, W.l Yang, L. Fitting, M. C. Zeman. Department of Physics, North Carolina State University, Raleigh NC 27695-8202

With a goal of self organization of nanostructures on surfaces, it is necessary to understand the sometimes competing phenomena related to strain, surface diffusion, and ripening. In this study we present results of silicide and SiGe nanostructures on silicon surfaces, where the nanostructure evolution is imaged in real time using photo electron emission microscopy (PEEM). Two specific examples are presented and related to strain, surface diffusion and ripening. In the first case the evolution of TiSi2 islands is shown to exhibit a new phenomena of attractive migration and coalescence. This phenomena is shown to effect a high fraction of evolved islands. The second phenomena is the shape transition of circular or compact islands into elongated or wire shaped islands. This process is observed for a range of silicide and SiGe structures, and results indicate that island width and orientation are related to interface strain. In developing this understanding of the competition of the interactions, we suggest approaches that may be employed to obviate the ripening process and consequently lead to self organized arrays of dots or wires.

B-I008 NUCLEATION AND GROWTH AND SPINODAL DEWETTING PROCESSES IN THIN POLYMER FILMS

P. F. Green. Texas Materials Institute and Department of Chemical Engineering. The University of Texas at Austin, Austin TX 78712

A thin liquid film in the nanometer thickness range may exist in an stable, unstable or metastable state on an underlying substrate. Under certain conditions, an instability may develop in the film wherein fluctuations of the local film thickness develop into certain patterns. These patterns may be spinodal patterns or a distribution of holes throughout the film and, typically, the patterns eventually evolve to form droplets. In principle, unstable films dewet via a spinodal process whereas metastable films detwet via a nucleation and growth. The particular dewetting mechanism is determined by the nature of the effective interface potential. This phenomenon is ubiquitous in thin films and occurs in various materials, from polymeric to metallic. I will discuss this phenomenon as it occurs in homopolymeric films as well as in other polymer systems that exhibit a tendency to self organize on a substrate. Analogies to the nucleation and growth and spinodal processes in liquid-liquid mixtures undergoing phase separation will also be discussed.

B-I009 A METALLIC GLASS THAT IS NOT A FROZEN MELT: DISCOVERY AND IMPLICATIONS OF METALLIC GLASS FORMATION BY A NUCLEATION AND ROWTH MECHANISM.

J. W. Cahn, L. A. Bendersky. NIST. Gaithersburg, MD 20899-8555, USA.

There are two quite different views of metallic glasses: both have disparate realizations. One, which we dub u-glass, is well established and holds that u-glasses form when undercooled melts become kinetically frozen. Such u-glasses incorporate all that is dissolved in the melts. This mechanism is quite universal and in principle occurs for all melts if they can be cooled rapidly enough. This view has been very productive for finding condition for this kind of glass formation. Multicomponent u-glasses are always unstable with respect to crystallization (devitrification). The other view holds that glasses are efficient high-density low-energy packings of atoms in an aperiodic isotropic structure. In this view such a glass competes with crystal structures, especially with structures with similar atomic motifs. Like crystals these q-glasses are expected to be less tolerant of concentration variations and are expected to be more stable than u-glasses of the same composition. Glass formation from the melt as a primary phase by a nucleation and growth reaction before there is any crystallization was found to occur in Al-Fe-Si. The glass has a quite narrow range of concentration near 15 % Fe, 20 % Si. The growing q-glass rejects some components of the melt from which it grows. This and DSC data suggest that the q-glass is chemically ordered with local atomic environments similar to the motifs of these competing crystals.

ORAL PRESENTATIONS

B-O001 ACTIVATION ENERGIES FOR NUCLEATION, GROWTH AND DIFFUSION IN THE Cu-2%Al ALLOY WITH AG ADDITIONS

A. T. Adorno, R. A. G. Silva. UNESP - Instituto de Química. Caixa Postal 355, 14801-970 Araraquara-SP, Brasil

Transformation in heterogeneous systems involves the formation of new domains of product referred to as nucleation and the advancement of the phase boundaries termed growth. The rate of such a transformation depends upon the rate of nucleation and of growth of individual domains and the effect of mutual interference of neighboring domains either through direct impingement or by long range competition for solute atoms. In this work, the study of the kinetics of Ag-rich precipitates formation in the Cu-2wt.%Al alloy with additions of 2, 4, 6, 8, 10 and 12 wt.%Ag showed that, in these alloys, nucleation and growth may be considered as distinct and consecutive processes. The linear logarithmic plot of silver concentration against the incubation period indicated a first-order reaction, with the slope corresponding to a constant rate associated to the nucleation process. The results showed that measurements of hardness changes with temperature and time associated with the transformed fraction allows the evaluation of the activation energies for nucleation, growth and diffusion of the Ag-rich precipitates formation process. The obtained values compare well with those existing in the literature and the average of the growth activation energies, summed with that obtained for nucleation, gives a value very close to that corresponding to the activation energy for Ag diffusion to grain boundaries, thus confirming that the process is diffusion controlled.

B-O002

NUMERICAL SIMULATION OF DENDRITIC GROWTH DURING THE PROCESS OF SOLIDIFICATION IN UNDERCOOLED Fe-C LIQUID SYSTEM

A. F. Ferreira, J. A. Castro, A. J. Silva. UFF. Rua: N, numero: 164, Bairro: Jardim Amalia I, Loteamento: Morada da Colina, CEP 27250-840, cidade: Volta Redonda, Estado: Rio de Janeiro.

Understanding dendritic solidification is of great interest because of the microstructural scales of segregation pattern determines the proprieties of the materials. In the last decades, significant understandings of dendritic structures were obtained. However most of the development has been obtained based on experiments and idealized theoretical models. The phase field model overcome these limitation and has shown to be an efficient technique to represent the complex diffuse interface pattern evolution. This method treats the interface as a finite volume where the phase changes from solid to liquid gradually. Hence all the governing equations can be written without distinguishing the interface which is an advantage in numerical calculation. In this study, the phase-field is applied to simulate microstructural evolution during dendritic growth within undercooled Fe-C alloy. Realistic structure of dendrites could be obtained for this alloy by using the phase field model. The calculated results showed various solidification features consistent with solidification experiments. The undercooling Parameter affects significantly the interface mobility and the thickness of the diffusion boundary layer in the liquid, as expected by available theoretical models. The calculated pattern of the solute showed the typical distribution of the solute in front of the dendrite tip and among the secondary and tertiary arms. The competitive growth also is observed during the solidification.

BO-003 CRYSTALLIZATION OF A NIOBIUM PHOSPHATE GLASS

L. Ghussn. CAB/IPEN. División Materiales Nucleares, Av. Bustilo, km 9.5, Bariloche, Río Negro, Argentina, CP 8400. M. O. Prado, D. O. Russo. CAB. J. R. Martinelli. IPEN.

Niobium phosphate glasses with composition 37P2O5×23K2O×40Nb2O5 are usually very stable concerning the crystallization, and are potentially suitables for nuclear waste immobilization. The crystallization behavior of these glasses was evaluated by optical microscopy after proper heat treatments, showing that surface crystallization is the main process occurring during the heat treatment. This result was confirmed by differential thermal analysis, since the location of the crystallization peak moves towards lower temperatures when the glass particle sizes are decreased. Two main crystalline phases, not yet identified by the X-ray diffraction patterned, were observed. These crystalline phases were named P1 and P2. Surface crystal growth rates were measured in the temperature range of 806 - 972°C (Tg=744°C) for both crystalline phases. Apparent crystallization enthalpies were determined through the Arrhenius plots of ln(U) vs 1/T. The enthalpies are 496 kJ/mol and 513 kJ/mol for the P1 and P2 phases, respectively. The surface density of nucleation sites (Ns) on 3mm diamond paste polished surfaces is (2.4 ± 0.7) x 108 nucleus/m2 for the P1 crystalline phase and (9.8 ± 0.8) x 109 nucleus/m2 for the P2 crystalline phase, when revealed at 838°C for 17.5 hours, and these values show a slight variation with time and temperature. At the tested temperatures, only P1 crystals appeared into the volume, and a volume density of nucleation sites Nv = 2.3 x106 nucleus/m3 was measured.

BO-004 NUMERICAL SIMULATION OF THE PURE IRON SOLIDIFICATION VARYING THE PARAMETERS OF THE PROCESS

A. C. Mossi, M. M. Pariona. UEPG. Campus Uvaranas, Bloco CIPP, Laboratório LIMAC.

In the study reported in this work, two-dimensional numerical simulations were made of pure iron solidification in industrial AI 50/60 AFS greensand and mullite molds, using the finite element technique and the ANSYS software program. For this purpose, the thermo-physical properties of iron were considered temperature-dependent, while for sand and mullite these properties were considered constant, and the convection phenomenon was also considered on the external surface of the mold. With intuitive of to study the influences of the parameters in the solidification process, such as, mold type sand and mulita, preheating temperature of the mold (ambient and heated), superheating temperature of the liquid metal and loss of heat on the mold by convection, with these parameters was accomplished the optimization through the factorial design. Owing to the iron's temperature-dependent thermo-physical properties, this type of problem is of nonlinear characteristic. This study is a comparative work of the numerical simulation, by the finite element method, of the process of solidification of pure iron in sand and in mullite molds, during 1.5 h of solidification. Results in 2D were obtained, such as the heat transfer, thermal flow, thermal gradient, cooling curves during the solidification process, factorial design of the result and heating or/and cooling in the molds during the solidification process. The result was completely different in both systems.

B-O005 MAGNETO-VOLUME EFFECT IN ULTRATHIN FexNi1-x FILMS ON CU90Au10(100)

W.A.A. Macedo, P. L. Gastelois, M. D. Martins, L. C. M. Belo, R. Landers. CDTN
Laboratório de Física Aplicada, Centro de Desenvolvimento da Tecnologia Nuclear, CP 941, 30123-970 Belo Horizonte, Brasil.

Bulk FexNi1-x alloys show the invar effect, i.e., a structural transformation from fcc to bcc and a collapse of the Fe magnetic moment at a critical concentration around 65% Fe. For ultrathin films, previous works on FexNi1-x / Cu(100) have shown that the fcc regime can be extended by the epitaxy, and that, for the 65% < Fe < 80%, it is observed a transition to smaller magnetic moment, associated to a atomic volume reduction of ~ 2 %. In this work, we explore the effect of an expansion of the lattice parameter of the fcc substrate on the structural and magnetic proprieties of epitaxial FexNi1-x monolayers. FexNi1-x ultrathin films (0.60 < x < 0.80) were epitaxialy grown on Cu90Au10(100), a substrate with lattice parameter of 3,65 Å (1.4 % > Cu). The substrate, and the Fe-Ni films were characterized by low-energy electron diffraction (LEED), Auger- and photoelectron spectroscopy. The magnetic properties of the Fe-Ni films (8 monolayers thick) were determined in-situ by linear magnetic dichroism in core level photoemission (LMDAD) using the TGM beamline at the LNLS synchrotron storage ring in Campinas. Our results indicate good epitaxy of FexNi1-x films on Cu90Au10(100), and a high magnetic moment (high asymmetry in LMDAD) at least up to ~ 75% Fe, i.e., the Fe-concentration in which it is observed a significant magnetic instability is clearly shifted to values higher than those obtained for FexNi1-x ultrathin films grown on Cu(100).Work supported by the CNPq, CNEN, LNLS

B-O006 STRAIN PROFILE AND CHEMICAL COMPOSITION OF SiGe:Si(001) DOMES

M. S. Leite, G. Medeiros-Ribeiro. LNLS. PO Box 6192, Campinas, SP 13084-971 Brasil. A. Malachias. UFMG. R. Magalhães-Paniago. UFMG/LNLS. T. I. Kamins, R. S. Williams. HP Labs.

SiGe:Si(001) has been considered a model system for strained epitaxial growth. This assumption stems from the fact that only two elements are involved. Nevertheless, there is a wealth of morphologies that can be achieved given a particular set of growth conditions. Two samples were grown by chemical vapor deposition (CVD) at 6ML/min: (A) 12 ML deposited at 600 C for a total deposition time of 120s and (B) 9 ML deposited at 655 C for a total deposition time of 90s, both producing dome-shaped islands. They were used to explore the temperature influence in SiGe alloys growth. The surface morphology and strain profile were studied using Atomic Force Microscopy (AFM) and X Ray Diffraction (XRD). AFM results indicated a very uniform island size and shape distributions. To examine the chemical composition and stress of the domes, angular and radial XRD scans were performed at two different energies: 11.0 and 11.1 keV around the (4 0 0) reflection. The scattering factor is notably different near the Ge K-absorption edge (11.1keV) and 11.0 keV, causing intensity variations that are related to the Ge concentration in the islands. The effect of temperature on the 3D composition profile was investigated with AFM on as-grown and selectively etched samples and compared to the XRD derived profiles. The authors acknowledge the financial support from FAPESP (03/09374-9), CNPq and HPBrazil.

B-O007 THE EFFECTS OF H+ AND He+ IMPLANTATION AND ANNEALING PARAMETERS ON BLISTERING AND ION-CUT PROCESS IN Si.

A. A. D. de Mattos. ULBRA , PGMICRO-UFRGS. Caixa Postal 15051, 91501-970 Porto Alegre RS, Brasil. S. Reboh. PGCIMAT-UFRGS. T. L. Marcondes. PGMICRO-UFRGS. P. F. P. Fichtner. DEMET-UFRGS.

The combination of He and H implants is studied as a function of implantation and annealing parameters in order to improve the ion-cut and Si layer transfer technology for microelectronic photovoltaic substrates. In the present work we study the formation of blisters as a precursor of layer transfer process. The implantations were performed at room temperature and the energies for He and H were scaled to provide bubble formation at a depth of 300nm (low energy case) and 1500nm (high energy case). For each case, the implanted fluences were between 1 to 5x10^16cm^2. The samples were annealed in high vacuum, either directly at 700°C for 1800s or with a pre-annealing step of 350°C for 1800s, and characterized by Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Rutherford Backscattering spectrometry and chanelling (RBS/C). The observed microstructure evolution depends on implantation energy, fluence and annealing conditions. The results were evaluated in terms of the observed blistering fractional area (BFA). We demonstrate that the BFA values can be optimized for medium fluence and high energy implant cases. In comparison with pure H implants, our results show significant improvement of the BFA using a combination of H and He implants with lower total fluences. The results are discussed in terms of bubble nucleation and growth mechanisms.

B-O008 XANES CHARACTERIZATION IN PECVD-SiOxNy FILMS

D. Criado, M.I.Alayo, I. Pereyra. POLI-USP. Av. Professor Luciano Gualberto, 158 trav. 3. M.C.A. Fantini. IF-USP.

Combining the advantages of SiO2 and Si3N4, SiOxNy films present suppression to boron penetration and higher dielectric constant solving many dielectric-induced instabilities in microelectronic devices with thin gate dielectric. Besides this, SiOxNy films are also very attractive in optical applications due to the low absorption losses in the visible and near infrared wavelength range and because by simply varying the deposition parameters, materials with different refractive index and low stress are obtained. In this work, we report studies on the Si, N and O local bonding insilicon oxynitride films deposited by PECVD technique, with chemical compositions varying from SiO2 to Si3N4, utilizing N2O+N2+SiH4 and N2O+NH3+SiH4 precursor gaseous mixtures. These films are characterized by X-Ray absorption near edge structure at the Si, N and O-K edge, to determine the local bonding and by Rutherford Backscattering Spectroscopy to obtain their chemical composition. The Si-K edge results show a homogeneous structure compatible with a random distribution of Si-N and Si-O bonds (Random Bonding Model). On the other hand, the N-K edge analyses demonstrate two edges, which are attributed to two different nitrogen sites, a trigonal Si3N4-like site and another one corresponding to nitrogen substituting O in Si-O-Si bridges. Finally, the O-K edge shows only one edge attributed to O-Si bonds for both gaseous mixtures.

B-O009

INFLUENCE OF THE NANO-CRYSTALS IN THE ION DYNAMICS OF Pb – Cd FLUORGERMANATE GLASS-CERAMICS INVESTIGATED BY CONDUCTIVITY AND NMR

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Rare-earth ion doped transparent oxyfluoride glass-ceramics containing PbF2 nanocrystals has attracted great interest because of their optical, mechanical and chemical properties. In this work, the temperature dependence of the electrical conductivity and the F-19 nuclear magnetic resonance (NMR) of PbGeO3 – PbF2 – CdF2 glass ceramics are investigated. Crystal sizes in the range 100 – 1000 nm are observed in the glass ceramics obtained from thermal treatment of the oxyfluoride glass at high temperature (462 C). The conductivity of the glass ceramics (around 10-7 S/cm at 500 K) increase with increasing lead fluoride content and were found to be smaller than that in the corresponding glasses, suggesting that there are poor ionic conducting regions in the interface between the nanocrystals. Activation energies extracted from the conductivity data are in the range 0.6 – 0.8 eV. The temperature dependence of the F-19 relaxation times, measured in the range 100 – 800 K, exhibit the qualitative features associated with high fluorine mobility in both, glass and glass ceramics materials. Deconvolution of the spin-lattice relaxation rates observed in the glass ceramics suggests that the observed high temperature rate maximum is associated with the diffusional motions of the fluorine ions in PbF2 crystals, whenas those observed at low temperature is probably caused by motion of ions belonging to different fluoride species, such as fluogermanate and Cd1-xPbxF2 solid solutions.

POSTER PRESENTATION

B-P001 PHOTOLUMINESCENCE OF Ge NANOCLUSTERS IMPLANTED IN NANOPOROUS Al2O3 FILMS

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Nanoporous Aluminum Oxide (Al2O3) films with uniform porous size of 45 nm were prepared by the electrochemical process in inorganic acid medium. The films were then implanted at room temperature with Ge+ ions in a fluence of 1.2 x 1016 cm-2 and energy of 120 keV. In order to induce the growth of Ge nanoparticles, post implantation thermal annealing in Argon atmosphere was carried out, in the temperature range from 100 to 600 oC. The photo-luminescence spectrum as a function of annealing temperature shows that at low annealing temperatures (200 oC) the samples present a low intensity and broad emission band centered at 545,6 nm consistent with the emission band characteristic of Ge nanoclusters, with diameters in the range of 4 to 8 nm. As the annealing temperature is increased to 400oC or the annealing time is increased at the same temperature, the PL intensity increases by a factor of » 20 and the emission band does not shift in energy. The increase in intensity can be related to the increase in the number of Ge nanoclusters. At the annealing temperature of 600 oC the emission band is red shifted by about 20 nm and the emission intensity decrease significantly, again in agreement with the fact of an increasing size of the Ge nanoclusters. Theses results will be discussed in the framework of the growth dynamics of the Ge clusters over the nanostructured aluminum oxide surfaces, and explained based on a three-dimensional quantum confinement model.

B-P002 CHARACTERIZATION OF THE SIO2-CAO-MGO-P2O5-(NA2O) GLASS SYSTEM

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Glass-ceramics are so named because they are obtained from glassy systems which, when submited to a controlled cristalization process, convert into policrystalline ceramics. In the present work, are presented results referent to the study of the effect of the magnesium oxide in the glass-ceramics proprieties of the SiO2-CaO-MgO-P2O5-(Na2O) system. The characterization of the samples consisted in mineralogical and microstructural analysis, Vickers microhardness and assays of chemical resistance and in vitro, trough the immersion in SBF (Simulated Body Fluid). It was observed the crystallization of hidroxiapatita for the formulation with more concentration of magnesium oxide. The results obtained characterized the system studied and indicated the potential use of these compositions as bioceramic.

B-P003 GROWTH OF CVD DIAMOND FILM ON Ti6Al4V ALLOY WITH THE ADDITION OF CF4 IN THE PRECURSORY MIXTURE

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This work analyzed the CVD diamond film deposition on Ti6Al4V alloy with the addition of CF4 gas in the precursory mixture, looking forward to gain benefits in terms of film quality and nucleation tax. Adherence of CVD diamond film to substrate surface was evaluated by indentation method, using same tests of Rockwell A hardness, under 60 Kg loads. Morphology of the diamond film and indentation diameter were analysed and measured by Electronic Scattering Microscope. The results showed a significant augment in the nucleation tax with benefits in the film quality. The study of CVD diamond film deposition can also be extended to other titanium alloys in order to verify the influence of other chemical elements present in the mentioned alloys. Support: Fapesp, CNPq, Fundunesp, PROPP-Unesp, LDDCVDNM-Unesp.

B-P004 CVD DIAMOND FILMS GROWTH ON TITANIUM ALLOYS WITH VARIOUS METHANE CONCENTRATION

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The large specific strength is an effect which makes titanium alloys a basic structural material for aircraft and aerospace applications. Nevertheless, that alloy presents low resistance to weariness. CVD diamond coating on the surface of Ti6Al4V alloy will increase its lifetime. In this work, CVD diamond films were deposited by hot filament technique with various methane and hydrogen concentrations, in order to study the adherence between film and substrate. Film quality and stress were evaluated by Raman Scattering Spectroscopy (RSS). Total residual stress (extrinsic and intrinsic) in diamond films were calculated from the shift of the stressed CVD diamond Raman line relative to the basic line at 1332.5 cm-1. Thermal stress (extrinsic) was evaluated from the numerical integration of thermal expansion coefficients of the film and the alloy, starting from environment temperature until film growth temperature. Intrinsic stress was calculated considering the difference between the total and the thermal stresses. Film morphology was analysed by Scanning Electron Microscopy (SEM). Adherence tests were done with indentation technique. Support: Fapesp, CNPq, Fundunesp, PROPP-Unesp

B-P005 CVD DIAMOND FILM DEPOSITION ON DENTAL IMPLANT WITH SMOOTH TITANIUM ALLOYS

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Chemical vapor deposition (CVD) diamond film is possible form on the surface of diverse materials, between the which they are the diamond. The diamond by be an organic material, him confers the estates of biocompatible and hemocompatible, being these of extreme importance dental implant, deeds of titanium and its alloys, enabling diminish the symptoms of rejection. With the samples of dental implants, they went deeds cleaning from the surface with oxalic acid and afterwards with ultra-sound, weighing and determination from the morphology of the body of test by observations in scanning electronic microscope. The surface of the sample prepared had sweats composition analyzed with X-rays and EDX. CVD diamond film was deposited, utilizing a hot filament reactor. After the depositions utilized himself, for analysis in scanning electronic microscope, x-rays diffraction and Raman Spectroscopy. Initially the deposition went deed utilizing the sample with smooth titanium shown in Figure 2. The first results showed a typical adhesion for this kind of aterial. It estimates the thickness of CVD diamond films are less then 10 micra. As work after sample will be submitted implant in authorized centers.

B-P006 SYSTEM TO OBTAIN InSb INFRARED DETECTORS

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InSb is a member of the class of photo detective materials known as III-IV semiconductors. The crystal structure of pure InSb is zinc-blende cubic, similar to the compound InAs and the alloy system Hg1-xCdxTe. The p-type material can be formed if more indium is used in the compound and a n-type material can be formed if more antimony is used. However this technique is not stable and easy to be controlled, consequently a best best choice is to introduce specific impurities in order to obtain the p-n junction. In this work the InSb n-type wafers were obtain by introduction of Te, then InSb with Cd was diffused in order to obtain p-type material and an abrupt junction of about 1 µm. The diffusion was performed in a modified Liquid Phase Epitaxy (LPE) oven, which has a quartz tube opened instead of a quartz ampoule sealed . The advantages of this method are: the diffusion can be performed in bigger substrate areas improving the device production; this method decrease the device manipulation, decreasing human mistakes, increasing the process reproducibility. So far, our best result is a IR detector with 8,9 x 10 7 cmHz 1/2 W -1, measured at 4,5 µ.

B-P007 TRIBOLOGICAL BEHAVIOR OF HARD COATINGS IN LUBRICATING CONTACT USING BIODIESEL

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Strict environmental norms and the searches for new source of energy have led to development of alternative fuels to the traditional oil fuels. In this way, biodiesel appears as a promising option. It is used in diesel engines in pure form or in mixtures with diesel from oil. Although literature shows positive results by the adoption of this fuel, not much is known about the effects of biodiesel originated from soybean in the reply of materials used in fuel injection pumps. On the other hand, the pressure level that material is submitted is extreme, being relevant in the life and performance of mechanical components. The use of hard coatings in machine elements has suggested satisfactory results by combining friction reduction and increase of wear resistance. The present work aims to identify the tribological behavior of materials used in fuel injection pumps. The adopted coatings, titanium nitrate (TiN) and amorphous carbon (diamond like carbon - DLC), allows to evaluate the evolution of friction for different mixtures (B5, B10, B15, B20 and B100) in comparison with diesel itself. Different parameters of deposition are used, in order to optimize the tribological behavior.

B-P008 MECHANICAL AND TRIBOLOGICAL PROPERTIES OF CARBON AND NITROGEN IMPLANTED ALUMINIUM

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Aluminum has been used for industrial applications mainly in the automotive and aerospace technologies. Even at alloyed form, they still present poor mechanical and tribological properties. Improvement on these properties is made by nitriding and carburising. The present study relates hardness and elastic modulus profiles and wears of C and N implanted Al. The ion energy was 25 keV for C and 30 keV for N. Both ion fluences were 4x1017 ions.cm-2 at RT and at 500°C. Surface characterizations included grazing incidence X-ray diffraction, elastic recoil detection analysis and Raman spectroscopy. Elastic modulus and hardness profiles were determined by nanoindentation according to the Oliver-Pharr method. Frictioncoefficient profiles were obtained by linear reciprocal ball-on-flat sliding test. X-ray results indicated the presence of embedded AlN and l4C3 precipitates. Elemental profiles showed ballistic and temperature effects on the C, N and O distribution. Raman analysis showed amorphous carbon clusters (peaks D and G). Highest hardness (10 GPa) was obtained for N + C implantation at RT. Implantations performed at high temperature reduces surface hardness to 2 GPa. Subsequent thermal treatment at 400°C/1h reduces the surface hardness in about 50% in respect to the not annealed samples. Friction coefficient results suggested that after the thermal treatment, the running-in performance was improved. The friction coefficient at these conditions is about 0.2 while Al bulk is 0.7

B-P009 KINETIC AND CRYSTALIZATION MECHANISM OF BISMUTH ZINC NIOBATE PYROCHLORE POWDERS

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Pyrochlore and pyrochlore-related compounds occurring in the Bi2O3-ZnO-Nb2O5 system exhibits high dielectric constants (e), low dielectric losses, and compositionally tunable temperature coefficients of capacitance (tc). These properties, allied to the low sintering temperatures (less than 950 oC), make these compounds attractive candidates for capacitor and high-frequency filter applications in multilayer structures co-fired with metal electrodes. Bismuth zinc niobate pyrochlore (Bi2O3-ZnO-Nb2O5) powders have been successfully prepared by a chemical method using the polymeric precursor route. The principal aim of this work is to study the crystalization kinetic of cubic BZN nanopowders, using a chemical method based on polymeric precursors. The formation mechanism of the pyrochlore phase was investigated by TG-DSC and X-ray diffraction. The powders morphology was examined by scanning electron microscopy. Pyrochlore phase is already present at 500 oC and the treatment at 700 oC leads to a well crystallized nanopowder. The kinetic parameters were determined by JMAK model, based on R.A. Ligero method for non-isothermal crystallization. The apparent activation energy is 293±10 kJ/mol, the exponent of Avrami is 1.3±0.1, and the frequency factor is 2.3x1016 s-1. The exponent of Avrami indicates that the crystallization mechanism is controlled by diffusion.

B-P010 FORMATION OF CARBON FIBER-REINFORCE CERAMIC-MATRIX COMPOSITES WITH POLYSILOXANE/SILICON DERIVED MATRIX

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Carbon-fiber-reinforced ceramic matrix composites (CMC)s areatractive materials for applications requering low specific weight andhigh strength and toughness at elevated temperatures. A ceramic matrix forcarbon-fiber-reinforced ceramic composites has been developed frompolysiloxane/silicon mixtures. On pyrolysis, the polymer-filler mixture isconverted to a ceramic matrix through reactions between the Si filler andthe gaseous and solids products from the polymer degradation and theatmosphere. Matrix sample without fibers was prepared as slurry with Sipowder dispersed into polyorganosiloxane/trietoxysilane solutions. Thisslurry was used to stack eight-harness satin weave of carbon fibersforming a laminated composite, which was pressed and cured up to 200ºC.The cured laminates were then pryrolysed in N2 atmosphere up to 1500ºC.After pyrolysis the resulting multiphase ceramic matrix consists ofcrystalline phase of silicon carbide, silicon oxinitride and a siliconoxycarbide (SiOC) glass. The carbon fibers are surroundind by thesematerials that can protect them from further oxidation acting as an oxigendiffusion barrier. The nitridation of silicon filler leads to a volumeincrease avoiding multiple reinfiltration of the structure that is oftennecessary to achieve a dense matrix.

B-P011 COMPARISON OF THE DISTRIBUTIONS OF GRAIN SIZE AND OF NUMBER OF SIDES PER GRAIN OBTAINED FROM A 2D MONTE CARLO SIMULATION AND FROM ANALYTICAL MODELING

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It is a well-known characteristic of grain growth evolution that the microstructure approaches the so called self-similar state. In the self-similar state the microstructure remains invariant with time if it is appropriately scaled. For example, the grain size distribution remains invariant when the grain size is scaled by the average grain size. This state has been the subject of analytical theories of which Hillert's theory, based on the classical work of Lifshitz and Sliozov and also by Wagner, was the first. In a recent work, Rios and Glicksman have extended Hillert's approach and were able to derive the distribution of the number of sides of the grains in addition to the grain size distribution. In the present work the distribution number of sides of the grains obtained by analytical methods is compared with the corresponding distribution obtained by Monte Carlo simulation of grain growth. The results are not encouraging, suggesting that the analytical theory is at best a first approximation. Reasons for this are discussed.

B-P012 INTERFACE ANALYSES OF TITANIUM THIN FILM DEPOSITED BY ELECTRON BEAM ON 304 STAINLESS STEEL

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The 304 SS surfaces covering with titanium thin film has been studied with objective to created an intermediate film for CVD diamond and TiN film deposition. In this work, interface analyses of Ti film-304 SS substrate system are presented and discussed. The titanium film was obtained by vapor phase deposition produced by electron beam device. The 304 SS surface, the Ti film surface and its interface were then characterized by Auger electron spectroscopy (AES), nanoindentation technique and X-ray diffraction (XRD). AES, which was employed to examine the concentration depth profile of the sample before and after the film deposition, showed a presence of oxygen on 304 SS surface and interdiffusion between Ti and 304 SS during the growth of the film. The Ti film-304 SS system mechanical behavior were measured using the continuous stiffness measurements technique. It was found that Ti film-304 SS interface had a higher hardness value than the Ti film and 304 SS substrate.The authors would like to thank FAPESP for financial support.

B-P013 STUDY OF THE CVD DIAMOND FILM DEPOSITION ON STAINLESS STEEL AIMING TO HUMAN BODY IMPLANTS

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This project intends to study, develop and optimize the process of the CVD diamond film deposition on stainless steel, aiming at human body implants. Presently, chromium and titanium alloys - cobalt – molybdenum, applied in permanent implants are somewhat very expensive (US$ 4,500); whereas a stainless steel implant is less then US$ 600. Stainless steel has been used in orthopedic implants to fix bone fractures and substitution of joints. The wide use of stainless steel for orthopedic implants is due to a favorable combination of properties such as: resistance to corrosion, good mechanical resistance, workability, good physiological tolerance and low cost. However, stainless steel is susceptible to rejection when implanted to the human body; besides, stainless steel is not biocompatible. Diamond, on the other hand, presents some peculiar characteristics such as high hardness (one of the hardest ever known), high thermal conductivity, low thermic expansion coefficient, excellent superficial finishing and, for being an organic material, confers total biocompatibility and hemocompatibility. To overcome the rejection and hemocompatibility problems in the human body, deposition of CVD diamond film on stainless steel surface solves both undesired consequences.

B-P014 INVESTIGATION OF BUBBLE FORMATION IN Ne+ IMPLANTED IN Si

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The formation of Ne induced nanocavities is studied as a function of the implantation and annealing temperatures (25°C-600°C) for a fixed annealing time and implantation parameters. As implanted and annealed samples were investigated by RBS/C, TEM and WDS in order to quantify and characterize the lattice damage and the retained Ne content. The RT implants lead to the formation of an amorphous layer extending from the surface to 250 nm. For high fluence RT implants, thermal annealings leads to the formation of {311} defects underneath a polycrystalline layer, which is partially disrupted by the formation of blisters. In contrast, for samples implanted at 250°C a high density of small bubbles is observed along the ion distribution. Clusters of interstitials defects are also present in the deeper part of the layer corresponding to the end of range of ions. After annealing, cavities and {311} defects are formed. Upon 250°C implants, the obtained system contains a high concentration of small cavities (1 - 4nm), that characterize a low thermal budget process for impurities gettering in microelectronic and photovoltaic device application. The WDS measurements shown that the Ne atom stay in the bubbles even after annealing at 1100°C. The results are discussed in comparison to the case of helium implantation and in terms of the bubble formation mechanisms and coarsening evolution of the cavities size distribution.

B-P015 ELECTRODEPOSITION OF NiFe THIN FILMS

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Given that the NiFe alloy has a high magnetic susceptibility, it has been used in many electronic devices that need a high magnetic flux. Among its basic applications is the magnetic sensor called fluxgate, which has many possible structures. An appealing construction is the planar geometry where a thin film of NiFe is the active part of the sensor, and the excitation and detection coils sit on the same plane either above or below the NiFe film. The aim of this work is the fabrication of NiFe thin films using electrodeposition, which is based on the reduction of Ni and Fe ions on a copper substrate. Ni is used as counter electrode, and both have about 1 cm2. The ions are formed from a starting diluted solution (DI water) of NiSO4 (0.7 mol/l), FeSO4 (0.03 mol/l) and NiCl2 (0.02 mol/l). During the deposition process, the current density was always kept constant. We adopted seven steps of current density with values from 4 mA/cm2 up to 28 mA/cm2. For each current density, 4 steps of total deposition time from 10 minutes up to 40 minutes were investigated. The total deposited mass was measured, and correlated to the deposition
conditions. The final structure of the films was investigated by X-ray diffraction, and the its surface and its thickness were investigated by Scanning Electron Microscopy. The magnetization curves are under investigation and the results are correlated and discussed. This work has been supported by CAPES, CNPq and FAPESP under contract number 01/08221-9.

B-P016 MILLIMETER-SIZED CRYSTALS AND THIN FILMS OF MERCURIC IODIDE GROWN BY THE SOLVENT EVAPORATION METHOD

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Mercuric Iodide (HgI2) is a promising candidate for the construction of X- and gamma- rays detectors for digital medical imaging. This material may have three 3 different structural phases, which also have different colors: the α-HgI2 is reddish and has a tetragonal structure, the orange-HgI2 has obviously an orange color and is also tetragonal (the only difference to the previous material is the position of the Hg atoms in the crystal), and the β-HgI2 is yellowish and has an orthorhombic structure. In our work, HgI2 crystals were produced by the solvent (ethanol, acetone or ether) evaporation technique. The evaporation conditions (temperature and rate) were varied in order to produce different final crystals. In the super-slow evaporation rate it was possible to obtain millimeter sized red crystals on the bottom of the dish. Increasing the evaporation rate it was possible to observe the formation of a film only on the wall of the dish, with color varying from red to yellow depending on the rate. The formation of crystals on the wall of the dish is explained by the Bènard-Maragoni convection, similar to the coffee-stain effect. X-ray diffraction, Scanning Electron Microscopy, and Raman Spectroscopy were used to characterize the crystals. This work was funded by CAPES, CNPq and FAPESP under contract 01/08221-9.

B-0017 SURFACES COATED WITH CARBON FILMS BEHAVIOUR. STUDY OF CASE IN COMPONENTS OF INTERNAL COMBUSTION ENGINES

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The increase of energy demand combined with the no renewal of oil sources demands that internal combustion engines are each time less pollutant, and at the same time economic. An alternative to carry through improvement is the application of new materials with interesting properties severe systems as engines. Great part of the internal losses of the engine is resulted of the friction between certain components. In the valvetrain, the sliding contact between the command axle cams and the valve lifters represents 15% of friction loss. It is possible to apply in the surface of lifters coatings of materials that present low friction coefficiet, thus preventing part of this loss. This project have chosen DLC (diamond-like carbon), that besides having low friction coefficients, has also high wear resistance. However, the amorphous carbon film still presents problems, as lack of adhesion to the substrate and precocious breakings. In trying to solve this problem, four different coatings are adopted , in which combinations of interlayers and incorporation of elements as Si and Cr help to prevent the presented problems. It had been coated 37 samples, where the sample is the lifter itself. These samples are submitted to tribological and physic-chemical tests, as nanohardness, relation between sp3/sp2 bonds and the amount of hydrogen contained in thefilm. From this, it is possible to evidence the influence of structure in the system tribological behaviour.

B-P018 AUSTENITE DECOMPOSITION IN A HIGH NITROGEN AUSTENITIC STAINLESS STEEL: A TEM STUDY

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Addition of nitrogen to austenitic and duplex stainless steels has been proposed within the mainframe of improving strength and corrosion behavior of these materials. This approach poses however problems for high-temperature applications, as chromium nitrides tend to precipitate, affecting negatively corrosion behavior and mechanical properties. Phase transformations in a high nitrogen (0.87 wt%) fully austenitic 25Cr–5Ni (%wt) steel were studied after solution annealing and ageing at 960ºC for 2 hours by using TEM. The heat-treatment induced the precipitation of Cr2N and ferrite formation. Evidence of competition between continuous and discontinuous precipitation (CP and DP) could be found, with a predominance of the latter regime. The preferred OR between Cr2N and austenite lamellas in DP is close to (111)gamma/(0001)Cr2N;[1-10]gamma/[1-100]Cr2N. Slight misorientations and zones of irregular morphology indicate, nevertheless, that Cr2N has some difficulty in maintaining (0001) as the rapid growth plane and habit plane. Ferrite plates formed following a Kurdjumov-Sachs type of OR. Often, Cr2N used ferrite plate fronts to nucleate and grow cooperatively with ferrite according to a Fournelle & Clark - type of mechanism. Conversely, ferrite frequently used the Cr2N/gamma interface to nucleate and partially replace lamellar austenite. Both situations show that Cr2N/alfa interfacial energy is lower than the alfa/gamma and Cr2N/gamma ones.

B-P019 DETAILED INVESTIGATION OF THE GMI IN THE FERROMAGNETIC Co70Fe5Si15B10 AMORPHOUS ALLOY

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The dependence of the giant magnetoimpedance (GMI) with the length l of the sample, the frequency f and amplitude of the ac electrical current Iac, and amplitude of a dc electrical current Idc, applied simultaneously with the ac current, are investigated in pieces of ribbons of the amorphous Co70Fe5Si15B10. The slightly positive magnetostrictive alloy was prepared by melt-spinning and the room temperature data were obtained for as quenched samples for 5 ≤ l ≤ 90 mm, 105 ≤ f ≤ 3.106 Hz, 5 ≤ Iac ≤ 60 mA, 5 ≤ Idc ≤ 60 mA and for applied magnetic field H in the range -30 ≤ H ≤ 30 Oe. The samples were characterized by x-ray diffraction and by electron diffraction microscopy. Magnetic susceptibility measurement showed that the alloy is ferromagnetic below 586 K. GMI as high as 60 % was observed for l = 15 mm, f = 1.5 106 Hz, Iac = 10 mA and Idc = 0. The maximum value of the GMI is nearly independent of the length of the sample while the position of the maximum is shifted to higher values of H when l is diminished for l ³ 15 mm. The asymmetric GMI, induced by Idc, was found to increase when l diminishes and reaches 80% for Idc = 50 mA. The magnetic permeability of Co70Fe5Si15B10 is smaller than the one found for zero magnetostriction alloys yielding an overall smaller GMI. However, this drawback is compensated by the fact that Co70Fe5Si15B10 can be cut in smaller pieces without degrading further the magnitude of the GMI which is desirable for some technological applications.

B-P020 MELTING AND FREEZING OF BI NANOCLUSTERS EMBEDDED IN BORATE GLASS

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Bi-glass nanocomposites were obtained by quenching homogeneous Bi-doped soda-borate melts followed by an annealing at a constant temperature Ta. This process induces the formation of spherical Bi nanoclusters embedded in a glass matrix [Phys. Rev. B 67, 085405 (2003)]. Since Ta Tmb, Tmb being the melting temperature of bulk Bi, we have in fact obtained liquid Bi droplets that, after cooling, become spherical nanocrystals. The nanocrystal-to-liquid transition was investigated using simultaneously the SAXS and WAXS techniques [Phys. Rev. B 65, 134204 (2002)]. The melting temperature, Tm, decreases for decreasing radius and is a linear function of (1/R), i. e. Tm=Tmb–a(1/R), a being a positive constant related to surface energy parameters. For Bi nanocrystals with R=1.5 nm, the magnitude of the melting temperature reduction is about 200K. An additional study of the liquid-to-crystal transition was performed. The freezing temperature of bulk Bi, Tcb, is much lower than Tmb (Tmb-Tcb=150K). We have established that the freezing temperature also decreases linearly for increasing values of (1/R), but at a rate lower than the melting temperature, in such a way that the magnitude of the overcooling is progressively reduced for nanodroplets with decreasing radius. The overcooling vanishes for droplets with radius close to 1 nm. The linear nature of Tc(1/R) is explained by using a simple thermodynamic model of heterogeneous crystal nucleation at the liquid-glass interfaces.

B-P021 EFFECT OF V/III RATIO ON COMPOSITIONAL MODULATION OF InGaP LAYERS GROWN ON BY CHEMICAL BEAM EPITAXY

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In this work we investigate the influence of V/III ratio employed during growth of In-rich coherent InGaP layers in the compositional modulation phenomenon. Our results show that the InGaP layer growth conditions are crucial in order to compositional modulation to be present. In agreement with other works in semiconductor alloys, the compositional modulation depends on both temperature and substrate miscut. On the other hand, our transmission electron microscopy (TEM) and atomic force microscopy (AFM) measurements point out the V/III ratio chosen for InGaP deposition also plays an important role in the compositional segregation. No contrast variations in the dark-field (220) cross-sectional TEM images of InGaP layer grown at V/III=32. However, changing only the V/III ratio to 17, a clearly periodic dark/bright contrast can be identified in the dark-field images. This contrast is related to the periodic strain field created by the compositional segregation inside the InGaP layer. The AFM analysis shows that when the V/III ratio is further decreased to 10, the surface becomes rougher due to formation of a bi-dimensional pattern of structures. The presence of such pattern points out that the compositional modulation can be coupled to the surface morphology evolution. These results suggest that the P2 flux during InGaP growth should influence the surface diffusion of In and Ga adatoms since plays an important role in both compositional modulation and surface morphology.

B-P022 CELLULAR AUTOMATA SIMULATION OF THE EFFECT OF NON-RANDOM NUCLEATION ON THE EVOLUTION OF THE GRAIN BOUNDARIES DURING RECRYSTALLIZATION

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Analytical modeling of recrystallization considers two arrangements of nuclei: a) random, b) “periodic”. If nucleation departs from these extremes, no exact analytical treatment is available. In a former paper the extent the exact analytical theory is valid if the nucleation departs from randomness in this way. In this work, this study is continued by investigating the evolution of the two kinds of grain boundaries present: grain boundaries between transformed regions and grain boundaries between transformed and untransformed regions. The possibility of using the grain boundaries to assess the extent of the non-randoness is investigated. In this work, recrystallization is simulated using cellular automata in two dimensions in order to investigate the effect of nuclei distribution. Computer simulations are carried out for nuclei distribution ranging from periodic to random. The simulation allows one to concentrate on the geometrical issues without the interference of the considerable experimental errors often observed in recrystallization studies. Although this work has recrystallization in mind its results are general, valid for any nucleation and growth transformation complying with the assumptions of the simulation.

B-P023 MICROANALYSIS OF PRECIPITATION PROCESSES IN THE ALLOY 33 (Fe-Ni-Cr-Mo-N)

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The role of interface and the relevance of interfacial energy in controlling the properties of engineering materials have long been recognized. In particular, resulting from the decomposition of a supersaturated solid solution, some alloy systems develop lamellar structures through discontinuous precipitation (DP) processes. Here, the precipitates grow by diffusion of atoms along grain boundaries that, whileacting as reaction fronts, migrate consuming the supersaturated living behind the lamellar precipitate structure. In some alloy systems a continuous precipitation (CP) can be observed inside the matrix. While the first reaction, DP, is due to interfacial diffusion at the grain boundaries, the latter, CP, is controlled by diffusion in the matrix. This combined phenomena has been found in the alloy 33, a Fe-based system, containing Cr, Ni, Mo and a high N concentration. Both diffusional transformations generate a microstructure that will incorporate an excess of interfaces in both the lamellar and the homogeneous precipitated products. The material was received with the nominal composition (given in atomic percent) of 30% Fe 33% Cr- 1,5% Mo – 0,5% N. After homogenization treatment, samples were isothermally aged at 700ºC and 900ºC. A wide range of microstructure features has been reveled. We focus our attention to the precipitation processes developed upon aging at the above-mentioned temperatures, which are typical of the alloy operational conditions.

B-P024 MECHANISMS OF LATTICE AND GRAIN BOUNDARY DIFFUSION CONTROLLING PRECIPITATION PROCESSES Al-15 at.%Ag

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The Al-Ag alloy system has been studied due to its several possibilities concerning the precipitation processes. Matrix continuous precipitation (CP) of the metastable γ’ phase always takes place in the temperature range investigated here. Concurrent with CP and dominant under certain aging conditions, discontinuous precipitation (DP) of the equilibrium γ phase is developed through grain boundary diffusion and motion. The present study attempts to focus on the characterization of the interfacial structure developed upon precipitation and growth of both γ’ and γ phases. Transmission electron microscopy, operating in diffraction contrast as well as in phase contrast modes, has been used as a primary technique. The alloy used for this investigation, Al-15at.%Ag, was cold rolled and then heat-treated for isothermal aging at several temperatures ranging from 250 to 400°C. SEM and TEM observations of as-aged and quenched microstructures show both matrix CP and grain boundary DP products. It was confirmed that γ’ precipitation rapidly takes place and grow in a plate-shape morphology with size and interplate distance proportional to the aging temperature. In order to preserve the crystallographic orientation relationship with respect to the matrix, γ’ is able to operate a branching mechanism upon growth. Observations of this area under different TEM imaging modes permit to gain information on the interface structure.

B-P025 RETROGRESSION AND REAGING OF ALLOY 8090 (Al-Li-Cu-Mg-Zr)

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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.

B-P026 CONFIGURATIONS OF EFFECTIVE SEGREGATION COEFFICIENT UNDER EFFECT OF AN ELECTRIC FIELD DURING CZOCHRASLKI CRYSTAL GROWTH - APPLICATION TO GROWTH OF LiNbO3:Cr+3

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The most usual description of the effective segregation coefficient is based on the theory of Burton, Prim and Slichter (the BPS theory), where only the normal crystal parameter dependence is considered. In some reports it has been observed that the effective segregation coefficient, and consequently the dopant/compounds incorporation is also a function of the applied electric field during the crystal growth process, or of the electric field that appears in the crystal-melt interface due to the great thermal gradient, as in the single crystal fibers growth of oxide materials. In a growth process of LiNbO3:Cr+3 an electrical field applied produce changes in the effective segregation coefficient. These results are explained by changes in the growth velocity using theoretical and experimental results. The crystals obtained are analyzed by Atomic Absorption Spectroscopy (AAS).

B-P027 EFFECT OF AN ELECTRICAL FIELD APPLIED DURING CZOCHRASLKI CRYSTAL GROWTH OF SILICON-GERMANIUM ALLOYS - PRELIMINARY RESULTS

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Electrical fields can produce changes in the effective segregation coefficient during the crystal growth process. The most usual description of the effective segregation coefficient is based on the theory of Burton, Prim and Slichter (the BPS theory), where only the normal crystal parameter dependence is considered. In some reports it has been observed that the effective segregation coefficient, and consequently the dopant/compounds incorporation is also a function of the applied electric field during the crystal growth process.This phenomenon is applied to the growth process of Silicon-Germanium alloys (Si80Ge20), and a theoretical model is used to explain the experimental results. The crystals obtained are analyzed by chemical etching, Atomic Absorption Spectroscopy (AAS), and Energy\'s Dispersive X-Ray (EDX).

B-P028 ELECTRON BEAM PHYSICAL VAPOR DEPOSITION (EB-PVD) OF Ni-Al COMPOUNDS – PRODUCTION AND MICROSTRUCTURAL INVESTIGATIONS

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Turbine blades work under corrosive atmosphere and high temperature and pressure. One way to improve life or work temperature of blades is by the use of thermal barrier coatings (TBCs) over metallic substrate applied by several techniques. The most usual configuration for the TBCs is a ceramic material, applied over an intermetallic bond coating (BC), both applied over a metallic substrate. The aim of this paper is show the results of the deposition of the BC applied by EB-PVD. The BC has two functions: improve adhesion between ceramic layer and substrate and work as barrier against oxidation of the substrate. The usual techniques for production of BC are plasma spray and diffusion techniques, these methods are limited. Here is used the EB-PVD technique, where a Ni, Cr, Al and Y alloy (MCrAlY) and a more simple alloy (Ni-Al) are evaporated. Na advantage of EB-PVD is the possibility to deposit multi-component alloy coatings with high production. However, there are drawbacks, due to the difference in the vapor pressure of the individual components, the evaporation of alloys is selective. Samples obtained in different deposition conditions were analyzed using XRD, SEM and EDS. EDS analysis how the variation in chemical composition across the cross section of the deposit layer. Despite of the compositional variations along the layer, XRD analyses had shown the presence of AlNi3 phase, which is in good agreement with Ni-Al phase diagram for the composition of the MCrAlY used.

B-P029 ADSORPTION OF H2S FROM NATURAL GAS ONTO ACTIVATED CARBON

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Processing natural gas usually aims at eliminating impurities which can affect the final quality of the product. One of the main impurities is hydrogen sulfide (H2S). In this work, activated carbon obtained from olive pits was used to adsorb H2S from natural gas. Activated carbon is a crystalline form covalently bonded carbon atoms in he form of graphite or diamond. This material can be prepared by carbonizing a wide variety of materials including wood, seeds, coconut endocarp or rind, and babassu, among others. Activated carbon samples were calcinated and later treated in 1,0 mol/g HNO3 solution and H2O2 at 110°C. The samples were then maintained under N2, rinsed to neutralize the pH and oven dried. Characterization took place by BET surface area, SEM, TPD and TPR to establish the reduction temperature. The isoelectric point was also determined. The resulting powder was then extruded into pellets. The adsorption capacity was evaluated on the basis of rupture curves. The resulting isotherms have then been fitted to traditional adsorption models (Brunauer, Emmett and Teller, Langmuir, Freundlich).

B-P030 STUDY OF SILICON OXYNITRIDE FILMS WITH LOW MECHANICAL STRESS

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Silicon oxynitride have become a promising material in optoelectronic applications due to the possibility of adjusting the refractive index over a wide range (1.46 to 2.0) by changing the films chemical composition. This flexibility permits the fabrication of optical waveguides with different geometries and low absorption losses in the visible and near infrared wavelength range, making this material very attractive for applications in optical devices. Besides this, the mechanical properties of the SiOxNy films are also strongly dependent of the film composition, thus allowing the deposition of thick films with low mechanical stress values, as required by these applications. In this work we report the fabrication and characterization of silicon oxynitride films deposited by PECVD with different chemical composition, which results in low mechanical stress values. The stress response to films heat treatments at different annealing temperatures is analyzed and correlated with Rutherford Backscattering Spectroscopy, ellipsometry, Fourier Transform Infrared Spectroscopy, Small-angle X-ray scattering and X-Ray absorption near edge structure at the Si-K edge measurements. The results show that the stress values vary from compressive to tensile for the as-deposited samples. On the other hand, after annealing a tensile tendency was observed for all samples, except the very low stress as-deposited samples which showed to be very stable with temperature.

B-P031 IRREPRODUCIBILITY IN SOLID ELECTRODE ELECTROCHEMISTRY AND NUCLEATION. THE CASE OF Pb IN H2SO4.

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One of the problems of the Solid Electrode Electrochemistry to study nucleation and growth phenomena on the metal/solution interface is related with the reproducibility of measurements,which in general are in the order of 20%. This value does not arrives to compromise mechanistic conclusions when a reasonable number of measurements are made.This irreproducibility is fundamentally related with the surface preparation procedure,specially,the reproducibility of the roughness factor of the surface which also can affect the nucleation. The problem is that in several cases,mainly for the soft non noble metals,the irreproducibility can arrive to values which make difficult to obtain mechanistic information from the measurements. One of these cases is the Pb electrode. With only polishment of the electrode surface,between each measurement, the irreproducibilities can arrive to 40%. Using a chemical attack between the polishing and the measurements it give about 30%. These results compromise the deduction of any mechanistic description. To improve that it is necessary to increase the reproducibility. In the present paper it is describe one way to do that for Pb electrode to be studied in H2SO4 solutions. The various reasons of the irreproducibility are discussed together with the independence of nucleation on the reproducibility for the studied case. The proposed method is based on a reduction/dissolution technique where the disruptive force of the hydrogen evolution can be also used.

B-P032 SYNTHESIS AND CRYSTAL GROWTH OF Bi12[Ti(1-x)V0.8x]O20

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Sillenite crystals are photorefractive materials with high optical sensitivity and fast response. These properties make the sillenite crystals useful for many advanced and promising applications, such as reversible recording medium for real-time holography or image processing. The general formulae of sillenite compounds can be written as Bi12MO20, with M = Ge, Si, Ti, V, and others. The Bi12TiO20 (BTO) crystals have been considered as the most adequate photorefractive sillenite material because of its practical advantages, such as, lower optical activity, higher electro-optic coefficient and increased sensitivity to red light, which suits the wavelengths of the low-cost He-Ne and diode lasers. Some articles have been published about the optical characterization and photorefractive properties of vanadium doped BTO (BTO:V) crystals, but we do not know any other publication concerning the detailed study of growth conditions of this crystal. This work presents the results of a systematic investigation of crystal growth and solubility in the system (1‑x)Bi12TiO20 : xBi12 [V0,80,2] O20. Using solid-state reaction synthesis we have shown that a continuous solid solution is formed in this system. However, the results set a vanadium concentration limit if we intend to grow good quality single crystals of BTO:V. A detailed study using x-ray powder diffraction, differential thermal analysis, electron microprobe, optical microscopy and selective chemical etching will be presented.

B-P033 CHROMIUM DOPED L-ARGININE PHOSPHATE MONOHYDRATE SINGLE CRYSTALS

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L-arginine phosphate monohydrate (LAP) is a highly transparent monoclinic crystal with attractive properties for frequency conversion of infrared lasers. Many works concerning the growth and characterization of pure LAP crystals, mixed crystals, and chemical analogs of L-arginine phosphate have been published. Also some authors have studied the properties of LAP crystals doped with transitions ions, such Cu, V and Mg. But the effect of chromium ions in this crystal was not studied yet. The aim of this work was investigate the effect of chromium ions in the growth morphology and optical spectra of LAP crystals. Chromium doped LAP crystals were grown by aqueous solution using isothermal solvent evaporation and slow cooling techniques. Chromium was directly added to saturated LAP aqueous solution with different concentrations. Good quality green blueish crystals with dimensions of about 8x8x10 mm were grown. The crystal morphology is severely affected by chromium doping and growth temperature. A detailed study of the crystal growth habit was performed. Optical absorption spectra were collected at room temperature and at 10K, both from crystals and growth solutions. At room temperature, the optical absorption spectrum shows the characteristics bands of Cr3+ which becomemore strong in that spectrum collected at 10K. This temperature dependence indicates that a vibronic mechanism participates in the transitions. Na acurate analysis of the optical spectra will be presented.

B-P034 CARBON ION IMPLANTATION INTO ALUMINIUM: MECHANICAL AND TRIBOLOGICAL PROPERTIES

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Aluminium is a material with industrial applications, mainly for automotive and aerospace technologies. However, even in alloyed form its mechanical and tribological properties are poor. To improve these properties, carbon implantation at 25 keV at fluences of 1 and 2x1017 C+.cm-2 were performed into Al at RT and 400°C. Surface characterizations included Grazing X-ray Diffraction analysis, Elastic Recoil Detection Analysis and Raman spectroscopy. Elastic modulus and hardness profiles were determined by nanoindentation technique according to the Oliver-Pharr method. Friction coefficients were measured by pin-on-plate reciprocal scratch tests. Al4C3 embedded precipitates were identified in sizes from 3-4 nm to 10 nm. Raman analysis showed the presence of carbon clusters (peaks D and G) after carbon implantation. For high temperature, the carbon clusters presence was reduced at expenses of the precipitate size growing. Higher surface hardness (2 GPa) in respect to Al bulk (0.3 GPa), were obtained at RT implantations. Elastic modulus profiles showed a strong dependence on the surface bond type(Al-C-O stoichiometry), changing from 40 - 60 GPa at surface until 70 GPa at deeper regions (Al bulk). Friction coefficient results suggest that the carbon fluence of 2x1017 C+.cm-2 at RT has a running-in behavior larger in comparing to the lower fluence and to the high temperature implantations. The friction coefficient for this sample is 0.25 while Al bulk the value is 0.7.

B-P035 INFLUENCE OF THE HEAT TREATMENT PROFILE ON THE FORMATION, EVOLUTION OF PHASES AND TEXTURING OF AG/BI2212 TAPES

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Tapes of Ag/Bi2212 have been revealed as promising materials for superconducting applications in low temperatures and high fields. The reaction and formation of phases are related to the ceramic densification and to the alignment of the grains, important for the improvement of the superconductor transport properties. Slow cooling from the liquid to the solid state and long annealing heat treatment at temperatures below the melting temperature allow the conversion of the ceramic powder to Bi2Sr2CaCu2O8+ Bi2212. These results do not affect the texturization obtained during the cooling. Composites of the system BiSrCaCuO can be produced in the form of films, tapes, bulk, being emphasized the phases Bi2212 and Bi2223, due to their superconducting characteristic and technological interest. The tapes of Ag/Bi2212 were treated in a horizontal tube furnace under pure flow of O2. The heat treatments consisted of the use of two maximum temperatures of treatment (890oC and 905oC), with two different permanence times (10 minutes and 2 hours), with a cooling rate of -5oC/h, both with a annealing isotherm at 830oC for 5h. Then the furnace temperature was decreased to room temperature in 1 hour. The samples were characterized using by Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XDR). The obtained results will allow the evaluation of formation and evolution of phases in textured samples of Ag/Bi2212 texturing. Financial Support by FAPESP.

B-P036 CHARACTERIZATION OF PURE AND ND-DOPED BAY2F8 SINGLE CRYSTALS GROWTH BY THE ZONE MELTING TECHNIQUE

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In this work, we have studied the synthesis, purification and growth of pure and Nd3+ doped BaY2F8 (BaYF) by the Zone Melting Technique. The starting fluorides YF3, BaF2 and NdF3 were obtained by hydrofluorination from commercial Y2O3, BaCO3 and Nd2O3 (99.99%). These materials were mixed in a nonstoichiometric composition, according to the BaF2-YF3 phase diagram, and melted under HF flow in order to prevent oxide and hydroxyfluoride contamination. The Nd3+ doped BaY2F8 compound was prepared with a nominal dopant concentration of 2 mol%. The synthesized ingots were characterized by TG/DTA and x-ray diffraction measurements. A well-defined endothermic peak was observed at the onset temperature of 955 oC, corresponding to the melting temperature of BaYF. A mass-loss event starts close to 750 oC, ongoing after melting of the compound. Zone melting experiments were accomplished under HF flow using a zone speed rate of 4 mm/h. A large region, bright and transparent, was observed in 80% of the melted ingot. Nd-doped ingots showed an approximately uniform dopant distribution. X-ray powder patterns were measured with samples obtained from synthesized BaYF and single crystals of pure and Nd3+ doped BaYF. The Rietveld method was then applied in order to determine the concentration of phases. The analysis of the synthesized sample showed two phases: YF3 and BaYF, the latter one as a major phase. The analyses of the samples obtained with the single crystals showed BaYF as a unique phase.

B-P037 EFFECT OF AL CONTENT ON THE AG-RICH PRECIPITATES NUCLEATION IN Cu-Al-Ag ALLOYS

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In diffusional phase transformations a new phase, differing from the parent phase in structure and/or composition, is built up, atom by atom, by thermally activated atom transfer across the interface. When this new phase reaches the critical radius and acquires the critical free energy increase, the nucleus become potentially stable and, after the addition of one further atom, it can grow with a continuous decrease of free energy. In this work, the isothermal microhardness changes vs. Ageing time curves, obtained for the Cu-3wt.%Al-4wt.%Ag and Cu-7wt.%Al-4wt.%Ag alloys, in different ageing temperatures, showed a hardness increase always preceded by an incubation period that is shorter the higher the ageing temperature. During this period nucleation is the dominant process and growth starts to dominate at its end. From the variation of the incubation period with ageing temperature and considering that the inverse of the incubation period is proportional to the nucleation rate, it was possible to evaluate the activation energy for the nucleation of Ag-rich precipitates. The value obtained for the Cu-3%Al-4%Ag is close to that found in the literature and the value for the Cu-7%Al-4%Ag is higher, which seems to indicate that the increase in the Al content enhances the solute-solute interaction, decreasing the Ag diffusion rate and increasing the supercritical nuclei formation energy barrier.

B-P038 PURE AND RARE-EARTH DOPED LiYF4 SINGLE-CRYSTALLINE FIBERS GROWN BY MICRO-PULLING-DOWN TECHNIQUE

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Pure and rare-earth doped yttrium-lithium tetrafluoride (YLF) fibers have been successfully grown by the Micro-pulling-down (M-PD) technique. The use of the M-PD technique is original for this purpose since, in general, fluorides are highly reactive and moisture sensitive. The commercial equipment was specifically modified to grow fluoride fibers with good optical quality. In order to obtain the phase YLF a non-stoichiometric composition was used as starting material since YLF is a line compound which melts incongruently. After processing, homogenous and regular diameter (800 mm) YLF fibers were obtained. The growth atmosphere plays an important role on the stoichiometry changes and consequently, on the melting behavior of YLF. Additionally, the YLF phase formation requires a very strict pulling rate in order to assure chemical Homogeneity along the fiber. The stoichiometric phase, in pure YLF fibers grown from a melt with 5 mol% excess of LiF, was colorless, transparent and quite long (»60 mm), after a short transient of the peritectic reaction (»10 mm). Erbium and neodymium doped fibers have shown similar behavior to the pure material, but were colored depending on the nature of dopant. The dopant distributions along the fibers were regular after the initial transient. This work reports a stable growth process for long and high-quality YLF single-crystalline fibers for the first time in the literature.