

## Symposium M

### POSTER SESSION PM1- MONDAY, OCTOBER 9

#### M503 - ABNORMAL GRAIN GROWTH IN SILICON STEEL

Maria das Graças Melo Moreira César (Acesita S.A.), Paulo César Luna (Acesita S.A.), Fabrício Luiz de Alcântara (Acesita S.A.), Marco Antônio da Cunha (Acesita S.A.) and Damiane Tavares Benevides (UnilesteMG)

To investigate the process of growth selection of Goss grains in regular grain oriented silicon steel primary recrystallized samples were annealed to produce partial secondary recrystallization. The orientation distributions of growing secondary grains, matrix grains and the matrix grains neighbouring the secondary grains were measured by the EBSD method. The results showed that primary grains that make CSL S5 boundaries with the Goss oriented grains are preferentially consumed as the secondary grain boundaries migrate, indicating that CSL S5 boundaries play important role in growth selection.

#### M509 - YTTRIA AND GADOLINIA DOPED CERIA NANOPOWDERS FOR FUEL CELL ELECTROLYTES

Dulcina M. P. F. de Souza (DEMA - UFSCar) and Camila Maria Lapa (DEMA - UFSCar)

Ceria solid solutions are good candidates for fuel cell electrolyte for operating at 700°C with high efficiency but have problem with its poor sinterability. Nanopowders have higher driving force for sintering and densification kinetics. 10 mol% yttria doped ceria and 20 mol% gadolinia doped ceria nanopowders were prepared based on Pechini's technique. Several gel compositions and drying temperatures were used. It was possible to obtain powders with nanometric dimensions but it is necessary high careful with organic burn out regarding the powder agglomeration caused by elevated temperatures.

#### M510 - MICROSTRUCTURAL CHARACTERIZATION OF RARE EARTH DOPED CERIA

Dulcina P. F. de Souza (UFSCar) and Guilherme Baltar Crochemore (UFSCar)

SOFCS produce electricity directly from combination of fuel and oxidant agent with high efficiency and in a ecological way, but it operates in high temperature which increases its costs. Many researches are working on the development of intermediate temperature cells, changing the electrolytes based on YSZ for Ce-doped ones, which show the same electrical conductivity at lower temperatures. In this work it was studied the development of Gd and Pr doped Ce microstructure and its relationship with the behavior observed in the electrical characterization of these materials.

#### M512 - A NEAR SINUSOIDAL SURFACE MORPHOLOGY CREATED BY SCAN-INDUCED CONTACT AFM NANOLITHOGRAPHY ON PMMA POLYMER

Mauro Sergio Dorsa Cattani (IFUSP), Ronaldo Domingues

Mansano (EPUSP), Fernanda de Sá Teixeira (IFUSP e EPUSP) and Maria Cecilia Salvadori (IFUSP)

We have produced unidirectional near sinusoidal surface regions on PMMA (poly methylmethacrylate) using tip-scan-induced contact AFM (Atomic Force Microscopy) nanolithography. To fabricate this morphology, parallel lines was scratched with a pyramidal silicon tip through the same scan mechanism used to image samples. The array of these very adjacent lines results on the sinusoidal morphology. The results were mainly dependent on the normal force applied by the tip, the scan size and the number of scan lines. The process was found to be very repetitive for the same experiment conditions.

#### M516 - MORPHOLOGICAL CHARACTERIZATION OF SLAG OF BLAST FURNACE, MICROSILICA AND KAOLIN WASTE COMPOSITE FOR PAVEMENTS

Clesianu Rodrigues de Lima (IME), Nilson Belo Mendonça (IME), Alisson C. Rios Silva (IME) and Felipe F. Costa Tavares (IME)

Recycling of residues is primordial for the preservation of the environment, for the risk of contamination of the ground and the freatic sheet and for the possibility of reduction of cost and consumption of energy. Analyze of the morphology of fracture surface of a composite obtained by cracking, sintering the 1,100 °C/1h of a mixture of 70% siderurgy slag, 20% of kaolin and 10% of microsilica. It can be noticed the presence of empty spheres of different sizes, evidencing the gas formation during baking. The EDS analysis shows the presence of Si, Ca, Al and Mg. Spheres are composed of SiO<sub>2</sub>.

#### M517 - MORPHOLOGICAL ANALYSIS OF THE COMPOSITE POSTCONSUMER PET/ALUMINA

Nilson Belo Mendonça (IME), Felipe F. Costa Tavares (IME) and Clesianu Rodrigues de Lima (IME)

PET has been employed as additives, aiming cost reduction and increasing in material resistance, generating a composite with improved properties when compared to the polymeric matrix without additive. An example is the composite PET/alumina, that present increased friction resistance than the PET matrix without alumina. The composite sample consisted of a 50/50% postconsumer PET/Alumina, obtained by compression at 300°C and room temperature cooling. Observed interaction between the alumina inclusions and the composite matrix. The presence of accumulated alumina was confirmed by EDS.

#### M519 - EBSD STUDY ON THE MICROSTRUCTURE AND TEXTURE OF AN ANNEALED MULTIFILAMENTARY CU-Nb COMPOSITE WIRE

Heide Heloíse Bernardi (EEL-USP), Maria José Ramos Sandim (EEL-USP), Hugo R.Z. Sandim (EEL-USP), Jefferson F.C. Lins (EEIMVR-UFF) and Dierk Raabe (MPI-E)

We report the main results concerning the microstructural evolution and texture development in Cu-15%Nb composite wires upon annealing. The microstructural characterization showed important changes in niobium ribbons in terms of morphology (spheroidization) and further fragmentation for temperatures higher than 700°C. For higher annealing temperatures there is a clear predominance of transverse grain boundaries in the Nb phase, which is strongly <1 1 0> oriented. Boundary splitting and further cylinderization of the Nb ribbons have already occurred in large extent.

**M524 - CHARACTERIZATION OF MINERAL ZIRCON THROUGH FISSION TRACK METHOD, MICRO-RAMAN AND SEM**

Carlos José Leopoldo Constantino (FCT - UNESP), Ana Maria Araya Balan Osório (FCT - UNESP), Carlos Alberto Tello (FCT - UNESP), Airton Natanael Coelho Dias (FCT - UNESP) and Ivaldete da Silva Dupim (FCT - UNESP)

Fission Track Method, FTM, in Zircão, micro-Raman spectroscopy and SEM, have been used to characterize the surface of zircon as function of etch. The micro-Raman results show that as etching time increases it can be seen areas where the density of tracks is uniform and areas where the crystalline structure is completely lost. The SEM results indicate that in the areas where the tracks are uniformly distributed, they are characteristic of zircon ( 55% Si, 15% Zr) and in the areas where the grain lost its crystalline structure, the variations of these chemical elements are significant.

**M528 - FUNCTIONALIZATION OF AFM TIPS IN ORDER TO PROBE THE ADHESION OF PARAFFINS ON METALLIC SUBSTRATES**

Sergio Souza Camargo Jr. (COPPE/UFRJ), Marta Elisa Rosso Dotto (COPPE/UFRJ) and Claudio Marcos Ziglio (CENPES)

For the oil industry, precipitation and adhesion of waxes are critical problems that may result in the clogging of pipelines therefore, is very important to obtain information about adhesion between paraffin and metallic substrates. In this work, the functionalization of AFM probes with thin films of paraffin is studied to estimate the interaction forces between paraffin and metallic substrates by force curves. The functionalization of tip was obtained by evaporation technique. Parameters such as the spring constant, jump-in/out and pull-off distances, work and forces adhesion were analyzed

**M530 - INFLUENCE OF GRAIN SIZE AND AL AND MN ADDITIONS ON THE MAGNETIC PROPERTIES OF NON-ORIENTED ELECTRICAL STEELS WITH 3%SI**

Marco Antonio da Cunha (Acesita S.A.), Luiz Paulo Mendonça Brandão (IME) and Rodrigo Felix Cardoso (IME)

Steels for electrical use or electrical steels play a vital role in the generation, transmission, distribution and use of electrical power and are one of the most important magnetic materials of great relevance for the industry worldwide. There are three principle types: Grain oriented electrical steels are 3% silicon-iron alloys developed with a grain orientation to provide very low power loss and high permeability in the rolling direction, for high efficiency transformers; Non oriented fully processed electrical steels are iron-silicon alloys with varying silicon contents and have similar magnetic properties in all directions in the plane of the sheet and Non oriented semi processed electrical steels, both mainly for electrical motors. These steels are mainly used in electrical engines and transformers, since the energy efficiency of these electrical machines depends on magnetic permeability and "magnetic losses" associated with alternating current excitation. Grain size has a very strong effect on magnetic losses. As grain size increases, hysteresis losses decrease, but the so called anomalous losses increase. Therefore, there m [1]. Another important is an optimal grain size, between 100 and 150 parameter in these materials is the crystallographic texture, due to a high anisotropy of the magnetic properties caused by the easy magnetization of iron in the <100> direction relative to all other directions, since the easy crystals is the magnetization axis in Fe <001> direction and the hard magnetization axis is the <111> direction [2]. The purpose of this work was to study the effect of the Al and Mn additions and the influence

of grain size on the magnetic properties of fully processed low-carbon grain non-oriented (GNO) electrical steel with about 3%Si. This took place through processing of the steel since forged ingots by hot rolling, coiling simulation, temper rolling, hot-band annealing, cold rolling and final annealing. In each stage of processing microstructural and crystallographic texture analysis were carried out using "Leica" model DMRM optical microscope and X-ray diffraction, respectively. Figure 1 shows the microstructures of the studied steel with Al addition: (1a) in the state of hot-band annealing at 800°C and (1b) the final annealing at 1000°C. On the other hand, figure 2, shows the same steel with Mn addition as it was processed: (2a) Hot rolling, (2b) Hot-band with temper rolling and annealed at 800°C, (2c) cold rolling and (2d) final annealing at 1000°C. Comparing the two pictures, it can see that, the sample with Mn addition, in the state of final annealing, presented larger grain size in relation to the other one with Al addition. This larger grain size gives rise to a lower magnetic loss in the case of Mn addition, which is in accordance with the literature [1]. In the case of the hot-band annealing, an increase in the grain size for both the samples is observed (figures 1a and 2b). This increase of hot-band grain size led to an increase of the Goss texture component, {110}<001>, after final annealing which favored the magnetic permeability and is in accordance with the literature [3,4].

**M543 - MICROSTRUCTURAL ANALYSIS OF MO /STEEL SAE 1045 INTERFACE OBTAINED BY PLASMA SPRAY SUBMITTED TO THERMAL TREATMENTS**

Joao Roberto Moro (USF), Jose Eduardo M. Villas Boas (USF), Fernanda Regina Francisco (LNLS) and Osmar Roberto Bagnato (LNLS)

This work proposes to study the interfaces formed between films of Mo laid down by plasma spray over carbon steel samples surfaces under the effect of thermal treatments under vacuum to promote the interface homogenization by means of atomic diffusion with consequent pores reduction. After the diamonds deposition, probably due to superficial tensions, there was a loosening of the Mo/diamond films. The superficial chemical analysis in the Mo film indicates Fe presence, showing that there was diffusion in this sense. A significant diffusion of Mo in the steel was not identified.

**M544 - ELECTROLYTIC AND CHEMICAL DEPOSITION ANALYSIS OF NICKEL IN BRAZED JOINTS, TYPE AL2O3/CUAG/FE-NI-CO**

Osmar Roberto Bagnato (LNLS) and Osmar Rodrigues Gacia Jr. (LNLS)

The objective of this work is to characterize the union between metals and ceramics, from a metallization layer of nickel-phosphorus and electrolytic nickel under alumina substrate (Al2O3), producing brazed joints in high vacuum, type Al2O3/CuAg/Fe-Ni-Co. SEM was used to determine the nickel layer thickness, the particles medium size of Ni-P and the film uniformity. Results indicate that the film is stable and continuous even after the operations in high temperatures. The braze surfaces were also studied and they have showed the presence of intermetallics that can weaken the filler region.

**M550 - DENSIFICATION OF BORON CARBIDE: ANALYSIS BY X-RAY RIETVELD AND SEM**

Roberto Ribeiro Avillez (PUC), Célio Albano Costa (UFRJ), José Brant Campos (INT) and Pedro Augusto Cosentino (CTEx)

High energy milling was used to produce submicron boron carbide (B4C) particles to increase its final density. The milling process introduced high

quantity of ZrO<sub>2</sub> nanoparticles from the milling balls. During sintering, the ZrO<sub>2</sub> nanoparticles reacted with the B<sub>4</sub>C and other carbides used as sintering additives. The resulting phases were analyzed by Rietveld X-ray Diffraction and scanning electron microscope (EDS/SEM). The results clearly showed the contamination of the B<sub>4</sub>C powder by ZrO<sub>2</sub> (from the milling balls) and iron (from the milling walls) and, for the sintered parts, many different phases were identified by the combination of both techniques.

### **M553 - BLENDS OF PCL/PLGA: IN VITRO DEGRADATION OF TISSUE ENGINEERING SCAFFOLDS**

Cecilia Zavaglia (FEM/UNICAMP), Kátia Silva (FEM/UNICAMP), Samuel Barbanti (PUCSP) and Eliana Duek (PUCSP)

Synthetic biodegradable polyesters have been used as scaffolds in tissue engineering and in various fields of medicine. The present work investigates the degradation process of PCL/PLGA blends in phosphate-buffered. Membranes of blends were prepared for solvent evaporation process in proportion of 70/30, 50, 50 and 30/70. The in vitro degradation was evaluated using PBS, pH 7.4, maintained at 37°C. After periods of 0, 30, 60 and 140 days, samples were observed by SEM. The results show that higher the proportion of PLGA in the blend, higher is the morphologic degradation. The degradation process presented suggests a preferential attack of the amorphous areas directly proportional with the time of exhibition in vitro.

### **M555 - SCANNING FORCE MICROSCOPY AND SPR CHARACTERIZATION OF ELETROPOLYMERIZED THIN FILMS DERIVATED FROM [Fe(BR-PH-TERPY)2](PF6)2 COMPLEX**

Henrique Eisi Toma (IQ-USP), Sérgio Hiroshi Toma (IQ-USP), Juliano Alves Bonacin (IQ-USP) and Marcelo Nakamura (IQ-USP)

Thin films derived from [Fe(Br-ph-terpy)<sub>2</sub>](PF<sub>6</sub>)<sub>2</sub> were prepared and analysed by means of SPM, SPR and optical microscopy. Topographic characterization was made by AFM indicating significant changes on the morphological domains as compared with the momeric thin film, after the electropolymerization process. Conductivity behavior of thin films was evaluated by Conductive AFM measurements allowing to access the rectification behavior. An abrupt behavior change was observed below -4V, ascribed to the reduction of the ph-terpy ligand in the polymeric film.

### **M573 - THE INFLUENCE OF DIFFERENT THERMOMECHANICAL TREATMENTS ON THE MICROSTRUCTURE OF A TRIP STEEL**

Cláudia Nazaré dos Santos (IME), Almir Gonçalves Vieira (CEFET/MG) and Carlos Sérgio da Costa Viana (IME)

The present data confirm that TRIP effect assisted steels offer a good compromise between strength and ductility as a result of their microstructure being composed by pro-eutectoid ferrite, austenite transformation products and significant amounts of retained austenite. Here the microstructure of two TRIP steel conditions were compared.

### **M576 - SYNTHESIS OF NANOMETRIC ZNFE2O4 BY THE POLYMERIC PRECURSOR METHOD**

Severino Guedes Lima (UFPB), Carlos Alberto Paskocimas (UFRN), Elson Longo Silva (UNESP), Luiz Bastos Soledade (UFPB), Camila Soares

Xavier (UFPB), Ieda Garcia Santos (UFPB) and Antonio Gouveia Souza (UFPB)

In this work, the synthesis of ZnFe<sub>2</sub>O<sub>4</sub> was done by the polymeric precursor method. The resins were prepared from the polymerization between the metallic citrate and ethylene glycol in pH values of 2 and 7. The first calcination was done at 300 °C/1 h, leading to the formation of the powder precursor. The powder precursors were calcined in O<sub>2</sub> atmosphere at 300 °C/ 12 h. The material was further calcined between 700 and 1000 °C for crystallization. An unmilled precursor in pH 7 was also calcined at 1000 °C, for comparison. Characterization was done by XRD and SEM.

### **M584 - APPLICATION OF ATOMIC FORCE MICROSCOPY IN THE IDENTIFICATION OF MICROSTRUCTURAL PHASES AND CONSTITUENTS IN LOW CARBON MICROALLOYED STEEL**

Margareth Spangler Andrade (CETEC), José Mário Carneiro Vilela (CETEC), Tiago Felipe de Abreu Santos (CETEC) and André Barros Cota (UFOP)

In the past decades many investigations were performed in order to identify the characteristic microstructure of low carbon microalloyed steels, which consist of a complex mixture of bainite, martensite, pearlite, ferrite and a MA constituent (martensite and retained austenite). In the present work atomic force microscopy analysis was performed in a Nb microalloyed steel submitted to different heat treatments to induce distinct microstructures. The results were compared to optical microscopy analysis. It was shown that the AFM technique is useful to identify different phases in steels.

### **M586 - HOT-BAND TEXTURE OF A 0.6% MN ELECTRICAL STEEL**

Nilza Zwirman (CSN), Jorge Trota Filho (Inmetro), Leandro R. Lidizio (Inmetro), Taeko Yonamine (Inmetro), Marcos Flavio de Campos (Inmetro), Augusto C. L. de Oliveira (CSN), Verner Wolgien (CSN), Carlos A. Achete (Inmetro) and Paulo R. Rios (EEIMVR-UFF)

High manganese electrical steel are of industrial interest because Mn increases the electrical resistivity, avoiding the addition of other elements like Si, Al and P. This study is about the effect of an annealing at 600°C on the grain size and texture of a hot band. A time greater than 10h is necessary for producing grain size larger than 100 microns. The hot band grain size greater than 100 microns leads to an improvement on the texture because the intensity of {111} decreases, the intensity of {310} increases, while the intensity of {100} planes almost does not change.

### **M587 - THE MORPHOLOGY AND COMPOSITION OF OXIDE SCALES FORMED AT DIFFERENT TEMPERATURES ON THE AISI 52100 STEEL**

Luís Frederico P. Dick (UFRGS), C. Marckmann (UFRGS), L. G. Andres (UFRGS) and Pedro Carlos Hernandez Jr (UFRGS)

In this work wire rod samples of the AISI 52100 steel were submitted to three different oxidation conditions concerning coiling temperature and cooling rate during industrial hot rolling. The structure of this "hot-rolling"-scales was compared with that of laboratory scales produced at different constant temperatures simulating the coiling conditions. The oxides were characterized by SEM-EDS and by quantitative X-ray diffraction (Rietveld method). Higher coiling temperatures and higher cooling rates led to thicker

and more porous scales. Porosity and oxide fractions were determined.

**M590 - EBSD QUALITY INDEX MAPS AS TOOLS TO IDENTIFY AREAS WITH HIGH DENSITY OF DISLOCATIONS IN COLD ROLLED STEELS**

Fernando J. G. Landgraf (EPUSP), Marcos Flavio de Campos (Inmetro), Maria do Carmo Amorim da Silva (EPUSP) and Ivan G. S. Falleiros (EPUSP)

The TSL-EBSD software suggests the use of IQ ("Index of Quality") to study deformed materials. The IQ parameter can give a qualitative description of the strain distribution in a microstructure. Areas with high dislocation density tend to appear darker in the IQ images. Grain boundaries are readily identified, because the diffraction pattern will be composed of a mix of the two patterns of both grains leading to a lower quality pattern. In the present study, IQ maps of deformed electrical steels were evaluated. The studied steel has 1.25% of Si, and was cold rolled from 0 to 50% of reduction.

**M599 - HOMOGENIZATION EVALUATION OF FE-19.5CR-5NI MECHANICAL ALLOYED MIXTURE USING EDS COMPOSITIONAL LINESCAN**

José Deodoro Trani Capocchi (EPUSP), Eduardo Franco Monlevade (INdT), Evaldo Toniolo Kubaski (EPUSP), Cleverson Moinhos (UEPG) and Osvaldo Mitsuyuki Cintho (UEPG)

The Fe-19.5Cr-5Ni high purity powder mixture was mechanical alloyed in a high energy ball mill in order to produce a duplex stainless steel. Samples of milling products were formed in pastilles and heat treated. The partial linescan EDS analysis for Ni and Fe shows the gradually homogeneity increase according milling time by the shape similarity evolution in this way. Despite of the same structure (CCC) the Cr has more difficult to solve in Fe then Ni (CFC) probably in function of the higher hardness and brittleness presented by the Cr. The ductility and the lower hardness of Ni powder collaborate for the cold welding of particles during the mechanical alloying process.

**M600 - MICROSCOPY ANALYSES OF MORPHOLOGIC, STRUCTURAL AND BIOLOGIC CHARACTERISTICS OF CAP/COL COMPOSITES DOPED WITH DIVALENT CATIONS**

Herman Sander Mansur (UFMG), Luiz Guilherme Dias Heneine (FUNED), Cintia Tereza Pimenta Araújo (UFVJM) and Maria Helena Santos (UFVJM)

Hydroxyapatite (HAP) and hydroxyapatite-tricalcium phosphate (HAP TCP) doped with Zn+2 were synthesized and produced with collagen and HAPZn/COL and HAPZn TCP/COL composites were developed. SEM/EDX showed morphologic, structural and chemical composition similar to their constituent materials. The XRD spectrum showed characteristic picks of the hydroxyapatite and organic material. The alkaline phosphatase production of osteoblasts in presence of the composites showed cytocompatibility. The developed biocomposites could be used for rebuilding bone tissue in medical and dentistry applications.

**POSTER SESSION PM2 - TUESDAY, OCTOBER 10**

**M513 - MORPHOLOGICAL ANALYSIS OF COMMERCIAL CATALYSER NIOBIUM PHOSPHATE (NBOPO4)**

Wilma Araújo Gonzalez (IME), Clesianu Rodrigues de Lima (IME), Felipe F. Costa Tavares (IME) and Nilson Belo Mendonça (IME)

Niobium phosphate is used as catalysers in cracking and dehydration reactions. Aimed to analyze the morphology, a commercial niobium phosphate catalyser. Morphological characterization was made by scanning electron microscopy (SEM). SEM analysis revealed uniformity in grain size, which can have direct influence in the outcome of catalytic tests. EDS analysis shows the composition of the individual grains containing the elements Nb, P and O. Statistical analysis of mean and dispersion were carried out using 15 different EDS spectra. The samples present good homogeneity and surface distribution.

**M515 - MORPHOLOGIC ANALYSIS OF CATALYSER PHOSPHORIC ACID SUPPORTED ON SILICA (H3PO4/SIO2) USED IN CATALYTIC TESTS FOR BIOFUEL PRODUCTION**

Clesianu Rodrigues de Lima (IME), Felipe F. Costa Tavares (IME), Nidia M. Ribeiro Pastura (IME), Wilma Araújo Gonzalez (IME) and Nilson Belo Mendonça (IME)

The present work aimed the evaluation of morphology, homogeneity and surface distribution the catalyser phosphoric acid supported in silica (H3PO4/SiO2) containing 12.2% (p/p). We also evaluated the distribution and compositional analysis using a SEM coupled to an EDS. Fifteen different regions of each sample were analyzed. The percentage of each analyzed region is presented as a histogram. The analysis revealed uniform grain size and agglomerates. Catalyser grains presented spherical form which can influence the effectiveness of the catalytic reactions

**M522 - CERIA- BASED CERAMICS: EFFECT OF SINTERING AID ON THE MICROSTRUCTURAL DEVELOPMENT**

Dulcina Pinatti de Souza (UFSCar DEMa) and Glauber Silva Godoi (UFSCar DEMa)

Electrolytes materials for using in solid oxide fuel cell (SOFC) are required to show good sinterability and electrical properties. In this work, two ceria-based ceramics for using as eletrolytes, yttria-zirconia doped ceria (CZY) and yttria-zirconia doped ceria with sintering aid (CZYA) were prepared by oxide mixture process in a vibratory mill. The results allow to conclude that CeO2 raw material with 99,9% purity is not appropriated to prepare yttria-zirconia doped ceria for applications where high electrical conductivity is required even using alumina as sintering aid.

**M523 - LSM-YSZ INTERFACE: MICROSTRUCTURE AND ELECTRICAL RESISTIVITY**

Charles Luis Silva (DEMA/UFSCAR), Dulcina Ferreira De Souza (DEMA/UFSCAR) and Thiago Dias (DEMA/UFSCAR)

The solid oxide fuel cells efficiency can be increased using LSM-YSZ composites cathodes. However when the interface LSM-YSZ is maintained at high temperature inter-diffusion process occurs modifying the microstructure and electrical properties near the interface. The SEM analysis of the interface

LSM-YSZ shown that inter-diffusion promoted a YSZ dense layer near the LSM and exaggerated grain growth occurred in this region. The electrical conductivity of the YSZ grains of this region is lower than pure YSZ.

### **M525 - A METALLOGRAPHIC STUDY OF THE BARZILIAN METEORITE PATOS DE MINAS (HEXAHEDRITE), MINAS GERAIS, BRAZIL**

Loiva Lizia Antonello (Museu Nacional), Maria Elizabeth Zucolotto (Museu Nacional) and Franklin Santos Antunes (PUC)

The Patos de Minas a meteoric mass of 32 kg is a normal hexahedrite. When etched exhibits a mottled appearance which is mainly due to differences in phosphide populations. Neumann bands extend across the whole section from border to border showing that the meteorite is one ferrite grain. A sulphide nodule is formed of daubreelite  $\text{FeCr}_2\text{S}_4$  and troilite with FeS. The sulphide nodule is bordered by a rim of schreibersite with  $(\text{FeNi})_3\text{P}$ . Very interesting is the presence of a silicate with Al, Si, Cl, Ca, Cr and Fe Ureyite? Silica was not mentioned as occurring in any other known hexahedrite and is not in agreement with the formation process of hexahedrite.

### **M526 - SURFACE INSPECTION OF SILICON MICROTIP FABRICATION PROCESSES**

Edmundo Silva Braga (DEMIC/FEEC/UNICAMP), Marco Antonio Robert Alves (DEMIC/FEEC/UNICAMP), Marcelo Nogueira Tiroli (DEMIC/FEEC/UNICAMP), Carlos Luciano Danieli (DEMIC/FEEC/UNICAMP) and Douglas Freitas Takeuti (DEMIC/FEEC/UNICAMP)

We report a fabrication process of silicon microtips using amorphous hydrogenated carbon or silicon oxide thin films as masks for silicon etching. A two-stage reactive ion etching plasma process has been employed to fabricate the microtips, in which the first stage was carried out in  $\text{SF}_6/\text{O}_2$  plasma and the second stage used pure  $\text{SF}_6$ . During the fabrication process, scanning electron microscopy was employed as a surface inspection tool, in particular to study the residual structures that were observed under the masks.

### **M529 - GRAIN BOUNDARY POTENTIAL BARRIERS INVESTIGATED THROUGH ELECTRIC FORCE MICROSCOPY IN LA-DOPED BARIUM TITANATE THERMALLY ATTACKED WITH DIFFERENT TEMPERATURES**

Pedro Íris Paulin Filho (UFSCar) and Marília Wellichan Mancini (UFSCar)

PTC thermistors are made of polycrystalline n-type  $\text{BaTiO}_3$  with a low concentration of donor dopant and obtained with an appropriated sintering temperature and cooling cycle after sintering. Such procedure leads to formation of grain boundaries with high electrical resistance potential barriers that surround the semiconductor grains. In this work we investigate the grain boundaries potential barriers in the semiconductor La and Mn doped barium titanate PTC system ( $\text{Ba}_{1-x}\text{La}_x\text{TiO}_3$ ),  $x=0,004$ , by atomic force microscopy (AFM) operating in the electric force microscopy mode (EFM).

### **M537 - NANOLITHOGRAPHY OF DIAMOND LIKE CARBON FILMS USING SCANNING TUNELLING MICROSCOPY**

Valéria Batista Nunes (PUC-Rio) and Rodrigo Prioli (PUC-Rio)

In this work, we present the use of the tunneling electrons, from a STM tip, to induce nano-sized graphitization to diamond like carbon films. The experiments are made in a UHV system with integrated STM. The STM is operated in the constant current mode at relatively high voltage and low current. We are trying to control the V and I parameters on the patterning process for the each surface modification. After the local graphitization in the UHV-STM, both the modified areas and original films are investigated by STM-based current-distance spectroscopy.

### **M538 - DUAL BEAM FIB/SEM – UNIVERSAL TOOL FOR NANOSCIENCE**

Stanislav A. Moshkalev (CCS-UNICAMP), Carla Verissimo (CCS-UNICAMP), Alfredo Rodrigues Vaz (CCS-UNICAMP) and Jacobus W. Swart (CCS-UNICAMP)

In 1974 Selinger and Fleming demonstrated a “unique microfabrication technique” by the incidence of a focused ion beam (FIB) on a silicon surface. Doped regions written in a silicon substrate by a focused boron beam were obtained. This result led to a maskless implantation of dopants into silicon surface [1]. A continuous development on the focused ion beam (FIB) technology has been observed. A FIB instrument is similar to a scanning electron microscope (SEM) operating with a focused ion beam. Usually, gallium is used to generate an ion beam ( $\text{Ga}^+$ ) due to its properties (liquid state near room temperature and the ability to be focused to a very fine probe size). A schematic diagram of a liquid metal ion source (LMIS) and lens system of a FIB is showed in the Fig. 1 [2]. The incidence of a focused high-energy ion beam can promote a number of phenomena during the interaction with the target material producing, for example, the ejection of secondary ions which can be used for the image formation [3]. In this direction, Anazawa et al. [4] reported the construction of a scanning ion microscopy (SIM) facility with gallium ion source. Several examples about the development of the FIB technology in the 80's can be cited: Orloff and Sudraud [5] proposed a design for a focused ion beam system for lithography and implantation with an ultra-fine 10-nm source; Kato et al. [6] pointed out the advantages of the FIB technology for the fabrication of sub-micron structures and other maskless processes, including scanning ion microscopy, maskless ion implantation, maskless etching, resist exposure and maskless deposition. Due to the characteristics of this technology, FIB instrumentation has been used mainly to improve semiconductor devices and to fabricate devices for microelectronics. However, the continuous development on the FIB technology has promoted its application in a variety of purposes [7]. For example, since 90's, FIB technology is becoming a powerful tool for transmission electron microscopy (TEM) sample preparation [2,8,9]. Nowadays, the state of the art about FIB technique is the attracting interest for fabrication of nano-devices. The use of FIB as a milling tool allows quick and direct probe patterning by sub-micron resolution sputtering removal, avoiding the multiple-step processing of conventional lithography [10]. In addition, the possibility to design the active area is a great advantage for nano-scale application where coupling between sample and sensor is crucial. The milling operations are achieved through site specific sputtering of the target material. Since sputtering is the base for the milling operations, is important to understand the ion beam-solid interactions and the sputtering process. When an energetic incident ion hit with a target material, it will produce a collision cascade in the target material. If a surface atom receives enough of a normal component of momentum from collision cascade to overcome the surface binding energy, the surface atom leaves it and is said to be sputtered [3]. The factors that affect sputtering include the atomic number, energy, and angle of incidence of the ion beam, the atomic density of the target, surface binding energy of the target, and crystallographic orientation of the target [11]. The FIB system model used in our experiment is a dual beam from FEI (model Nova 200). This system combines a ion beam column with a scanning electron microscope (SEM)

working at coincidence on the sample. This allows us to monitor ion beam processing with simultaneous SEM imaging. The FIB column is equipped with a Ga<sup>+</sup> source and has a minimum spot size of 5 nm at 30 kV whereas the SEM column has a thermal field emission gun which can achieve a 2 nm resolution over a wide energy range. References: [1] R. L. Selinger and W. P. Fleming, *Journal of Applied Physics* 45 (1974) 1416-1422. [2] L. A. Giannuzzi and F. A. Stevie, *Micron* 30 (1999) 197-204. [3] B.W. Kempshall et al, *J. Vac. Sci. Technol. B* 19(3), (2001) 749-754 [4] N. Anazawa, R. Aihara, M. Okunuki and R. Shimizu (1982) Development of a gallium ion source scanning ion microscope and its applications. *Scanning Electron Microscopy IV*, AMF O'Hare, Chicago: 1443-1451. [5] J. Orloff and P. Sudraud *Microelectronic Engineering*, 3 (1985) 161-165. [6] T. Kato, H. Morimoto, K. Saitoh and H. Nakata, *J. Vac. Sci. Technol. B* 3 (1985) 50-53. [7] M. J. Vasile, R. Nassar, J. Xie and H. Guo, *Micron* 30 (1999) 235-244. [8] A. De Veirman and L. Weaver, *Micron* 30 (1999) 213-220. [9] M. Sugiyama and G. Sigesato, *Journal of Electron Microscopy* 53 (2004) 527-536. [10] Takashi Nagase et al. *Microelectronic Engineering* 78-79 (2005) 253-259 [11] M. Natasi, J.W. Mayer and J.K. Hirvonen, *Ion-Solid Interactions: Fundamentals and Applications* (Cambridge University Press, Cambridge, UK, 1996) 218-250

### M541 - NEW BUILDING FOR THE ELECTRON MICROSCOPY FACILITY AT LNLS

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An expansion of the Electron Microscopy Laboratory at LNLS is on course by the installation of two new Transmission Electron Microscopes. The identification of possible vibrations and magnetic field sources, such as complementary equipment, is extremely important in order to minimize their effects. A complete analysis of the actual HRTEM, SEM-FEG, SEM-LV and Chillers rooms was realized to identify these disturbance sources. Thus, the previous knowledge of site and equipment conditions and possible sources will reduce the time and cost in the new building design and construction.

### M547 - GROWTH OF SELF ORGANIZED INAS QUANTUM DOTS ON PATTERNED INP SUBSTRATES

Maurício Pamplona Pires (UFRJ), Rodrigo Prioli Menezes (Puc-Rio), Henrique Duarte Filho (Puc-Rio), Artur Lopes (Puc-Rio) and Patricia Lustosa Souza (Puc-Rio)

In this work, the self organization of indium arsenate (InAs) quantum dots grown on indium phosphate (InP) patterned substrates using atomic force microscopy is presented. The influence of the dot growth parameters and different lithography methods were investigated with the aim on the control of the quantum dots density, lateral position and size distribution on the substrate. The lithographed patterns with different quantum dot densities will be used on the study of the coupling between dots. We aim to determine the relative stray field contribution on the energy levels and gap of the quantum dot network.

### M551 - MORPHOLOGICAL AND STRUCTURAL CHARACTERIZATION OF ALUMINA NANOTUBE

Nelcy Della santina Mohallem (UFMG), Cristina Fontes Diniz (UFMG) and Karla Balzuweit (UFMG)

Alumina nanotubes were obtained by hypercritical drying of wet gels prepared by sol-gel process. The structural characterization, particle size

and shape of these nanoparticles were evaluated by means of a scanning electron microscope and high resolution transmission electron microscope.

During heating up to 1200°C, the powder phase evolution led to the formation of a mixing of transition nanoalumina phases gamma, delta and theta from boehmite.

### M552 - THERMAL DEACTIVATION STUDY OF AUTOMOTIVE CATALYST BY SCANNING ELECTRON MICROSCOPY

Marcelo Machado Viana (UFMG), Ronald Arreguy Silva (UFMG) and Nelcy Della Santina Mohallem (UFMG)

The deactivation of automotive catalyst by thermal effects was studied using scanning electron microscopy and energy dispersive spectroscopy analyses to characterize the variation of contaminant relative to concentration and changes in the catalyst morphology with the operation temperature. Other complementary techniques such as thermal analyses, X-ray diffractometry, picnometry and gas adsorption were also used to analyze the structural and textural changes in the material.

### M562 - SURFACE MORPHOLOGY OF ELECTRODEPOSITED NI ON GAAS

Wagner Figueiredo (Dep. Fisica - UFSC), Giovanni Zangari (University of Virginia), David M. Kirkwood (University of Virginia), Maximiliano Luis unford (Dep. Fisica - UFSC), Vinicius Claudio Zoldan (Dep. Fisica - UFSC) and André Avelino Pasa (Dep. Fisica - UFSC)

In this study, the surface morphology of electrodeposited Ni on single crystal GaAs (001) was investigated by atomic force microscopy (AFM). The images show granular deposits with stepped contours typical of single-crystalline grains. The correlation length correlates very well with the size of the grains, indicating that the layers grow as columns with diameter increasing with thickness. This growth mechanism is observed for layers with thicknesses in the range of 10 to 500 nm at a deposition rate of ~0.5 nm/s.

### M565 - MAGNETIC AND STRUCTURAL PROPERTIES OF FESINBCUB THIN FILMS DEPOSITED BY RF SPUTTERING

Rubem Luis Sommer (CBPF), Ricardo Barreto da Silva (UFSM), Marcio Assolin Correa (UFSM) and Alexandre Da Cas Viegas (UFSC)

Amorphous thin films, precursors of nanocrystalline Finemet material, deposited by RF magnetron sputtering under different conditions present distinct magnetic and structural properties. This work presents the magnetic and structural properties (studied by TEM) of 500nm thick samples of Fe<sub>73.5</sub>Cu<sub>1</sub>In<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub>, deposited with the substrates moving at constant speed through the plasma, and with the substrate stopped over the sputtering gun. Structural analysis obtained by cross section transmission electron microscopy (TEM) images, reveals spinodal decomposition and self-ordered structure.

### M567 - METROLOGY OF ENDODONTIC INSTRUMENTS THROUGH SEM AND DIGITAL IMAGE ANALYSIS

Sandra Rivera Fidel (Endodontia/UERJ), Rivail Antonio Sergio Fidel (Endodontia/UERJ), Claudia M. Reis (DCMM/PUC-Rio), Sidnei Paciornik (DCMM/PUC-Rio), Otavio da Fonseca Martins Gomes (CETEM/MCT e

DCMM/PUC-Rio), Marcelo Rolla (Endodontia/UERJ)  
and Helio Pereira Lopes (Endodontia/UNESA)

This work proposes a new method for the metrology of endodontic instruments based on SEM and digital image analysis. A specific sample holder was developed to allow automating image acquisition. For each file, 4 adjacent fields at 100x are automatically imaged, to cover its entire length with the required magnification and resolution. The images are processed and analyzed by AxioVision and KS400. D3, D8 and tapering of 8 instruments FKG Pre RaCe were measured. The obtained results are consistent with manual measurements but the proposed method is faster and much less subjective.

### **M575 - SIMULATION AND ANALYSIS OF EGG SHELLS SURFACES**

M P M A Baroni (INPE), J A K Freire (UFC), M R Oliveira (UFC),  
E F Costa (UFC), L M Rebelo (UFC), R R Rosa (INPE) and J Pontes (UFRJ)

In this work, we present a computational study of KPZ equation, which due to its physical properties; the solution is adopted to simulate the egg shells surfaces. The analyses of the simulated and real AFM surfaces are done using both scaling concepts and the Gradient Pattern Analysis. The samples of egg shells had been dealt with deionized water, dry with nitrogen spurt and characterized by means of Atomic Force Microscopy (AFM).

### **M579 - QUANTITATIVE FORCE MODULATION MICROSCOPY**

Rodrigo Prioli (PUC-Rio) and Clara Muniz Almeida (PUC-Rio)

It was developed a calibration procedure of a force modulation microscope (FMM) in order to measure mechanical properties whit high spatial resolution. Modulating the force in an atomic force microscope opens the possibility to characterize the local elastic properties of a surface trough tip-sample interaction. This system was built and tested with two different polymers samples: PS/PMMA and PVC/PHB blends. There was enhancing in the contrast caused by different polymers present in the surface showing clearly regions with different mechanical properties.

### **M580 - DEVELOPMENT OF A NANOPARTICLES GENERATOR**

Antonio Domingues dos Santos (IFUSP), Sergio Antonio  
Romero (IFUSP) and Simone Camargo Trippe (IFUSP)

We are developing a nanoparticles (NPs) generator. The methodology of the NPs generator is based on physical processes of atomic clustering. In order to simplify the project we are using a magnetron sputtering gun as an ionic source. In the frontal part of the gun exist a small chamber operated under very low vacuum, where is possible to cooling down the ionic flux and permits its condensation to promote the cluster formation. As the system has three sputtering guns it is possible by co-deposition to insert magnetic (metallic or dielectric) NPs in a metallic or dielectric (magnetic) matrix.

### **M583 - CHARACTERIZATION OF TIN AND TIC FILMS DEPOSITED BY MAGNETRON SPUTTERING USING NANOINDENTATION AND ATOMIC FORCE MICROSCOPY**

André Paulo Tschiptschin (EPUSP), Roberto Martins Souza (EPUSP),

Homero Santiago Maciel (ITA), Maria Cristina Moré Farias (EPUSP),  
Ivo Castro Oliveira (ITA), Marcos Massi (ITA),  
Abel André Cândido Recco (EPUSP) and Clarice T. Kunishi (EPUSP)

TiN and TiC films were deposited by reactive magnetron sputtering. The ratio of hardness to Young's modulus (H/E) was determined using the nanoindentation technique. The chemical composition of the films was obtained by WDX and the surface topography through AFM. The H/E ratio depends on the proportion of the reactive gas in plasma atmosphere, which is related to the chemical composition of the films

### **M588 - NANOLITHOGRAPHY OF SILICON SURFACES USING ATOMIC FORCE MICROSCOPY**

Diego Kops Pinto (LSI-EPUSP)  
and Sebastião Gomes dos Santos Filho (LSI-EPUSP)

Atomic Force Microscopy (AFM) has recently presented a strong breakthrough in the field of nanolithography. In this work, it was investigated local anodic oxidation (LAO) using silicon nitride tips onto p-type (111) silicon surfaces and the LAO mechanism with the aid of numerical simulations. It was noteworthy that a last cleaning based on ammonia and hydrogen peroxide (SC1) was a necessary pre-conditioning for anodic oxidation to occur using anodic potentials ranging from 15 to 22.5V. Also, it was established that the higher the time interval and the number of scans, the higher the thickness.

### **M589 - HIGH RESOLUTION ANTIDOT LATTICES IN ALGAAS/GAAS PARABOLIC QUANTUM WELL HETEROSTRUCTURES OBTAINED BY FIB**

Stanislav Moshkalev (CCS-UNICAMP), Alfredo Rodrigues Vaz (CCS-  
UNICAMP), Nilo Mauricio Sotomayor (UFT)  
and Guennadii Mikhailovich Gusev (IFUSP)

In the present work we explore and report the possibility of fabrication of sub-micron diameter antidot lattices in high mobility AlxGa1-xAs/GaAs semiconductor heterostructures containing two and three-dimensional electron gases (2DEG, 3DEG), by means of Focused ion beam (FIB). The FIB technique used as a milling tool allows quick and direct probe patterning by sub-micron resolution sputtering removal, avoiding the multiple-step processing of conventional lithography.