3RD BRAZIL MRS MEETING

October 10-13, 2004

SYMPOSIUM I:

MICROANALYSIS AND ANALYTICAL MICROSCOPY OF MATERIALS (co-sponsored by SBMM as part of the 9th Micromat (Brazilian Symposium on Materials Microscopy)

Symposium Organizers:

Fernando Galembeck (Unicamp)

Carlos Alberto P. Leite (Unicamp)

Maria do Carmo Gonçalves (Unicamp)

SYMPOSIUM I

MICROANALYSIS AND ANALYTICAL MICROSCOPY OF MATERIALS

Monday, October 11

16:20 Poster Session

Tuesday, October 12

Session Chair: Maria do Carmo Gonçalves (Unicamp/IQ)

8:30 Invited NEW APPLICATIONS OF ELECTRON MICROSCOPY TO NANOTECHNOLOGY RESEARCH
Miguel Jose Yacaman (I – I01)

9:10 TRANSMISSION ELECTRON MICROSCOPY OF POLYMER DERIVED CERAMIC IN THE Si-Al-O-N-C SYSTEM
R. M. Rocha, J. C. Bressiani, A. H. A. Bressiani (I - O05)

9:25 COMPOSITIONAL AND STRUCTURE ANALYSIS OF Pt/C AND Pt-Ru/C NANOCATALYSTS BY IN-SITU EDS AND HRTEM
E. Teixeira-Neto, William H. Lizcano-Valbuena, Ernesto R. Gonzalez (I - O03)

9:40 DIRECT OBSERVATION OF IONS, POLYMERS AND SURFACTANTS SORBED ON STÖBER SILICA NANOPARTICLES
C. A. R. Costa, C. A. P. Leite, F. Galembeck (I - O02)

9:55 MORPHOLOGICAL EVALUATION AND MINERAL PHASE IDENTIFICATION IN CALCINED PHOSPHATE SAMPLES
E. A. B. Francisco, S. L. de Jesus, L. I. Prochnow, M. C. M. de Toledo (I - O09)

10:10 COFFEE BREAK

Session Chair Fernando Galembeck (Unicamp/IQ)

10:30 Invited COMPOSITIONAL ANALYSIS OF INTERFACES AND NANOSTRUCTURES USING Z-CONTRAST IMAGING AND ELECTRON ENERGY-LOSS SPECTROSCOPY
Peter A. Crozier (I - I02)

11:10 SELECTING NEWLY NUCLEATED GRAINS FOR TEXTURE COMPONENTS DECOMPOSITION OF PARTIALLY RECRYSTALLIZED STEELS
E. G. de Souza, A. L. Pinto, C. S. C. Viana (I - O08)

11:25 TEM STUDY OF ION IRRADIATION SURFACE EFFECTS IN BETA PHASE Cu-Zn-Al SINGLE CRYSTALS
E. Zelaya, A. Tolley, A. M. Condo, F. C. Lovey, P. Fichtner (I – O10)

11:40 ULTRAFINE FERRITIC GRAIN OBTAINMENT THROUGH SUBCRITICAL TORSION OF A 0.16C STEEL
O. V. Silva Neto, O. Balancin (I - O07)

11:55 ELECTRON MICROSCOPY AND MICROANALYSIS APPLIED IN THE BENEFICIATION OF CELULIGNIN CATALYTIC FUEL
J. P. B. Machado, R. A. Conte, D. G. Pinatti, D. Rodrigues Junior (I - O04)

12:10 SUPERFICIAL POROSITY CLASSIFICATION BY IMAGE ANALYSIS USING OPTICAL AND SCANNING ELECTRON MICROSCOPY
M. A. Castro, A. N. Bonetti, A. M. Maliska, A. N. Diógenes, E. A. Hoff, C. P. Fernandes, H. C. Pavanatti (I - O01)

Wednesday, October 13

16:20 Poster Session

I – I01 NEW APPLICATIONS OF ELECTRON MICROSCOPY TO NANOTECHNOLGY RESEARCH

Miguel Jose Yacaman
Chemical Engineering Department and Texas Materials Institute University of Texas Austin

Electron Microscopy has been at the center of development of many areas of the science such as: Microbiology, materials science, etc. In the case of Nanotechnology, TEM has made mayor contributions. In fact Electron Microscopy have already made many of the most significant discoveries in the field. In this presentation we review the new advances in TEM such as spherical aberration correction, phase reconstruction, HAADF and several other that have resulted a quantum leap on the knowledge of the matter at the nanolevel .However the maximum impact is expected in the biological sciences. We present examples of ultrahigh resolution images of soft matter such as bacteria and virus.

I – I02 COMPOSITIONAL ANALYSIS OF INTERFACES AND NANOSTRUCTURES USING Z-CONTRAST IMAGING AND ELECTRON ENERGY-LOSS SPECTROSCOPY

Peter A. Crozier
Center for Solid State Science, Arizona State University, Tempe, AZ 87287-1704

Scanning transmission electron microscopy (STEM) offers a powerful combination of techniques that may permit structural and compositional information to be obtained with better than 0.2 nm spatial resolution in the current generation of commercially available instruments. For STEM imaging, the probe is focused and scanned over the sample in a raster pattern and an image is formed by measuring a signal arising from the beam-specimen interactions. The elastically scattered fast electron signals can be employed to give bright or dark-field images. High-angle annular dark-field imaging (so-called Z-contrast imaging) is a particularly powerful STEM technique for characterizing nanostructures. In Z-contrast imaging, the high-angle scattering is collected and used to form an image which has a strong dependence on atomic number. Alternatively, a spectroscopic signal can be measured and used to generate chemically specific information. The low-angle electron scattering can be used to form the electron energy-loss spectrum (EELS) making the combined use of Z-contrast imaging and EELS a powerful approach for extracting structural and compositional information at the atomic level. Here we use STEM to determine the compositional variations at the nanometer level in a variety of problems in interfaces, quantum dots and nanoparticles. We discuss factors which influence the accuracy of spectral quantification at nanometer resolution.

Oral Presentations

I – O01 SUPERFICIAL POROSITY CLASSIFICATION BY IMAGE ANALYSIS USING OPTICAL AND ELECTRONIC SCANNING MICROSCOPY

M. A. Castro, A. N. Bonetti, A. M. Maliska - Laboratório de Caracterização Microestrutural - Labmat,Bloco B - EMC, Campus Universitário, Trindade, Florianópolis, Brasil, Caixa Postal 476, 88040-900; A. N. Diógenes, E. A. Hoff – ESSS; C. P. Fernandes, H. C. Pavanatti- LMPT - UFSC

the microstructure characterization is one of the most important tools to study the materials properties. specially, for powder metallurgy, the superficial porosity displays an important role on the tribological behavior. therefore, it is necessary the use of an adequate methodology for its characterization. in order to measure the superficial porosity, unalloyed iron powder were compacted to 600 mpa and sintered in resistive furnace at 1150ºc during 1 hour. for the surface porosity determination, 40 images with 200x magnification were acquired by optical and scanning electron microscopy microscopy using the same region for both cases. in the optical method the images were acquired using bright –field ilumination whereas in the scanning electron microscopy it was used the back-scattering electron (bse) detector. the quantification analysis was carried out using the image analysis microstructural characterization software imago 2.1, released by esss – engineering simulation and scientific software. the superficial porosity was measured using a set of 10 specimens taking account 4 field for each one. it was observed that the superficial porosity measurements from the acquired images of the optical microscopy technique show a relevant difference when compared with that acquired by scanning electron ones.

I – O02 DIRECT OBSERVATION OF IONS, POLYMERS AND SURFACTANTS SORBED ON STÖBER SILICA NANOPARTICLES

C. A. R. Costa, C. A. P. Leite, F. Galembeck - UNICAMP – IQ, Cidade Universitária Zeferino Vaz, s/n, Campinas, SP, Brazil, Caixa Postal 6154, 13084-971

Ion, polymer and surfactant adsorption on silica particles is usually determined by a host of indirect methods (e.g. zeta potential, light scattering, IR, UV-vis and other spectroscopies) that do not inform on the adsorbate distribution throughout the particles. ESI-TEM examination of dry Stöber silica nanoparticles (? = 77 nm) previously exposed to aqueous solutions of NaCl, poly (isopropylacrylamide) (PNIPAM) and surfactants (anionic - SDS, cationic -CTAB and nonionionic - RENEX) reveals sorption patterns that were not previously evidenced by any other technique and were indeed unsuspected so far. In the case of NaCl, EELS spectra showed that Na+ and Cl- local concentrations absorbed from 1M NaCl within the particles are always close to stoichiometry with an excess 14% Na+ within the particle aggregates and 3% in the substrate surrounding the aggregates. Polymer and surfactant chains are detected adsorbed over the particles but not absorbed within them. On the other hand, the respective counter-ions (Na+, Br-) are detected within the particles, irrespective of the charge even though silica particles are negative, as evidenced by the -46 mV zeta potential in aqueous media. These results show that Stöber silica has interesting features that may lead to its use as a carrier of small molecules and ions due to the small particle size, high colloidal stability and the unique sorption behavior revealed by ESI-TEM.

I – O03 COMPOSITIONAL AND STRUCTURE ANALYSIS OF Pt/C AND Pt-Ru/C NANOCATALYSTS BY IN-SITU EDS AND HRTEM

E. Teixeira-Neto, W. H. Lizcano-Valbuena, E. R. Gonzalez - Instituto de Química de São Carlos, Avenida Trabalhador Sãocarlense, 400, Caixa Postal 780, CEP: 13560-970, São Carlos – SP

The development of new electrocatalysts is a very relevant topic, among the research areas on low temperature fuel cells. These materials consist of metal nanoparticles (2 - 5 nm), formed by Pt or Pt-M (M=Ru, Rh, Mo, Sn, W, Os) supported on high surface area carbon. The electrochemical performance of these catalysts is strongly dependent on the preparation method employed. In general, works associated to the preparation and characterization of electrocatalysts show information on lattice parameters and crystallite size effects using techniques such as X-ray diffraction (XRD) and transmission electron microscopy (TEM) to investigate the nanoparticle morphology and particle size distribution. However, the nanoparticles composition is only partially accessed, by XRD. The objectives of this work are the acquisition, by HRTEM and in-situ EDS, of the following information: i) crystalline planes or lattice parameters observed on nanoparticles surface, ii) shape and size distribution of the nanoparticles on carbon support, and iii) local and global composition for Pt and Pt-Ru/C catalysts with a very good dispersion of metal on carbon support prepared by a recently reported method. This information is correlated with the electrochemical performance of these materials in half-cell and in a single direct methanol fuel cell and may provide new information's for the understanding of the catalytic activity of the supported nanocatalysts.

Acknowledgements FAPESP, LME-LNLS.

I – O04 **ELECTRON MICROSCOPY AND MICROANALYSES APPLIED IN THE BENEFICIATION OF CELULIGNIN CATALITIC FUEL**

J. P. B. Machado, R. A. Conte, D. G. Pinatti, D. Rodrigues Junior - DEMAR-FAENQUIL, Polo Urbo-Industrial, Gleba AI-6, Lorena-SP, CP-116

The processing and/or integral recycling of the municipal solid residues is one of the urgent problems that demand solutions in the environmental and socio economic point of view. A processing methodology now in development in the DEMAR-FAENQUIL, and RM Materais Refratários Ltda. (Programa BEM), defines a manual separation of the solid residues to take advantage of the materials that are already recycled, proceeded by a pre-hydrolises of a mixture of wood and solid waste organic matter, obtaining celulignin and prehydrolysate. The main application of the celulignin is as fuel for ovens, boilers and gas turbines. The reduction of the ash content of the celulignin seeking its application as a clean fuel with high calorific power was made through controlled leaching and high gradient magnetic separation (HGMS). The leaching process was made by mixing celulignin with tap water or distilled water, in a suspension of 5 or 10 solid content %. Samples were taken from suspension in intervals of time and ash content were determined. For magnetic separation stage it was used a Boxmag Rapid and INBRÁS/ERIEZ magnetic separators. During all the processing, a LEO 1450 VP scanning electron microscope (SEM) with EDS coupled (spectrometer of dispersive energy) were used to qualify/quantify the inorganic and organic constituents. These analyses were essentials for the success of the beneficiation.

I - O05 TRANSMISSION ELECTRON MICROSCOPY OF POLYMER DERIVED CERAMIC IN THE Si-Al-O-N-C SYSTEM

R. M. Rocha - CTA-IAE, Pça. Marechal do Ar Eduardo Gomes, 50-S. José dos Campos-SP-12228-904; J.C. Bressiani, A.H.A. Bressiani - IPEN-CNEN/SP

Synthesis of Si-based ceramics via the polymer precursor routes has become of much interest in recent years due to their unique combination of low temperature processing, versatile shaping, microstructure and property tailoring capabilities. The ceramic material investigated in this work was processed by the active filler controlled pyrolysis using a polysilsesquioxane as a polymer precursor and silicon and aluminum as active fillers. A multiphase ceramic in the Si-Al-O-N-C system is obtained after pyrolisis at a 1500^{o}C/2h in nitrogen

atmosphere. Transmission electron microscopy (TEM) techniques (selected area diffraction, bright and dark field images) allowed the characterization of different phases formed upon pyrolysis: crystalline phases in the SiAlON's system; ?-SIC and AlN crystalline phases, SiOC amorphous phase yield from the polymer decomposition. The most observed phases in the SiAlON system were ?-SiAlON and SiAlON polytypoides (15R and 12H). Some minority phases as O'SiAlON, X-SiAlON and Al_4O_4C, which were not identified by X-ray powder diffraction (XRD), were observed and their structure investigated by electron diffraction.

I – O07 ULTRAFINE FERRITIC GRAIN OBTAINMENT THROUGH SUBCRITICAL TORSION OF A 0.16C STEEL

O. V. SILVA NETO, O. BALANCIN – DEMA/Ufscar, ROD.WASHINGTON LUIZ, KM 235, 13.565-905, SÃO CARLOS, SP, BRAZIL

This work presents a study of the ultrafine ferritic grain through thermomechanical treatment of a 0.16C steel in the subcritical range. By varying the subcritical tempering, the influence of cementite precipitation on the grain refinement was evaluated. All samples were austenitizing at 1100°C, quenched on water and tempered under 0.25, 0.5, 1, 12, 24 and 48 hours. The processing routes based on spheroidization tempering at 700°C (just below A_{e1}) followed the thermomechanical treatment through hot torsion. Before carrying out the torsion tests, the specimens were heated up to 700°C again, maintained by 10 minutes. After each a torsion test the samples were air cooled to environment temperature. The microstructural evolution was observed by optical and scanning electron microscopy. The high resolution electron backscattered diffraction (EBSD) has been used to analyse the boundary misorientations formed during deformation, determining both high and low angle boundaries. As a result after performing torsion tests, finer and dispersive carbides could be verified on samples submitted at lower tempering times. In addition, precipitate size caused little influence on the ferritic grain refinement. Therefore, the ultrafine grains were sufficiently reached with strains under the subcritical range. By this test procedure, it was possible to obtain ferritic grains smaller than 2 ?m for all tempering times.

I – O08 Selecting newly nucleated grains for texture components decomposition of partially recrystallized steels

E. G. de Souza, A. L. Pinto, C. S. C. Viana – IME, Pça Gen Tibúrcio, 80

a complete comprehension of the dominant mechanisms involved in recrystallization requires the knowledge of the orientation relationship between the deformed material and the newly nucleated grains. the software used together with ebsd accessory of the sem allows the manual selection of these new grains through the quality index (iq) maps – new grains without strain have high iq – thus generating an orientation distribution function (odf) of the selected grains. if there is a real concern about the odf's plotted statistical significance is also required. this leads to the need of multiple fields in each sample, but the iq will vary between scans collected with different conditions of sample preparation, filament life and column alignment. a method was developed in order to separate the newly nucleated grains. in each field, 36 points over new high iq grains were recorded and the mean iq was calculated together with the standard deviation. the 95% confidence interval regarded as new grains was considered to be two times the standard deviation as a normal distribution was taken into account to describe the variation of values. the orientations selected were summed over the entire set of fields belonging to the same sample and an odf of the new grains was calculated. this odf was subtracted from the one constructed with the role data set. this method was tested in order to study the partial recrystallization of extra low carbon steels with boron additions with excellent results.

I – O09 Morphological evaluation and mineral phase identification in calcined phosphate samples

E. A. B. Francisco, S. L. Jesus, L. I. Prochnow, M. C. M. Toledo - Esalq/USP, Av Pádua Dias, 11 (CP 9), CEP 13418-900 Piracicaba SP

Phosphorus plays an important role to plant growth once is associated with many metabolic functions. Traditionally, the use of high water solubility phosphate fertilisers has supported the increase of soil fertility resulting in crop production increment. However, the manufacture process of these fertilisers requires the utilisation of phosphate rocks with low quantity of metal impurities as iron and aluminium. This requirement

has forced industry to refuse large quantity of phosphate rock material, which is not used in the acidulation process to obtain high water soluble phosphate fertilisers. Aluminium phosphates, as crandallite, occur normally in weathering profile over primary materials rich in phosphorus as sedimentary rocks, igneous and metamorphic rocks. Samples were collected from three phosphate mines: Catalão-GO, Tapira-MG and Juquiá-SP. These samples were submitted to calcination to increase P solubility and were analysed using: (i) X-ray diffraction (XRD) to determine the minerals present; (ii) scanning electron microscopy (SEM) to determine morphology, clustering and intergranular pores; (iii) elemental composition analysis with coupled spectroscopy energy-dispersive (EDS); (iv) P total and soluble content. SEM and EDS made clear that the crandallite was close associated to the surfaces of iron minerals. Phosphorus solubility increased after the calcination process preliminary indicating to be a useful process to improve availability of P to plants.

I – O10 TEM STUDY OF ION IRRADIATION SURFACE EFFECTS IN BETA PHASE Cu-Zn-Al SINGLE CRYSTALS

E. Zelaya, A. Tolley, A. M. Condo - IB-CAB-CNEA-CONICET, CAB - Bustillo 9500, Rio Negro, Argentina, 8400; F. C. Lovey - IB-CAB-CNEA; P. Fitchner - Instituto de Física, Universidade Federal do Río Grande do Sul

The effects of irradiation with 170 keV Cu ions on L21 long range ordered b phase Cu-Zn-Al single crystals with surface orientation close to [001] were investigated. The resulting microstructure was studied with scanning electron microscopy, transmission electron microscopy and high resolution electron microscopy. Particles with a close packed structure were formed on the irradiated surface of the specimens. The size of the particles was in the range of 50 to 200 nm and their shape was approximately rectangular, with facets parallel to the [010] and [100] directions. The basal plane the particles was parallel to the (110) or (-110) matrix planes. High resolution images showed that the structure of these particles have predominantly an ...ABAB... stacking sequence with a high density of stacking faults. A low density of smaller, square-shaped particles, with facets parallel to the [110] and [-110] directions was also observed in the irradiated specimens. The observed effects are compared with surface phase transformations reported in these alloys.

Posters

I – P01 MICROSTRUCTURAL INVESTIGATION OF AN EB-PVD TBC

D. S. Almeida, C.R.M. Silva, C.A.A. Cairo - AMR/IAE/CTA, Divisão de Materiais, Praça Mal. Eduardo Gomes, 50, São José dos Campos, SP, Brasil, CEP:122228-904;

M.C.A.Nono - LAS-INPE.

Turbine blades of airplanes and thermoelectric plants work in adverse conditions, with corrosive environment and high temperature and pressure. One way to improve the life or the working temperature of the blades is by the use of special coatings over metallic material applied by Electron Beam - Physical Vapour Deposition (EB-PVD). The most usual material for this application is zirconia doped with yttria. Addition of niobia, as a new configuration in this system, can reduce the thermal conductivity and improve mechanical properties of the coating. The purpose of this work is to show the technique for production of such coatings and the results of the addition of niobia taking in to consideration X-ray diffraction and scanning electron microscopy observations. Results show a columnar structure with only tetragonal phase in the ceramic coating in the chemical composition range studied.

I – P02 PROPERTIES OF PIASSAVA WASTES COMPOSITES WITH POLYMERIC MATRIX

R. C. M. P. Aquino – CEFET, Núcleo de Pesquisas Avançadas na Construção Civil, Rua Dr. Siqueira, 273, Campos dos Goitacazes, Rio de Janeiro, Brasil; J. R. M. d'Almeida - PUC – RJ; S. N. Monteiro – UENF.

In the past years, scientific and technological researches have concentrated efforts in searching for materials that could present more diversified and improved properties as compared to those of conventional materials. The current tendency is to look for new materials, which are compatible to the environment without negative effects. The development of natural fiber reinforced composites with polymeric matrix is an example of an action to introduce a special material that is, at least for the fiber, renewable, low cost, biodegradable and non toxic. In the present paper, therefore, the physical-chemistry properties of the piassava (Attalea funifera Mart) fiber were evaluated. Polyester resin was used as matrix and specimens were made with piassava fiber with volume fraction varying from 10 to 40%. The mechanical properties of the composites were evaluated by bend tests. After the specimens were broken, the microstructure and the phase bonding in the region of fracture were characterized by scanning electron microscopy, SEM. The results have shown that this piassava fiber reinforced polymeric composites present a potential for competition with other wood based industrialized products.

I – P03 RELATIONSHIP BETWEEN CHEMICAL PROPERTIES AND SURFACE MORPHOLOGY OF PIII HMDSO-Ar THIN FILMS

J. R. R. Bortoleto, D.C. R. Santos, E. C. Rangel, N. C. Cruz, R. P. Mota, R. Y. Honda – UNESP - Unidade Diferenciada Sorocaba/Iperó, Av. Três de Março 511, Sorocaba-SP, Brasil, 18087-180.

Plasma immersion ion implantation PIII has been employed to modify surface properties of different

materials as metals, ceramics, semiconductors and polymers. It is an effective low cost technique which emerged due to limitations presented by the conventional beam bombardment. The target is placed in contact with the plasma and biased with high voltage negative pulses. In this work, thin polymer films were prepared from radiofrequency (13.56 MHz) hexametildisiloxane, HMDSO, plasmas. Ion bombardment was performed in the same equipment, using argon (13.56 MHz, 70 W, 5.3 Pa) PIII. Substrate holder was biased with 10 kV/100 Hz tooth-saw-like pulses. The treatment time, t, was varied from 0 to 7200 s. It was investigated the influence of t on the surface properties of the samples. Surface morphology was characterized by atomic force microscopy, while the molecular structure was probed by infrared spectroscopy. Film wettability was determined through contact angle measurements. It was observed the presence of absorption bands related to C-H, Si-O-Si and Si-O-C stretching modes. The proportion of C-H bonds decreased with t, indicating H emission. Besides, film surface becomes smoother with increasing treatment time. Through such results it was possible to evaluate the influence of roughness on the film wettability. Interpretation is proposed in terms of the energy delivered to the sample by ion implantation.

I – P04 A NEW AND EFFECTIVE PROCEDURE FOR POLYMER NANOCOMPOSITE FABRICATION

F. C. Bragança, L. F. Valadares, M. M. Murakami, C. A. P. Leite, F. Galembeck - IQ-Unicamp, Instituto de Química, Unicamp, Caixa Postal 6154, Campinas, SP, CEP 13083-970

Polymer nanocomposite materials are finding a host of new applications due to their extraordinary properties, that are observed e.g. when layered silicates are dispersed as 1-nm thin leafs within a plastic or rubber. Usual procedures require a previous chemical modification of the clay or chemical polymer synthesis in the presence or the dispersed clay that makes them rather expensive. A new procedure was developed in this laboratory (PI 0301193-3), based on the large and well-known clay exfoliating ability of water, as observed e.g. on montmorillonite. This method consists on mixing the latex and clay under conditions such that the clay is fully exfoliated, drying and processing taking care to prevent clay reaggregation. This procedure has already been successfully applied to many thermoplastics and rubbers. In the case of polymer lattices, nanocomposites were obtained with remarkable mechanical properties and, in some cases, excellent resistance to solvents. XRD and TEM examination of the nanocomposites showed that the clay particles are indeed exfoliated or intercalated with polymer. Transmission micrographs also show that capillary adhesion between clay leaves and latex particles is sufficiently large to ensure particle deformation upon drying and to provoke strong adhesion between rubber and clay nanosheets. In the case of polyethylene the method has not yet being applied successfully.

I –P05 MICROESTRUCTURAL ANALYZE OF CHEMICAL Ni COATING OVER ALUMINA

O. R. Bagnato, F. R. Francisco – LNLS, Laboratório Nacional de Luz Sincrotron, 10000, Guará, Campinas, SP, Caixa Postal 6192, CEP13 083 970; A. Felder, M. A. Gomila - UPC

The aim of this study is to verify the deposition aspects and chemical composition of NiP coating, chemically deposited over an alumina substrate, for metal / ceramic brazings. High alumina samples were prepared using a chemical NiP bath. Two class of samples were prepared: one “as prepared” and a second submitted a several heat treatment cycles in a moderated temperature. The second-class samples were submitted to 40 cycles of heat treatment during 2 hours at 250 oC in oxidizing atmosphere. All the samples were tested in four points bending machining, INSTRON 8511 and the fracture surface, transverse section and Ni coating were observed in a JEOL SEM. The study of Ni coating showed that the coating is not continuous, but it was consisted by small particles with diameter around 250 nm. The EDS chemical analyze did not indicate the presence of oxides or reaction products in the Ni – alumina, Ni - filler metal interfaces or in Ni particle. It was observed that the Ni particles are present in the entire surface and in the internal surface of the ceramics porous too. The heat-treated samples did not present changes in the particle sizes and shape, the chemical composition and interface weren’t changed too. In conclusion, the coating produced by NiP bath is not continuous, but cover a large area in the ceramic samples; their chemical composition was not affected by heat treatments in intermediate temperature.

I – P06 STUDY BY SEM AND EDS OF CALCIUM PHOSPHATE MONOLITHS OBTAINED BY HYDROTHERMAL METHOD

O. Bermúdez-Castillo - UNICAUCA, Calle 5 No 4-70 Popayán (Cauca), Colombia; C. V. Leal, C.A.C.

Zavaglia – UNICAMP; E. Delgado-Mejía - UNAL

Calcium phosphate cements (CPCs) are promising bioceramic materials. When mixed with water or an aqueous solution a CPC hardens (sets) to a rigid body composed of at least one calcium phosphate. CPCs based on B-tricalcium phosphate (B-TCP) are simple and inexpensive, but their end setting reaction product usually is dicalcium phosphate dihydrate (DCPD or brushite), which is acid and can cause tissue necrosis when implanted. On the contrary hydroxyapatite (HA) and octacalcium phosphate (OCP) are basic and biocompatible. One way to overcome the above mentioned shortcoming of brushite cements would be to convert the resulting DCPD into HA and (or) OCP, which can be done by hydrolysis in alkaline solutions. In this work monoliths were obtained by hydrolysis of two brushite cements and the conversion into HA and OCP was ascertained by means of EDS. The results were correlated with SEM and X-ray Diffraction. Two types of cements were studied: CPC 1 and CPC 2. The powder for CPC1 consisted in B-TCP prepared by heating calcium-deficient HA. For CPC2 the powder was a mixture of B-TCP prepared from eggshell and DCPA. For both cements the liquid was an aqueous solution of phosphoric acid. Set and dry cylindrical bodies of both cements were immersed for hydrolysis in NaOH solution at 60°C, and then washed, dried, and analyzed by SEM, EDS and XRD. Results: DCPD was transformed into OCP and HA as a consequence of hydrolysis. EDS proved convenient for the study of this process.

I – P07 FRACTAL DIMENSION AS A TECHNIQUE FOR PIT SHAPE EVALUATION IN ALUMINUM ALLOYS

W. R. C. Campos C. F. C. Neves, M. M. A. M. Schvartzman, N. N. Atanazio Filho - CDTN/CNEN, Serviço de Integridade Estrutural, Rua Prof. Mario Werneck, s/nº, Campus UFMG, Pampulha, Belo Horizonte, Brasil, Caixa Postal 941, CEP 30.123-970

Aluminum alloys have been used as cladding materials for nuclear fuel in research reactors. Aluminum owes its good corrosion resistance in most environments to the protective barrier oxide film formed and strongly bonded to its surface. When this film is damaged under conditions that normal self-healing does not occur, localized corrosion in the form of pitting or intergranular attack can occur. For aluminum-clad spent fuel, penetration of the clad results in corrosion of uranium or uranium-aluminum alloy fuels and release of fission products to the reactor pool water. In order to develop a fundamental understanding of the corrosion problems with aluminum alloys in TRIGA IPR-R1 reactor, located at CDTN/CNEN in Belo Horizonte, a monitoring program has been initiated. The program consists of immersion of corrosion surveillance coupons made of three different aluminum alloys 1050, 5052 and 6061 in the reactor pool water. After a large period of exposure the coupons were withdrawn and examined for pitting evaluation. It was noted by macrographic analysis that different aluminum alloys showed different pit sizes and shapes. It was carried out surface micrographic analysis of the different aluminum alloys and the pits shapes were evaluated and compared using fractal concept and digital image processing techniques. This paper describes the methodology used in image capture and the results of the pits evaluation.

I –P08 STEAM CURING AND DELAYED ETTRINGITE FORMATION IN BRAZILIAN CEMENTS

G. Camarini – Unicamp, Rua Francisco José Soares, 299 Limeira - SP, CEP 13480-737

Delayed ettringite formation may be defined as the formation of ettringite in a cementitious material by a process that begins after its hardening. The reaction occurs between the anhydrous cement compound C3A and sulphates in the paste. The ettringite formation can occur in fresh and in hardened concrete. Its

formation in a plastic fresh mixture does not produce any damaging expansion. But if it occurs in a hardened cementicious material it causes great expansion. This is a process which none of the sulphate comes from outside the cement paste. Delayed ettringite formation (DEF) can damage concrete that has experienced a temperature above about 70ºC by promoting its expansion, but the use of a cement with blastfurnace slag in its composition can diminish this formation because there is less clinker avaiable to react and form the expansive compound. The aim of this work was to observe the DEF in steam-cured concretes made with two Brazilian Portland cements (Blastfurnace Slag Cement – CP III, and High Initial Compressive Strength Portland Cement – CPV-ARI). The steam temperature was 80ºC. The image analysis showed that DEF was observed in both cements, and this can become a problem in precast concrete industries.

I – P09 MICROSTRUCTURAL EVOLUTION AND PRECIPITATES DISSOLUTION WITH THE REHEATING TEMPERATURE OF A Nb- AND N-BEARING AUSTENITIC STAINLESS STEEL BIOMATERIAL

E. J. Giordani, O. Balancin - DEMa/UFSCar, Rodovia Washington Luís (SP-310), Km 235, São Carlos - SP - Brasil, CEP: 13565-905

The addition of small quantities of strong carbides-, nitrides- and carbonitrides-forming elements, such as titanium, vanadium and mainly niobium, is a well known and very important practice in order to optimize the mechanical properties of low carbon steels. An improved combination of high yield strength and good toughness is attained primarily through the grain refinement favored by the desired precipitation during the thermomechanical control processing (TMCP) of the steel. Reheating, usually ranging from 1050ºC to 1300ºC, constitutes the first controlled stage of every regular commercial TMCP. The reheating temperature not only controls the evolution of grain size, but also the amount of microalloying elements into solution in the austenite prior to processing. To ensure an appropriate austenitic microstructure - uniform as-reheated grains and fine grains after the TMCP - it is necessary to define accurately the solubility product of the precipitates present. In this way, this work aims to characterize, through the extraction of precipitates techniques by electrolytic dissolution associated with SEM/EDS and X-ray analyses, the type and the amount of precipitates into solution with the reheating temperature (ranging from 1050ºC to 1300ºC) of a niobium- and nitrogen-bearing austenitic stainless steel biomaterial - ISO 5832-9 (ASTM 1586).

I – P10 MICROSTRUCTURAL EVOLUTION DURING PROCESSING AND HEAT TREATMENT OF MARTENSITIC STAINLESS STEEL FOR PROFESSIONAL CUTLERY

C. M. Galinari – CDTN,Serviço de Materiais e Combustível Nuclear, Rua Prof. Mário Werneck, s/n., Campus UFMG, Pampulha, Belo Horizonte - MG, Caixa Postal 941, CEP 30123-970; P. S. S. Bálsamo – ACESITA; W. L. Vasconcelos - UFMG

The martensitic stainless steels show an attractive combination of good corrosion resistance and high mechanical strength. They are suitable for applications requiring high wear resistance in aggressive environments, such as professional cutlery. The final microstructure of those steels is normally composed of a martensitic matrix with the presence of carbides. These carbides should be controlled, because they may improve or worsen the mechanical performance of the steels. Martensitic stainless steels can be hardened by quenching and are submitted to microstructure changes during industrial processing and heat treatments, which are made after product conformation. The microstrutuctural evolution was analyzed from the initial solidification to the final stage of heat treatment using scanning electron microscope (SEM) and energy dispersive spectrometer (EDS). The formation of primary carbides from the liquid segregation in the initial solidification and the presence of primary and secondary carbides in the other steps were observed. The carbides are M23C6 kind, where the metallic part (M) contains chromium, iron and molybdenum, showing different sizes and proportions in each step.

I – P11 AFM CHARACTERIZATION OF BARIUM TITANATE

S. M. Gheno, H. L. Hasegawa, P. I. Paulin Filho - DEMA - UFSCar - Rod. Washington Luiz, Km 235, CP 676, CEP: 13565-905, São Carlos-SP-Brasil, fone:(16)3351-8255; fax: (16)3351-1404

Barium titanate (BT) and derived material are extensively used in the manufacture of a large sort of electronic components like a multilayer ceramic capacitor (MLCC), semi-conductor devices, PTCR

(positive temperature coefficient resistor) and electro-optics devices. PTC effect is characterized by a jump of some orders of magnitude in electrical resistivity, starting from low resistivity values to high values, in a strict temperature range, which is attributed to the presence of potential barriers in the area of grain boundaries. The electric properties of PTC ceramic are sensitive to microstructure and defects, in atomic scale, that are significantly affected by processing parameters, such as chemical composition, sintering conditions and cooling. Barium titanate ($BaTiO_3$) doped with La and Mn is the principal constituent of PTC ceramic materials. Abnormal grain growth in barium titanate microstructure has been reported in latest years by the scientific community and industries of electronic devices. The microstructure of barium titanate were investigated using topographic images obtained by atomic force microscopy (NanoScope IIIa of Digital Instruments), operating in contact mode. The barium titanate used in this work was obtained after compacting barium titanate powders in a uniaxial press and conformed in pellets form, followed by sintering at 1350^{o}C for one hour. Pellets was polished and grains revealed by thermal etching.

I – P12 CHARACTERIZATION OF THE NANOMORPHOLOGY OF POLYMER-SILICA COLLOIDAL NANOCOMPOSITES USING ELECTRON SPECTROSCOPY IMAGING

F. Galembeck - Unicamp-Instituto de Química, CP 6154, CEP13084-971; J.I. Amalvy, M. J. Percy, S. P. Armes – Sussex; C.A.P. Leite - IFGW-Unicamp

There is currently a great interest in the synthesis of polymer composite particles due to the unprecedented properties displayed by the materials built with these nanoparticles. Silica-polymer particles prepared with different polymers present different complex morphologies that are revealed by combining ESI-TEM, EFTEM and other imaging techniques. Styrene and vinylpyridine were polymerized in aqueous emulsion in the presence of silica nanoparticles (13 and 22 nm). The polystyrene (PS) composite particles are rigid and uncoalesced, with a core-and-shell morphology and the 13 nm silica nanoparticles are strongly clustered at the composite shell, which is still covered by a thin polymer film. PS-22 nm silica composite particles are similar but the organic outermost coating is sufficiently thick to produce a plastic layer that covers particle aggregates. The polyvinylpyridine (PVPy)-silica particles have a completely different morphology of the \"currant bun\" type, with the silica particles evenly dispersed throughout the polymer. These results can be understood considering the relevant interfacial tensions and their changes during particle formation and drying: during the emulsion polymerization, the more wettable silica accumulates at the (poorly wettable) PS particle shell, while silica and PVPy both have similar interfacial tensions with water. In the dry samples, low-MW PS fractions migrate to the particle outer shell, due to their lower surface tension.

I – P13 ANALYSIS OF THE ASPHALT-RUBBER PROPERTIES FOR THE TECHNIQUES SEM AND RTFOT

B. C. Karina, A. B. S. Ariosvaldo, D. L. C. Michely – UFCG, Rua Aprígio Veloso, 885, Bodocongó. Campina Grande-PB; A. R. B. Marcos, A. P. Luiz Renato – UFPB; C. A. C. Regina, G. O. Márcia - INT

The use of modified asphalt through of the incorporation of crumb tire rubber is an excellent alternative to improve the rheological properties of the conventional asphalt. Therefore, it offers resistance cracking, improved aging, oxidation resistance and improved resistance to fatigue. The use of asphalt-rubber technology, known as ecological asphalt, offers a solution to the environmental problem too. The goal of this work was to produce modified asphalt using CAP 20 as asphalt, sulfur as activator and tire rubber as adducting. The obtained product was analyzed through the scanning electronic microscope (SEM) and of Rolling Thin Film Oven Test (RTFOT). It was observed a good homogeneity of the material and insignificant mass loss. This can be due to a good chemical interaction between the molecules of the asphalt and the rubber tire. Thereby, the technology asphalt-rubber is very important from an environmental and economic standpoint.

I – P14 MICROANALYSIS STUDY OF STYRENE-BUTYL ACRYLATE-ACRYLIC ACID NANOLATEX.

M. M. MURAKAMI, F. GALEMBECK - IQ – UNICAMP, BLOCO D-208, CAIXA POSTAL: 6154, CAMPINAS, SÃO PAULO, BRAZIL, CEP:13084-971; C. A. P. LEITE - IFGW – UNICAMP; E. PONZETTO - OXITENO S.A.

The application of transmission microscope using electron energy loss spectroscopy imaging (ESI-TEM)

techniques is revealing new and complex microchemical patterns in nanolatex particles. Low-Tg styrene-butyl acrylate-acrylic acid terpolymer latexes were prepared by pre-emulsion method, using different non-ionic (ethoxylated) surfactants. The effective diameters of latex particles prepared with nonylphenol EO40, 50,100 are respectively 58.6, 54.6 and 65.8 nm, as determined using photon correlation spectroscopy (PCS). ESI-TEM imaging reveals the distribution of C, S, O and Na constituents in the particles of different sizes and the inhomogeneity of the elemental distribution, within particles and among them. C is rather evenly distributed throughout the particles as well as O and S, following a pattern that has been previously observed in larger latex particles. Non-particulate latex components from serum solutes also contribute to the images: in some cases (e.g. the latex prepared using EO40 surfactant) sulfur compounds are abundant in the serum. Strong accumulation of S and O together with C depletion is observed in a bimodal latex, evidencing chemically differentiated domains within particles. Many current descriptions of dry latex particles show an ionic double layer at the particle surface, enclosing a hydrophobic bulk. An outer thin shell with accumulated ions is actually seen in some cases, but sulfate as well as sodium ions are largely trapped within the polymer bulk.

I – P15 INFLUENCE OF THE REACTION ATMOSPHERE ON THE MORPHOLOGY OF SIC PRODUCED BY MICROWAVE-ASSISTED CARBOTHERMAL REDUCTION OF RICE HULLS

V.T. Pierri, T.P. Deksnys, W.L. Idalgo, R.R. Menezes, E. Fagury-Neto, R.H.G.A. Kiminami – UFSCar, Rod. Washington Luis km 235, CP 676, CEP 13595-950, S. Carlos - SP, Brazil

Microwaves have been employed successfully to produce a wide variety of ceramic materials such as TiC, Al2O3/SiC, Si3N4 and SiC. Rice hulls, as a potential SiO2 source, can be used to produce SiC whiskers and particles by microwave-assisted carbothermal reduction reaction (MWCR). However, this reaction must be carried out under a reducing atmosphere in order to ensure the formation of SiC and to prevent carbon oxidation. Thus, the aim of this work was to evaluate the influence of the reaction atmosphere on the morphology of SiC and identify its role in the reaction kinetics. Rice hulls were previously pyrolized to release SiO2 and then blended with carbon black in a proportion of 1:3, yielding the precursor blend for MWCR. The reactions were performed in a semi-industrial microwave oven, in which a pipe fluidized-bed reactor was assembled to allow the insertion of reaction gases (argon and nitrogen). The gas flow rate was controlled and the time of the reactions was set for 30 minutes. X-ray results indicated that SiC phase was produced in both the atmosphere gases. The reaction performed under the N2 flow yielded b and a phases of SiC, whereas the Ar atmosphere produced only the a phase, indicating an improvement of reaction kinetics when the reaction gas was N2. The morphology of the powder from MWCR using N2 consisted of numerous spherical SiC nanoparticles and less SiC whisker clusters in comparison to the reaction with the Ar flow.

I – P16 ABNORMALGRAIN GROWTH IN BARIUM TITANATE DOPED WITH YTTRIUM

P. I. Paulin, H.L. Hasegawa, M. R. Morelli - DEMa/UFSCar, Rodovia Washington Luis km 235, São Carlos, SP, Brazil, caixa postal 676, CEP 13560-905

Barium titanate is widely used as a material for dielectric, pyroelectric and piezoelectric applications. The understanding of microstructural characteristics of barium titanate is important to increase and optimize their properties. A significant amount of work has been performed show the effect of dopant levels on coarse microstructure of BaTiO3. This work aimed to investigate the microstructural changes, mainly abnormal grain growth (sometimes referred to as secondary recrystalization), during the sintering of doped BaTiO3 with yttrium. Barium titanate powder (TAM ceramics, Niagara Falls NY) with average particle size of 0.9 ?m and [Ba]/[Ti] ratio = 0.998 was doped with 0.15% mol of yttrium (99,8%, AESAR) shows abnormal grains with sizes around 25 ?m on a fine matrix (grain size between 3 ?m and 5 ?m) as observed by Scanning Electron Microscopy (SEM), after sintering. The conditions to obtaining such large grains in BaTiO3 were chosen to induce and promote the nucleation of abnormal grains, by controlling the [Ba+Y]/[Ti] ratio (0.9995) and sintering conditions.

I –P17 ANALYSIS OF SCALES FORMED DURING HIGH-TEMPERATURE SULPHIDATION OF Fe-Nb ALLOYS AT 800 DEGREE CELSIUS

M. Castro-Rebello, T. Kunioshi, J. A. S. Tenório, S. Wolynec - PMT/EPUSP, Rua São Benedito, 146 / CEP 06700-296 / Cotia - SP

Two Fe-Nb alloys, containing about 15 and 30wt.%Nb (Fe-15Nb and Fe-30Nb, respectively), were prepared by repeatedly arc-melting appropriate amounts of the two pure metals (99.98%Fe and 99.8%Nb) on a water-cooled copper hearth under high-purity Ti-gettered argon. After annealing heat treatment the ingots were cut into slices (1,0 mm thick and with a surface area of around 2,0 cm2)and samples were ground down to 600-grit paper. Both alloys are two-phase: iron-rich and Fe2Nb laves phase. The isothermal sulphidation tests were carried out at 800C for periods of 18 hours, at a thermobalance. Sulphidized samples were examined by means of X-ray diffraction (XRD) for phase identification. Subsequently they were mounted in epoxy resin for examination at scanning electron microscope (SEM) and microanalysis with the energy dispersive spectrometer (EDS) X-ray attachment to the SEM, in order to identify the phases and to determine the element distribution in oxide scales. Both alloys showed linear sulphidation behaviour. The sulphidation of Fe-15Nb and Fe-30Nb alloys produced similar morphologies characterized by thick and porous iron sulphide external scale and an innermost heterophasic region (HR) where both phases and elements were attacked. Elemental analysis were concentrated in this HR in an attempt to understand the complex mechanism of sulphidation.

I – P18 MICROANALYSIS OF POLYCAPROLACTONE/CHLORINATED POLYETHYLENE (PCL/PECL) BLENDS

T. S. Plivelic - LNLS, Laboratório Nacional de Luz Síncrotron, Rua Giuseppe Máximo Scolfaro, 10000, Campinas, SP, Brazil, Caixa Postal 6192 - CEP 13084-971; C.A. Leite, I.L. Torriani - IFGW-UNICAMP; M.C. Gonçalves - IQ-UNICAMP

Polymer blends containing crystallizable components present a great variety of morphologies. The prediction and control of these morphologies as well as the understanding of the crystallization behavior during its processing will enable new materials to be developed. In this work a morphological characterization in the micron/nanometric scale of the polycaprolactone/chlorinated polyethylene (PCL/PECl) miscible blend is presented. The structural features of the crystalline regions (lamellae, lamellar bundles and spherulites) and the elemental composition of the amorphous regions were analyzed by TEM and EELS as a function of the PECl content for isothermally treated samples. For the PCL homopolymer and 80/20 and 60/40 blends a spherulitic volume filling structure were observed. When PECl was the major component, segregated amorphous regions were observed between the spherulites. These regions were rich in Cl as showed from EELS results. Typical Maltese Cross pattern observed by POM in PCL becomes more irregular and coarser when PECl is added in the blend. This coarseness was caused by the exclusion of uncrystallizable material into the interlamellar bundles as showed in TEM images. Experiments made with different compositions and thermal treatments showed that PECl caused a reduction of the growth rate of the spherulites and an increase in the nucleation rate when this component was gradually added into the blend.

I – P19 Morphology and thickness analysis of copper electrocoatings on titanium wire using scanning electron microscopy

J. L. Rosa, M. P. Peres, R. B. Ribeiro - Unesp-FEG, Rua Ten. Manuel Barbosa, 533 - V. Zélia / Lorena-SP/Brazil; A. L. M. Robin - Faenquil

Copper electrocoatings on titanium have two objectives: acting as lubricant during drawing of titanium

wire in order to improve the surface quality of the wire; providing electric properties to the coated titanium wire for application as mechanical reinforcement in superconductor magnets. Titanium is difficult to be electroplated in aqueous media due to the spontaneous formation of a passive film, that reduces the adhesion force of the coatings. In this work, the influence of the main electrodeposition variables, surface preparation of titanium, reagents concentration, cathodic current density, electric charge and stirring, on the adhesion of the copper coatings was studied in laboratory scale using the Taguchi L16 orthogonal array with the factors at two levels each (low/high). The best conditions of adjustment were applied for electrodeposition in pilot scale. The morphology and thickness of the copper layer were evaluated by optical and scanning electron microscopy in different regions of the coated wire. Variations in the topography and thickness of the copper deposit were observed. The measurement of titanium potential versus saturated calomel electrode potential (reference) at different regions of the wire during polarization showed that the current was not homogeneously distributed along the wire. The more polarized areas (near electrical contacts) presented a thicker and dendritic deposit, whereas in the less polarized areas, the coating was thinner and smoother.

I – P20 CAST Ti-Al-Nb ALLOYS PREPARED BY ARGON ARC MELTING FOR BIOMATERIALS APPLICATIONS: OBTENTION AND METALLOGRAPHIC CHARACTERIZATION

L. ROCHA-SOUSA, C. R. MEIRA, A. C. GUASTALDI - DFQ/IQ/UNESP/ARARAQUARA, GRUPO DE BIOMATERIAIS, RUA PROF. FRANCISCO DEGNI, S/N, SÃO PAULO, BRASIL; S. G. SCHNEIDER - DEMAR/FAENQUIL; E. PELAEZ-ABELLÁN - F. QUIMICA/U. HABANA/CUBA

The biomaterials represent one of the more and sophisticated trends in the worldwide medical practice. The cpTi and some of its alloys are considered very important in medical field because of its excellent biocompatibility and mechanical properties. In the last decades, new Ti alloy compositions, specifically tailored for biomedical applications, have been developed. The Ti-6Al-4V alloy with similar properties to the Ti-6Al-7Nb was developed in response to concerns relating V to potential citotoxicity and adverse reaction with body tissues. In the first time Ti alloys were produced by conventional methods like vacuum arc refusion (VAR). In the present study the Ti-Al-Nb alloys were prepared with Al and Nb contents ranging from 0 to 10 wt% by alternative method of argon arc melting and have been characterized its morphology and crystallographic structure by light and scanning electron microscopy. The results showed that the as-cast alloys contents 10Al and 8Al-2Nb wt% exhibited a typical rapidly-cooled metastable feather-like microstructure. When the content were 6Al-4Nb, 4Al-6Nb and 6Al-7Nb wt% the acicular shape of the alpha phase was present in an arrangement known as basket-weave which characterizes the Widmanstätten structure. The increase in the Nb content produced a higher volume fraction of the beta phase, as well as the fact that a more refined structure of alpha-phase was obtained. When the content were 2Al-8Nb and 10Nb wt% the acicular martensite alfa was observed.

I – P21 MORPHOLOGICAL ANALYSIS OF PITTING IN STAINLESS STEEL ABNT 310S AFTER CORROSION TESTING

R. B. Ribeiro, L. R. O. Hein, E. N. Codaro - FEG-UNESP, Rua Jovino Luiz dos Santos, 451, Lorena, BRAZIL, CEP 12604-370

The stainless steels are special alloys with a great versatility in terms of application due to the high

corrosion, oxidation and mechanical resistance, as for instance, the austenitic Stainless Steel 310S. This paper had as specific objective to study the temporary evolution that govern the growth of pits in the austenitic Stainless Steel 310S as received, and after being submitted in different times of exposition (48, 120, 168, 216 and 312 hours) in salt spray. The experimental methodology for the superficial characterization and for the pits profiles were accomplished in agreement with the following steps: materiallographic preparation, optical microscopy using brightfield illumination, image processing and scanning electron microscopy. The behavior of the morphological distribution and pits evolution presented the following results: hemispherical shape > A transition region > B transition region > irregular shape > conic shape. Hemispherical morphology and A transition region pits are present in larger quantity, but cylindrical pits have not been found in such a system. One can conclude that pits on Stainless Steel 310S as received, has a tendency of beeing wider than depth and grow preferentially in the width. It is evidenced that as larger the time of exhibition in salt spray, larger the pits width.

I –P22 SUPERCRITICAL INFUSION OF A SILVER-CONTAINING COMPOUND INTO POLYMERS FOR THE PURPOSE OF MAKING REFLECTIVE/CONDUCTIVE FILMS

A.F. Rubira, M. H. Kunita , E. Radovanovic, E. C. Muniz - Departamento de Química, Grupo de Materiais Poliméricos e Compósitos, UEM, Av Colombo 5790, 87020-900, Maringá-PR; N. Nazem, L.T.Taylor - Virginia Tech-VA-USA

Commercially available films of poly(ethylene terephthalate)(PET)and polysulfonehave been infused with (1, 5-cyclooctadiene-1,1,1,5,5,5-hexafluoroacetylacetonato) silver(I) dimer, [Ag-(COD)HFA](2) with supercritical carbon dioxide. Thermal cure at approximately 200 C results in reduction to silver metal and silver mirror formation on polymer surfaces. Reflectivity or conductivity of the polymer surface was dependent both on the CO2 infusion and cure conditions. Silver particles less than 10 nm appear in the film bulk near the surface. Different sized silver clusters can be grown by controlling the infusion/cure conditions. The surface silver strongly adheres to each polymer surface. As we have seen the transition from an electrically insulating to conducting state can be affected by changing the size of the atomic silver cluster. The main focus of this work was on the construction of silver cluster on polymer film but the work also has great applications as electrodes in biomaterials and in the fabrication of nanostructured materials.

I – P23 MORPHOLOGICAL ANALYSIS OF RECOVERED CONNECTORS FOR TELECOMMUNICATION

G. L. J. P Silva, H. J. Izário, M. L. C. P. Silva, R. B. Ribeiro, J. L. Rosa – FAENQUIL, Rodovia Itajubá Lorena km 74,5 LorenaS.P.12600-970 Cx.P.116.

One of the configurations of metallic connectors used in telecommunications is the terminal block configuration, the connection system of which is commutable and the connections established by electrical contacts normally closed. These materials may be used in external networks and make possible to use this electrical protection device in parallel/serial or with ground wire elements. These metallic blocks are manufactured in bronze or brass, and their surface is covered with a thin layer of metallic tin[1], with a thickness between 10 and 12 inches. Trace analysis of the contaminants presents into the metallic layer, based in the controlled dissolution of protective surface material without dissolution of the base materials. The optical microscopy before previous preparation of samples was performed and was an useful procedure to determine the deep of the recover avoiding the dissolution of the base materials. Ni and Zn levels in the removed samples was determined by AAS-graphite furnace. Typical values of 0.037 and 0.45 % (w/w)[2] was respectively obtained. Composition of the brass or bronze was determined too using an air acetylene flame and the main components hollow cathode lamps.

REFERENCES

1- ASTM B 539-80, “Standard Methods for measuring Contact of Electrical Connections Static Contacts”, SDT 235-430-723,”Requisitos para bloco terminal de rede externa”

2- Izário Filho, H.J.; Silva, G.L. J.P. da ; Silva, M.L.C.P. da; Ribeiro, R.B. ; Rodrigues, J. C. G. and Bombard, A .J. F., D

I – P24 MICROSTRUCTURAL CHARACTERIZATION OF HEAT-TREATED Ti-Zr-Si-B ALLOYS

G. Silva, A. S. Ramos - UNIVAP/IP&D, Avenida Shishima Hifumi, 2911, Urbanova; E. C. T. Ramos, G. Rodrigues, C. A. Nunes, FAENQUIL/DEMAR

Titanium silicides present in particular elevated oxidation resistance in high temperatures, and the addition of zirconium has been suggested to improve this one. In Ti-Si-B alloys, the ternary Ti6Si2B phase is

formed during solidification and after heat treatment at 1250oC. Two-phase Ti+Ti6Si2B alloys presented higher oxidation resistance than the eutectic Ti-13.5Si alloy. However, recent studies have shown that the ternary phase was not formed in these as-cast Ti-Si-B alloys with zirconium added (7 at-%). The present work reports on the microstructural characterization of heat-treated Ti-Si-B alloys containing zirconium added (7at-%). High-purity starting materials were used in these experiments: Ti (min 99.7wt-%), Zr (min 95 wt-% with 4.5 wt-%Hf), Si (99.999 wt-%), and B (99.5 wt-%). The arc melting process was carried out in a furnace under high-purity argon atmosphere using a water-cooled copper hearth, non-consumable tungsten electrode, and gettered by titanium. The heat treatment at 1200oC for 90h was performed to obtain the equilibrium microstructures. The heat-treated samples were characterized by means of scanning electron microscopy (SEM), and X-ray diffraction (XRD) techniques. Rietveld analysis was also realized to determine the Ti, TiSiB, and TiB lattices, depending the composition alloy. Results indicated that the added zirconium was preferentially dissolved into the Ti6Si2B lattice in heat-treated Ti-6.7Zr-22.2Si-11.1B, Ti-7Zr-10Si-5B, and Ti-7Zr-30Si-5B alloy.

I – P25 MORPHOLOGY AND THERMOMECHANICAL PROPERTIES OF CELLULOSE ACETATE AND POLYSILOXANE HYBRID FILMS

C. A. Silva, I. V. P. Yoshida, M. C. Gonçalves – UNICAMP, Cidade Universitária Zeferino Vaz s/n, Barao Geraldo, Campinas, Brasil, CEP 13084-971, Caixa Postal 6154, IQ/Depto Fisico-Quimica/UNICAMP,

The growing interest in hybrid organic-inorganic materials prepared from a sol-gel process is due to the possibility of morphological control in a nanometer scale and consequently the variety of physical properties that can be reached. In this work, highly branched hybrid units (coded HB) generated from aminopropyltrietoxysilane (APTS) with pentaerytritoltriacrylate (PETA) or polyisocianurate (PI), were used as an inorganic network for the modification of cellulose acetate (CA). The molecular structure of the modified-CA was characterized by infrared spectroscopy and the thermal behavior was evaluated by thermogravimetric analysis, differential scanning calorimetry and dynamic mechanical analysis. The results showed thermal stability similar to pure CA and several relaxations, which were associated to polymer domains with different crosslinked degrees. The glass transition temperatures of the hybrid materials appeared to increase slightly with the HB content, suggesting the presence of interactions between the inorganic network and the CA matrix. The morphology was investigated by transmission and scanning electron microscopies. Energy dispersive spectroscopic analysis in the scanning electron microscope confirmed the uniform dispersion of Si in the hybrids films. The morphology of the products was characterized by HB nanodomains dispersed in the CA matrix.

I – P26 ALKALINE DEGRADATION OF BIORESORBABLE POLYMERIC SCAFFOLDS PREPARED BY COMPRESSION MOULDING AND SALT LEACHING TECHNIQUE

S. H. Barbanti, C. A. C. Zavaglia - Depto. de Engenharia de Materiais, Fac. de Engenharia Mecânica, UNICAMP, Campinas, SP, Brazil, CP 6122, CEP 13083-970; E. A. R. Duek - PUCSP

Bioresorbable polymers play an important role in tissue engineering by serving as scaffolds to guide tissue

regeneration by releasing medicines and growth factors to stimulate the tissue response, or to create a new functional structure when damaged tissue does not regenerate. This study describes a simple technique that can be used to obtain porous and dense scaffolds of polycaprolactone and poly(lactide-co-glycolide) (50:50), a biodegradable and bioresorbable polymers widely studied for biomedical applications. Dense scaffolds were prepared by melting of polymer and porous scaffolds were prepared essentially as described above, including citrate sodium. The salt was removed by immersion in distilled water. The in vitro degradation of dense scaffolds was studied in different concentration of alkaline solution of NaOH, maintained at 37°C. The scaffolds were removed from the medium after 1, 2, and 4 weeks. The morphology was analysed using a scanning electron microscope. Thermal analyses were done using differential scanning calorimetry. During degradation samples underwent significant morphological changes. The autocatalytic effect of the poly(hydroxyl acids) was observed. As larger the concentration of the alkaline medium, larger the effect in the morphology. The presented method showed a useful and low cost technique for the production of porous and dense scaffold. The simulated hydrolysis in NaOH medium allowed us to shorten the degradation time.

I – P27 NYLON 6/EPDM-g-MA/SHORT SILICA FIBER COMPOSITES

M. G. Segatelli, C. A. R. Costa, M. C. Gonçalves - UNICAMP - Instituto de Química - Departamento de Físico-Química, Cidade Universitária, Zeferino Vaz", laboratório I-113, Campinas/SP, Brasil, CEP: 13083-970, Caixa Postal 6154

The appropriate incorporation of rubber particles in a brittle plastic matrix is a well-established means of improving fracture toughness. Unfortunately, the addition of an elastomer to a rigid matrix invariably reduces strength and stiffness relative to the unmodified material. The use of high modulus fibers, on the other hand, can increase the strength and stiffness of an engineering thermoplastic. In this work, new short amorphous silica fibers were used as an alternative reinforcement for Nylon 6. However, the incorporation of fiber in a thermoplastic matrix leads to an inherent loss in ductility of composite material. To recover this property, maleated ethylene-propylene-diene rubber (EPDM-g-MA) was used as an impact modifier for Nylon 6. The morphology and mechanical performance of the obtained composites were evaluated. All the materials were dried and compounded by simultaneous extrusion in a twin-screw extruder prior to injection molding. Dogbone bars were molded for tensile, flexural and impact testings which were performed according to ASTM D638, ASTM D790 and ASTM D256, respectively. The morphology of the molded parts was investigated by field emission scanning electron microscopy (FESEM) and atomic force microscopy (AFM). Fiber surface microanalysis was carried out by energy dispersive spectroscopy (EDS). The results showed that combining fiber reinforcement with rubber toughening provides an approach to materials with high stiffness, strength and toughness.

I – P28 MORPHOLOGY OF POLYBUTENE-1/POLY (PROPYLENE-CO-ETHYLENE-CO-1-BUTENE) BLENDS

S. Silva, C. A. R. Costa, M. C. Gonçalves - IQ – Unicamp, Cidade Universitária Zeferino Vaz, Barão Geraldo, Campinas, São Paulo, Brazil, Caixa Postal, 6154, CEP 13083-970

Isotactic polybutene-1 (PB-1) has a very complex polymorphic behavior, depending on the preparation conditions and the presence of other components. In this work, we report the crystallization and morphological behaviors of isothermally crystallized polybutene-1/poly(propylene-co-ethylene-co-1-butene) blends. Polymer blends with different compositions were prepared by solution-casting and thermal treatments. The crystallization behavior and morphology of the blends were characterized by Differential Scanning Calorimetry (DSC), Field Emission Scanning Electron Microscopy (FESEM) and Pulsed Force Mode Atomic Force Microscopy (PFM). DSC results indicated a strong effect of composition and crystallization temperature on the blend crystallization kinetics. The observation of phase segregation morphology was not possible from FESEM topographic images of the fractured surfaces. However, PFM images demonstrated the coexistence of different polymer domains by imaging material contrasts from local stiffness and adhesion.

I – P29 Ti6Al4V ALLOY METALLOGRAPHIC ANALYSIS BEFORE AND AFTER CVD DIAMOND FILM DEPOSITION

T. M. Souza, J. Possato, R.A.A. Costa, J. E. Bueno, J. Oliveira - Unesp-Guaratinguetá, Av. Ariberto Pereira da Cunha,333 - B. Pedregulho, CEP: 12516-410 Guaratinguetá, SP

This work focuses the metallographic characteristics, the surface analysis, interfaces and the core material of the Ti6Al4V titanium alloy and its morphological changes when submitted to CVD diamond film deposition. It is a complete report, based on essayed researches along the precedent and completed phases of the CVD diamond film deposition, through film to subtract microstructure deposition analysis. The results are judiciously described and offer a perfect routine for researchers in the area.
Support: Fapesp, CNPq, Fundunesp, PROPP-Unesp, CVMat

I – P30 RELATIONSHIP BETWEEN WEAR OF THE SILICON NITRIDE CUT TOOL WITH FINAL WORKMANSHIP OF COMPACTED GRAPHITE IRON WORKPIECE

J. V. C. Souza, C. A. Kelly, M. R. V. Moreira, M. V. Ribeiro, S. J. Crnkovic - FEG/;UNESP, Av. Ariberto Pereira da Cunha, 333, cep. 12516-410, Guaratinguetá-SP

The relationship between wear of the silicon carbide cut tools and final workmanship of compacted graphite iron workpiece has been analised. However, for obtaining of tool cutting bit was used a powder mixture composed of 78.84 wt % % ? -Si3N4, 2 wt % Al2O3 and 6.82 wt % Y2O3. Such powder mixture was homogenized in ethanol, dry at evaporator rotative and kiln, respectively. Then, uniaxially (100 MPa) and cold isostatically (300 MPa) pressed in 16.36x16.36x7.5 mm. The samples were sintered at 1900^{o}C for 1h, in graphite element resistive furnace under nitrogen atmosphere. After sintering, the samples showed relative density higher to 98 % of theoretical density, fracture toughness and hardness equal to 5.4 MP.m1/2 and 21 GPa, respectively. The phase analysis by X-ray diffraction and microscopy electron scanning (SEM) showed the presence of ? -SiAlON and ?-Si3N4. Machining tests were realized in test specimen with 375 mm lenght, internal and external diameter equal to 60 and 90 mm, respectively. After machining tests it was proven the difficulty of machinability of the compacted graphite iron, which superficial workmanship best results were obtained for Vc = 200m/min.

I – P31 PRELIMINARY ANALYSIS OF THE Ti-6Al-4V MACHINING WITH SILICON NITRIDE CUT TOOLS

J. V. C. Souza, C. A. Kelly, M. R. V. Moreira, M. V. Ribeiro, O. M. M. Silva - FEG/;UNESP, Av. Ariberto Pereira da Cunha, 333, cep. 12516-410, Guaratinguetá-SP

This work was proposed with objective of to realize preliminary studies with relationship to machinability of Ti-6Al-4V alloys with silicon nitride (Si3N4) cut tools. However, for obtaining of tool cutting bit was used a powder mixture composed of 78.84 wt % ? -Si3N4, 2 wt % Al2O3 and 6.82 wt % Y2O3. Such powder mixture was homogenized in ethanol, dry at evaporator rotative and kiln, respectively. Then, uniaxially (100 MPa) and cold isostatically (300 MPa) pressed in 16.36x16.36x7.5 mm. The samples were sintered at 1900oC for 1h, in graphite element resistive furnace under nitrogen atmosphere. After sintering, the samples showed relative density higher to 97 % of theoretical density, fracture toughness and hardness equal to 5.3 MP.m1/2 and 21 GPa, respectively. The phase analysis by X-ray diffraction and scanning electron microscopy (SEM) showed the presence of ? -SiAlON and ?-Si3N4. Machining tests were realized no cutting fluid, using different conditions. After machining tests was observed that Si3N4 cut tools showed average cut length equal to 200 m, with abrasion and crater wear predominance. In all tests realized was proven the same behavior of ceramic cut tools with relationship to machinability of the Ti-6Al-4V alloys, having at necessity of the subsequent processes utilization for a better superficial workmanship of the piece.

I – P32 MICROSTRUCTURE OF EUTECTIC Pb-Sn SOLDER INFILTRATED IN COPPER SOLDER WICKS

R. C. Yonekura, E. F. Monlevade – IndT, Rodovia Torquato Tapajós, 7200, 3o. Andar, Tarumã - Manaus - AM. 69048-660

Eutectic Pb-Sn solder metal has been infiltrated into a copper solder wick (used to remove solder material

from electronic assemblies by capilarity) at 227.5, 273, 318 and 364oC (equivalent to homologous temperatures of 1.1, 1.2, 1.3 and 1.4 times the fusion temperature). The samples were analysed at their surface and cross-sectioned. Several eutectic morphologies have been encountered on all samples, from lamellar structures to large nodular strucutures. The limit situation eutectic morphology (finest lamellae) varies with the treatment temperature. Intermetallic compound formation and microstructures are also affected by the variation of peak temperature along the process. In some cases, the eutectic structure delineates the molten metal flow. In these cases, the morphology varies with temperature indicating the variation in molten material viscosity.

I – P33 EFFECT OF THE SYNTHESIS ROUTE ON MICROSTRUCTURAL CHARACTERISTICS OF Ce0,85Y0,13Pr0,02O2-d SOLID ELECTROLYTES

S. K. Tadokoro, E. N. S. Muccillo - CCTM/IPEN - GCEE/CMDMC, Av. Prof. Lineu Prestes, 2242, São Paulo - SP, Brasil, CEP: 05508-000

Ceria-based solid solutions have received increased attention as electrolyte material for intermediate-temperature solid oxide fuel cell systems. The main problem using ceria-based ceramics is that they develop electronic conductivity in reducing environments. The use of co-dopants to increase the extent of the electrolytic domain was suggested as an alternative approach to overcome this problem. In this work some microstructural properties of yttria-doped ceria solid electrolytes with praseodymium addition were studied. Several characterization techniques were used such as X-ray diffraction, energy dispersive spectroscopy, scanning electron microscopy, Raman microscopy, and X-ray fluorescence spectrometry. The results show that microstructural homogeneity is strongly influenced by the synthesis route. Moreover, the addition of praseodymium influences the crystallite size of calcined powders and the average grain size of sintered pellets.

I – P34 INFLUENCE OF Er:YAG LASER ENERGY IN THE MORPHOLOGY OF ENAMEL AND DENTIN ADHESIVE INTERFACES

C.S. Delfino, J.F. Sassi, R.G. Palma-Dibb - FORP-USP, Departamento de Odontologia Restauradora, Avenida do Café, s/n, Ribeirão Preto, São Paulo, Brasil, 14040904.

This study investigated in vitro the influence of Er:YAG laser energy variation for cavity preparation on the morphology of enamel and dentin adhesive interfaces, using scanning electron microscopy. Buccal enamel and middle-coronal dentin were flattened with carbide bur for control groups (n=12), and prepared with Er:YAG laser 250mJ/4Hz and 300mJ/4Hz for experimental groups (n=24). Two adhesive systems were used: One-Step Plus (total-etching) and Tyrian SPE/One-Step Plus (self-etching). The interfaces were sectioned and prepared for SEM. The analysis of the photomicrographs revealed that the enamel and dentin adhesive interfaces with Er:YAG laser, in general, were more irregular than the control group. The enamel adhesive interface of the control group showed more defined, with evident tags for the total-etching. On the other hand, for dentin, no difference was observed between the adhesives. The laser irradiated dentin did not exhibit hybrid layer, moreover, few resin tags could be observed, mainly for Er:YAG laser 300mJ. For enamel, Er:YAG laser 250mJ promoted a more regular adhesive interface, with tags formation. However, Er:YAG laser 300mJ resulted in amorphous enamel and fused areas for both adhesives systems, and it was not observed resin tags. It was concluded that the preparation with Er:YAG laser influenced the morphology of adhesive interface, and the tissual alterations were more evident when the energy was increased.

I – P35 STUDY OF Fe-19.5Cr-5Ni ALLOY PROCESSING BY HIGH ENERGY MILLING AND HEAT TREATMENTS

O.M. Cintho, C. Moinhos, E.T. Kubaski – UEPG, Depto. Engenharia de Materiais, Av Gal Carlos Cavalcanti, 4748, Bloco L, Ponta Grossa, PR, Brasil, CEP 84030-900; R.C. Yonekura, E.F. Monlevade – IndT; J.D.T.Capocchi - EPUSP

A Fe19.5Cr5Ni elemental powdeR mixture was processed in a ATTRITOR type high energy mill under inert atmosphere during 5, 9 and 10 hours. The milling was carried out using a stainless jar and chromium steel balls with a 1:50 powder-to-ball ratio. The milling products were formed in 8mm diameters pastilles and treated under inert atmosphere at 900, 1050 and 1200°C during 1hour. The final products were analyzed by x-ray diffraction analysis (XDR), scanning electron microscopy and dispersive energy spectrometry mapping in order to evaluate the alloying process. The results showed a great influence of plastic deformation levels, according to milling times, on the microstructure and components distribution along the samples. Ferrite and austenite phase in very fine grained formations were detected after heat treatments.

I – P36 HIGH RESOLUTION QUANTITATIVE PHASE ANALYSIS USING EBSD

P. E. F. Côrtes, P. C. Luna, D. S. Amorin, J. S. Ferreira - Acesita SA, Praça 1 maio, 9 Timóteo, MG, Brasil, 35180-018

When people talk about phase quantification analysis is possible list different techniques: Optic, electronic and atomic force microscopy, x ray diffractometrie, etc… Some cases sample's morphology analysis is sufficient to clarify results (using Optic or AF microscopy), another cases is important obtain chemical information (using DRX or microanalysis in SEM). Exist situations that both information (morphological and chemical) is necessary to get results. This paper makes a discussion over phase analysis quantification in martensitic stainless steel 430 (ferrite + martensite) with very low carbon and show EBSD technique as good tool for resolve very small differences between phases.
Searching for the confirmation of the sample's martensite percentage, two techniques were used: optic microscopy and X-ray diffractometrie. However, due to a tiny difference between the phase's planning spacing(d), was not possible identify it, using this last one. For that reason, it was defined study the capacity of EBSD analysis to resolve this problem.
This work makes a comparison among the three techniques: optical microscopy, X-Ray diffractometrie and EBSD microscopy and yours applications in quantitative phase analysis, showing that EBSD technique is a good, efficient and high resolution tool for phase mapping analysis.

I – P37 FOLLOWING THE HYDRATION AT DIFFERENT AGES OF A NON STRUCTURAL PORTLAND CEMENT CONCRETE CONTAINING RECYCLED SANITARY WARE AGGREGATES

J. S. Costa, C. A. Martins, J. B. Baldo – Dema, UFSCar, Rodovia Washington Luis km 235, São Carlos, SP, Brazil, caixa postal 676, CEP 13560-905

In this work the SEM imaging was used to follow the hydration products development of a Portland cement concrete, made by means of recycled aggregates produced from rejects of the of the ceramic sanitary ware industry. The aggregates were produced by crushing and grinding toilet bowls in a grain size distribution of 100% passing 9,4 mm screen. The Portland cement concrete composition investigated was produced under the ratio 1:2.5:3.5 (cement:river sand:aggregate). The curing ages were extended for 28, 91 and 360 days. By using SEM imaging it was found that no important differences between the hydration products of the non structural Portland cement concrete containing the recycled aggregates comparatively to a common Portland cement concrete of similar composition containing limestone aggregate instead of recycled aggregates, with great benefits from the point of view of environment and mineral resources preservation.

I – P38 PLAIN CARBON STEEL EXPOSED TO AQUEOUS EXTRACT FROM SOIL – AN ANALYSIS OF THE CORROSION PRODUCTS

S. L. JESUS, D. W. A. JESUS, V. V. ALMEIDA - ESALQ/USP- NÚCLEO DE PESQUISAS EM GEOQUÍMICA E GEOFÍSICA DA LITOSFERA, DEPARTAMENTO DE SOLOS E NUTRIÇÃO DE PLANTAS, ESCOLA SUPERIOR DE AGRICULTURA LUIZ DE QUEIROZ, USP, PIRACICABA, AV. PÁDUA DIAS, 11 CP 9, BRASIL; I. COSTA, J. L. ROSSI - IPEN

Metallic materials such as those used in pipes and in storage tanks undergo a corrosion process when in contact with soil. This may result in severe structural damage, leakage of the transported or stored materials and eventually environment contamination. The aqueous extract from soil (soil-solution) is a highly reactive solution that concentrates chemical elements, either dissolved, as complexes or in suspension. This soil-solution can be used as a corrosion medium. In this study, plain carbon SAE 1020 steel coupons were immersed in soil-solution and the corrosion products were analyzed by scanning electron microscopy and X-ray diffractometry.

I – P39 SYNTHESIS OF POLIVYNILIDENE FLUORIDE CONTAINING BIOGLASS-ALUMINA AND HYDROXYLAPATITE PARTICLES FOR USE AS ARTIFICIAL TISSUE

Tsai Hui I, J. C. Z. Silva, S. M. Tebcherani, C. P. F. Borges – UEPG - CIPP-DEQUIM Av Carlos Cavalcante, 4748 Ponta Grossa Paraná, Brazil CEP-84030-900; A. V. C. Andrade - UEPG-DEFIS-CIPP; M. B. Marques - UEPG-DEQUIM

Allogenic and alloplastic bone grafts substitutes serves either as bionert or bioactive osteoconductors. The present research aims to evaluate the possibility of creating new bioactive composite based on a polyvinilidene fluoride polimer(PVDF) containing the bioglass system based on Na2O-CaO-SiO2-P2O5 and the PVDF-hydroxylapatite(HAP). Such combinations should allow for the development of bioactive composites with great potential for a restorations applications. A blend of PVDF was combined with bioglass-alumina(BV-AL) and HAP particles(0,1,5, 10, 30, 50% in weight) in a heated media(75 degrees) using Di-methilformamide(DMF)as solvent, and then put in a ultrassonic bath to obtain the particles dispersion. The BV-AL and HAP particles used had a median size 6.0 micrometers and the polymer used had a medium viscosity and hence was suitable for in a doctor-blade simulated. The blend was dried in a stirring-heater(60 degrees) to obtain a homogeneous solid thin film and the foam form. The analysis of samples by incidence grating X Ray Diffraction Patterns shows the crystals phases: PVDF and SiO2 and amount of non-crystalline material, for the PVDF-HAP shows the phases: PVDF and pure HAP. Analysis by SEM of thin films shows a uniform microstructure and the particles are infiltrated in the matrix polymer, and for the foam sample its observed interconnected porous structure with the particles and the integrity structural. These results are important issues for tissue fabrication.

I – P40 COMPARATIVE ANALYSIS OF DIFFERENT ETCHING METHODS TO REVEAL CEMENTED-CARBIDE TOOL MICROSTRUCTURE

M. R. V. Moreira, R. B. Ribeiro, M. V. Ribeiro, C. A. Kelly, J. F. Lins, E. A. Cunha, E. A. Raymundo - Departamento de Engenharia de Materiais – DEMAR; J. V. C. Souza - Faculdade de Engenharia Química de Lorena – FAENQUIL, Pólo Urbo-Industrial, Gleba AI-6, s/n, Lorena-SP, Cep. 12600-000

This work had as objective evaluates the efficiency of different etching methods for to reveal cemented-carbide tool microstructure (type TNMG 110408 ISO S10), provided for the SANDVIK-Coromant. Therefore, cut tool were submitted to different etching methods, chemical and thermal. Before of to be subjected to the etching, cut tool were sectioned longitudinally, it incrusted at fenolic resins and then submitted to the materiallographic preparation, using diamond sandpaper and paste until 3?m, in automatic buffer system. For chemical etching was used "Murakami" reagent (5g $K_3Fe(CN)_6$ + 5g KOH + 100 ml H_2O) in a 2 min interval until 10 minutes maxim time. Whereas, for thermal treatment, the samples were encapsulated under argon atmosphere and heated at 400 and 500^oC for 1h, respectively. After different etching, a better efficiency of the thermal treatment with relationship to the microstructural revelation of the cemented-carbide tool was characterized by light and scanning electron microscopy. Qualitative factor used for to determine the efficiency of the different etching was count of the intercepted grains. In cut tool chemically etched, the better micrographics were obtained for 10 minutes, but for thermal etching the best results were obtained at 500^oC by 60 min.

I – P41 ANALYSIS OF MICA FROM FLOGOPITE-BIOTITE GROUP BY OPTICAL MICROSCOPY, SEM AND EDS MAPPING

L. L. Antonello, C.A. Ávila, M.E. Zucolotto, H.R. Barrueto - UFRJ/MN/DGP, Museu Nacional, Quinta da Boa Vista- São Cristóvão. CEP: 20940-040- Rio de Janeiro-RJ; R.M. Pereira - UERJ/DG

Studying thin sections of metamorphic rocks from Piquete region (São Paulo, Brazil), it was noted the presence of mica crystals with a remarkable pleochoism, varying from yellow to brownish yellow colour spectrum. Thus, the aim of this study will be centered on the analysis of optical and chemical properties presented by these mica group, focusing if they belong to the phlogopite or biotite member, an important issue that can satisfactorily to establish the genetic constraints between these mineral occurrences and growth. Within the mica group, phlogopite constitutes an important end member, included in the tri-octahedral mineral class with layered structures having Mg:Fe ? 2:1; biotite, is an iron-rich tri-octahedral mica which is arbitrarily differentiated from phlogopite on its lower Mg:Fe ratio (? 2:1). Although in the transmitted light evaluation revealed the pale yellow colour frequently exhibited by phlogopite, in this case it is biotite that shows this yellowish colour because it is related to its composition an intermediate proportions of Ti and Fe^3. The backscattering electron image (BSEi) shows morphological aspects of biotite. The micro chemical analysis of biotite by SEM/EDS shows the elementary composition with presence of O, Al, Si, K, Fe, Mg, Ti and Zn. The lower Mg:Fe ratio (? 2:1) indicate biotite.

I – P42 CHARACTERIZATION OF TOPOGRAPHIC AND CHEMICAL MODIFICATIONS IN THE SURFACE OF PE AND PET SUBMITTED TO CHEMICAL OXIDATION

E. RADOVANOVIC, A. F. RUBIRA, S. L. FÁVARO - DQ - UEM, Av. Colombo, 5790, Maringá, Paraná, Brazil, CEP 87020-900

The macroscopic adhesion properties of pretreated polymer can be improved by changing the surface topography. The improvement can be attributed to chemical modifications (radical formation, incorporation of new chemical species, removing of contaminants), to physical changes (electrical and thermal conductivity, surface charging), or to new mechanical properties (increased roughness). In this work, PE and PET films were submitted to oxidative treatments with solutions of KMnO4 (0,1M or 0,5M) in HCl (0,2M or 1,0M, respectively) at temperatures of 298K or 318K, for 2h or 8h. Then, eight different treated films were obtained of each polymer and the chemical and morphological modifications were analyzed by drop water contact angle, FTIR-ATR, TGA, SEM and AFM. According to the effectiveness of the treatment the drop water contact angle decreased of 92 to 60 degrees in PE and of 79 to 63 degrees in PET. The oxidative process generated C=O groups in PE films, characterized by a peak at 1730cm-1 in FTIR-ATR spectra, and an increase in the intensity of this already present peak in the PET FTIR-ATR spectra. It was not observed changes in mass loss in the thermogravimetric analysis of the polymers, suggesting that the oxidative process does not affect the bulk of the films. The morphology of the polymer changed and it was possible to observe that the oxidative treatments could increase the roughness or scratch the polymer surface revealing their crystalline phase.

I – P43 MICROSTRUCTURAL STUDIES OF PtIr and PtIrMo ALLOYS PREPARED BY VACUUM ARC-MELTED FURNACE

D. M. DOS ANJOS, R. M. BEZERRA, G. TREMILIOSI-FILHO – IQSC, LABORATÓRIO DE ELETROQUÍMICA, AV. TRABALHADOR SÃOCARLENSE, 400, SÃO CARLOS, SP, BRAZIL, 13566-590.

Platinum is widely used as an electrode material in electrocatalytic process due to its high stability in several electrolytic mediums besides its high capacity to adsorb organic and inorganic species, being the main advantage with relation to the other noble metals. The application of platinum in energy conversion devices, as in Direct Ethanol Fuel Cells, is limited due to the strong adsorption of intermediate species, which impedes the continuity of the reaction, decreasing the energy density that could be reached. The addition of other metals to platinum, like Sn, Ru, Ir and Mo, forming binary and ternary alloys, has showed expressive enhanced in the activity for ethanol oxidation. In this work were prepared PtIr (80:20) and PtIrMo (81:13:06) alloys in vacuum arc-melt furnace, in way to obtain homogeneous materials, for further application in the electrooxidation of ethanol. According to the phase diagrams of these alloys, there are a great probability of phase segregation and formation of polyphasis microstructures. The optical microscopic analysis showed a granular structure for the PtIr alloy and a mixed structure of dendrite and granular phases for the PtIrMo alloy. The elemental chemical microanalysis by SEM-EDS suggests a homogeneous distribution of the elements that form the PtIr and PtIrMo alloys, without observing segregated phases.

I – P44 ANALYSIS OF MAGNETIC BEHAVIOR IN BARIUM HEXAFERRITE USING MFM

P. I. Paulin, S. M. Gheno, R. R. Correa, Departamento de Engenharia de Materiais – UFSCar - Rod. Washington Luiz, Km 235, Cx. Postal:676, CEP: 13565-905, São Carlos-SP, Brasil, Fone: (16)3351-8255; Fax: (16)3351-1404

The magnetic force microscope (MFM) is a special way of operation of atomic force microscope (AFM) and is an important and advanced tool that makes possible the detection of magnetic interactions, among others classic magnetometrics methods for observation of magnetic domains structures. Scanning microscopy using magnetic probes is an important tool for mapping magnetic structures of surfaces in magnetic materials. The barium hexaferrites are hexagonal ferrimagnetic oxides that perform a very important role in the technological applications of magnetism. In traditional applications such as motors, generators and transformers they are used as permanent magnets and have the property of generate a constant magnetic field. Another traditional application of the magnetic materials, which has acquired great importance over the last decade, is the non-volatile storage of information through recording and re-recording, which is essential for operation of tape and video recorders, equipment using magnetic cards and computers. The barium hexaferrite used in this work was obtained by sintering of powders processed by high-energy milling followed by thermal treatment and chemical attack to reveal the grains. Images of magnetic domains obtained using magnetic force microscopy. It was observed that magnetic domains became more intense and defined, showing sizes around 2.9μm, when the magnetic field of 8.40kOe was applied.

I – P45 PHASE DIAGRAM AND EUTECTIC MICROSTRUCTURE CHARACTERIZATION IN THE Al-Cr-Nb SYSTEM

R. Caram - UNICAMP-FEM, 13083-970, Campinas, SP, Brazil, C.P.6122; C.T. Rios, S.A. Souza, M.C. Gonçalves, S. Gama – UNICAMP; P.L. Ferrandini - Centro Universitário da FEI

Solidification behaviour and structural characteristics of eutectic alloys in many systems continue to attract

interest because of its influence on properties and performance of materials containing eutectic constituents. The eutectic alloys processed by directional solidification allow one to obtain highly anisotropic structures of two or more phases, with regularly arranged microstructures. In addition, it is a promising technique for production of the so-called in situ composites. This work reports a morphological study of a eutectic alloy in the Al-Cr-Nb system, in its Al rich region. The solidification morphology of the alloys was studied both in the as-cast and in the directionally solidified conditions. The samples were first obtained in an arc furnace and then directionally solidified using a Bridgman equipment. During the solidification process the growth rates utilized varied from 10.0 to 30.0 mm/h. The temperatures of phase transformations were identified by means of differential thermal analysis and the eutectic transformation temperature was found to be near 1366.2 °C. Optical and scanning electron microscopy were used in order to determine the influence of the solidification conditions on the microstructure. It was concluded that the alloy presents a near regular lamellar growth mode.

I – P46 SAMARIUM-HOLMIUM-IRON GARNET NANOPOWDER

V. R. Caffarena – CBPF, Rua Dr. Xavier Sigaud, 150 - Urca - RJ - Brazil - ZIP CODE: 22290-180; T. Ogasawara, J. L. Capitaneo - PEMM-COPPE; M. S. Pinho - IPqM

The aim of this work was the synthesis and characterization by XRD, XRF, SEM and EDX of the cubic ferrite nanopowder having a molecular formula that can be expressed as Sm3Fe5O12. The synthesis of this magnetic material was carried out by the co-precipitation method, starting from hydrated chlorides of the rare-earth elements and ferrous sulfate. A binder was added to the calcined powder in order to facilitate the production of a toroidal compact, which was submitted to sintering at six different temperatures in the range of 1200oC to 1450oC. We have studied the relation between its magnetic properties and morphology by SEM.

I – P47 EBSD ANALYSIS OF GRAIN BOUNDARY ENGINEERED INCONEL 600 SUBMITTED TO CREEP TEST

A. L. PINTO, C. S. C. VIANA – IME, PCA GEN TIBURCIO, 80; L. H. ALMEIDA - COPPE/UFRJ; H. R. Z. SANDIM - FAENQUIL

Grain boundary engineering has been proposed as a new way of enhancing materials properties which are sensitive to intergranular phenomena. The basic idea involved is to increase the amount of CSL (coincidence site lattice) boundaries through thermomechanical treatments. These boundaries are considered to have lower energy and greater resistance to dislocation dissociation. For CFC alloys with low to medium stacking fault energy such as the nickel alloys, treatments that have shown greater success in increasing the amount of CSL boundaries consist of medium deformation and recrystallization followed by iterative cycles of low deformation and annealing. The main types of CSL boundaries thus generated are primary and secondary twins. Inconel 600 samples submitted to such treatments were evaluated through creep tests showing great improvement in the resistance to creep. The EBSD technique was employed not only to evaluate the amount of CSL boundaries but also to evaluate the path of the growing cracks. The analysis of the microstructure of the samples thus tested has shown that cracks avoid CSL boundaries which explains the increase in the creep resistance observed.

I – P48 OPTICAL, STRUCTURAL AND ELECTRICAL CHARACTERISTICS OF ANNEALED ALEXANDRITE

R.M.F. Scalvi, N.M. Trindade, L.O. Ruggiero, M.J. Saeki - UNESP-FC, Av. Eng. Luis Edmundo C.Coube, s/n, caixa postal 473, CEP 17033-360, Bauru, SP; M. Siu Li - USP-DFCM

Alexandrite is a sort of chrysoberyl ($Be_2Al_2O_4$), with Cr^{3+} into its lattice. One of the most important alexandrite properties is as laser host, with tunable emission range of 100nm, centered at 750nm. This laser presents low threshold current for operation in a 4 level system at room temperature. However, the way that substitution of Al^{3+} ions by Cr^{3+} in the crystalline lattice takes place is rather unknown. Several reports have been done concerning its optical and magnetic properties, but very few concerns its electrical properties. In this work, we report optical absorption from ultraviolet to near infrared which allows verifying the optical properties of this material influenced by the presence of impurities in the host matrix. Besides, we also show electrical characterization using thermally stimulated depolarization current (TSDC) technique, which makes clear the formation of electric dipoles in this material. TSDC bands are broad and centered about 188K and 195K for natural alexandrite samples from Minas Gerais state, Brazil. Electrical characterization as well as optical absorption have been carried out as function of consecutive annealing processes, varying time and temperature, and it has been verified a vanishing of absorption bands and a TSDC band shift to lower temperatures. The characterization by electrical, optical and structural analysis will yield a complete understanding of dipole relaxation phenomena, which was obtained from TSDC results.

I – P49 PREPARATION AND CHARACTERIZATION OF SnO_2 THICK FILMS BY COATING METHOD FOR VARISTORS APPLICATIONS

M. A Ramírez, A. Z Simões, Y. Maniette, J. A Varela – IQ - UNESP, Rua Francisco Degni s/n, CEP 14800-900; E. Longo - UFSCar

Thick films of $SnO_2.CoO.Nb_2O_5.Cr_2O_3$ based varistors were obtained from powders (mixed oxide) dissolved in on organic resin with controlled viscosity. The solution was deposited on Al_2O_3 substrates by spin-coated technique by appling a rotation speed of 5000 rpm. The four-layered films were sintered at 1300°C by 2 hours. XRD analyses showed the formation of a single phase of cassiterite type. SEM analyses showed an uniform microstructure and films with 4.0 mm of thickness. AFM showed grains with size of about 2.2 mm. By TEM (surface analyses) no microprecipitates were observed in the grain boundary while the diffraction electron pattern indicated the formation of a very crystalline phase.