3RD BRAZIL MRS MEETING

October 10-13, 2004

SYMPOSIUM H:

PARTICULATE MATERIALS: PROCESSING, PROPERTIES AND APPLICATIONS

Symposium Organizers:

Antonio Eduardo Martinelli (DEM – UFRN)

Aloysio N. Klein (LABMAT - UFSC)

Carlos Alberto Paskocimas (DEM – UFRN)

Dulce Maria de Araújo de Melo (DQ - UFRN)

SYMPOSIUM H

PARTICULATE MATERIALS: PROCESSING, PROPERTIES AND APPLICATIONS

Monday, October 11

Session Chair: Uílame U. Gomes (UFRN)

08:00 Invited POWDER METALLURGY PROCESSING OF TITANIUM ALLOYS: RELEVANT MICROSTRUCTURAL ASPECTS
H. R. Z. Sandim (FAENQUIL) (H – I001).

08:45 PRODUCTION OF TI-35Nb BY POWDER METALLURGY TECHNIQUES
D. R. dos Santos, M. Pereira. FEG/UNESP. Praça Eduardo Gomes, N.50, Vila das Acácias, 12228-904, São José dos Campos-SP. V. A. R. Henriques, C. A. A. Cairo, M. L. A. Graça. CTA (H – O042).

09:00 MICROSTRUCTURE OF Ti-35Nb-7Zr-5Ta ALLOY PRODUCED FROM HYDRIDE POWDERS
E. B. Taddei. ITA. Instituto Tecnológico de Aeronáutica , Praça Marechal do Ar Eduardo Gomes, 50, São José dos Campos, Brazil,C.P. 6044. V. A. R. Henriques, C. R. M. Silva, C. A. A. Cairo. CTA (H – O037).

09:15 FABRICATION PROCESS BY POWDER METALLURGY FOR BIOCOMPATIBLE Ti-13Nb-13Zr ALLOY IMPLANTS
V. A. R. Henriques, C. R. M. Silva, C. A. A. Cairo. CTA. Rua Heitor de Andrade, N.1604, Jd. das Indústrias, CEP 12241-000, São José dos Campos, SP, Brasil. J. C. Bressiani. IPEN (H – O003).

09:30 KINETICS OF THERMAL DECOMPOSITION OF TITANIUM HYDRIDE POWDER USING IN SITU HIGH-TEMPERATURE X-RAY DIFFRACTION (HTXRD)
H. R. Z. Sandim, B. V. Morante, P. A. Suzuki. DEMAR-FAENQUIL. Polo Urbo-Industrial s/n, Caixa Postal 116, Lorena-SP, 12600-970, Brazil (H – O028).

09:45 STUDY OF Ni-Al INTERMETALLIC SYNTHESIS MECHANISMS BY TEMPERATURE EVALUATION DURING HIGH ENERGY MILLING
O. M. Cintho, E. T. Kubaski, C. Moinhos. UEPG. Depto. Engenharia de Materiais, Av. Gal Carlos Cavalcanti, 4748, Ponta Grossa, PR, Brasil, CEP 84030-900. R. C. Yonekura, E. F. Monlevade. IndT. J.D.T. Capocchi. EPUSP (H – O019).

10:00 COMBUSTION SYNTHESIS OF NbAl3 MECHANICALLY ACTIVATED IN SHAKE AND PLANETARY BALL MILLS
C. J. Rocha, V. S. Gonçalves, R. M. Leal Neto. IPEN. Av. Lineu Prestes, 2242, Cidade Universitária, São Paulo, Brazil, CEP: 05508-900(H – O044).

10:10 COFFEE BREAK

Session Chair: Hugo Sandim (FAENQUIL)

10:30 TUNGSTEN OXIDE REDUCTION BY METALLIC ALUMINIUM USING HIGH ENERGY MILLING
O. M. Cintho, H.-I Tsai, M. Bär. UEPG. Depto. Engenharia de Materiais, Av. Gal Carlos Cavalcanti, 4748, Ponta Grossa, PR, Brasil, CEP 84030-900. R. C. Yonekura, E. F. Monlevade. IndT. J. D. T. Capocchi. EPUSP (H – O020).

10:45 INFLUENCE OF THE PROCESS CONTROL AGENT (PCA) IN THE MECHANICAL ACTIVATION OF Nb75Al POWDER MIXTURES
V. S. Gonçalves, C. J. Rocha, R. M. Leal Neto. IPEN. Av. Lineu Prestes, 2242 - Cidade Universitária, CEP 05508-000, São Paulo, SP, Brazil (H – O004).

11:00 ELETROCHEMISTRY HYDROGENATION OF Nd2Fe17 AND Nd2Fe17Ga1,5
M. A. Zampronio, A. Destefano, C. C. Colucci, M. A. C. de Melo, C. S. Alves. UEM, Departamento de Fisica, Av. Colombo 5790, 87020-900 Maringa – PR. N. E. de Souza. UCP-Pitanga (H – O023).

11:15 REDUCTION-DIFFUSION PREPARATION OF Nd15Fe77B8, NdFe11Ti, NdFe10.5Mo1.5 AND NdFe10.75Mo1.25 ALLOYS FOR MAGNETS
E.G. Guilherme, J.O.A. Paschoal. IPEN. Centro de Ciências e Tecnologia de Materais, Av. Prof. Lineu Prestes, 2242, Cidade Universitátia, São Paulo, Brazil. H.R. Rechenberg. IFUSP (H – O038).

11:30 ELECTRODEPOSTION OF CoPd ALLOYS FROM CHLORIDE BATH AND THEIR MAGNETIC PROPERTIES
R. D. Noce, A. V. Benedetti. IQ/UNESP. Rua Prof. Francisco Degni s/n, Quitandinha, Araraquara-SP, Brazil , Cep 14.800-900. F. M. Takata, P. T. A. Sumodjo. IQ/USP/SP (H – O013).

11:45 MAGNETIC PROPERTIES OF Gd5Ge2Si2Hx ALLOYS, x  0.1
C. S. Alves, C. C. Colucci, M. A. C. de Melo, M. A. Zampronio. UEM. Univ. Estadual de Maringá, CTM, R. Cantor Raul Seixas, 501, Jd. Ipanema, 87053-240, Maringá, PR, Brazil. S. Gama, A. M. G. Carvalho, A. A. Coelho, M. A. Bolanho, M. A. B. Mendes, T. S. Docé. Unicamp (H – O043).

12:00 STRUCTURAL AND THERMAL PROPERTIES OF Co-Cu-Fe HYDROTALCITE STUDIED BY 57Fe MOSSBAUER SPECTROSCOPY AND X-RAY DIFFRACTION
D. X. Gouveia, A. G. S. Filho, J. A. C. Paiva, J. Mendes Filho. UFC, Depto de Fisica, Caixa Postal 6030, CEP 60455-900, Fortaleza, Ceara, Brasil; A. H. Iglesias, O. P. Ferreira, O. L. Alves. IQM-UNICAMP (H – O001).

12:15 MICROSTRUCTURAL CHARACTERISATION AFTER HEAT TREATMENT OF SINTERED VALVE SEAT INSERTS
E. S de Jesus Filho, L. Salgado, E. R. B. de Jesus, J. L. Rossi. IPEN. Av. Prof. Lineu Prestes, 2242, Cidade Universitária, São Paulo - SP, Brazil - CEP 05508-000. S. L. de Jesus. ESALQ. M. A. Colosio. GMB. J. C. Santos. GMB (H – O036).

12:25 LUNCH

Session Chair: Aloísio N. Klein (UFSC)

14:00 Invited MINERAL PROCESSING AND METALLIC PARTICULATE MATERIALS
U. U. Gomes (UFRN) (H – I002).

14:45 PLASMA DEBINDING AND SINTERING OF PARTS PRODUCED BY POWDER INJECTION MOULDING (PIM)
D. Fusão, A. N. Klein, J. R. Muzart, P. Wendhausen. UFSC. Laboratório de Materiais, Bloco B, EMC, Campus Universitário s/n, CP 476, Trindade, Florianópolis, CEP: 88040-900. L. Mendes. PUC-PR (H – O040).

15:00 TO BE ANNOUNCED (H – O017).

15:15 MICROSTRUCUTURAL DEVELOPMENT OF PLASMA SINTERED NbC- AND TaC-REINFORCED STEEL
A. E. Martinelli, R. M. do Nascimento, C. Alves Jr.. UFRN. Laboratório de Cimentos, Depto de Química, UFRN, Campus Lagoa Nova s/n, Natal, RN, 59072-970. D. S. A. Paulo. CEFET-RN. A. N. Klein. UFSC. J. H. E. Silva. UFPB (H – O051).

15:30 SURFACE CHARACTERIZATION OF PLASMA SINTERING UNALLOYED IRON ENRICHED WITH DEPOSITED NICKEL: CORROSION DEPENDENCE FROM POROSITY AND SURFACE COMPOSITION
R. Marchiori, A.M.Maliska, A.N. Klein, J.L.R. Muzart. UFSC. P. Borges. CEFET Curitiba, Paraná (H – O014).

15:45 A MODIFIED MASS SPECTROMETRY DIAGNOSTIC TECHNIQUE FOR THE STUDY OF PLASMA-SURFACE INTERACTION WITH HIGH TEMPERATURE SURFACES APPLIED TO POWDER METALLURGY STUDIES.
A. Seeber. UNOESC/UFSC. Labmat/Depto. Eng. Mecânica/UFSC, Trindade, Cep 88040-900, Florianópolis, Brazil, CP 476. C.V. Speller. UFSC. H. R. T. Silva, P. Egert. UNISUL. A. S. S. Sobrinho (H – O012).

15:55 COFFE-BREAK

16:20 POSTER SESSION

Tuesday, October 12

Session Chair: Dulce Melo (UFRN)

08:00 Invited SYNTHESIS OF ELECTROCERAMICS BY SOFT CHEMISTRY TECHNIQUES
E. N. S. Muccillo (IPEN) (H – I003).

08:45 SYNTHESIS AND STRUCTURE OF THE CRYSTALLINE Li2Zn1-xNixTi3O8
D. M. A. Melo. UFRN, Laboratório de Análise térmica e Materiais, Departamento de Química, Campus UniversitárioS/N, 59072-970, Natal/RN-Brasil. V. G. Oliveira, M. A. F. Melo. UFRN/DEQ. C. A. Pascocimas. UFRN/DEM. M. Sassaki. UFC/DF. E. Longo. UFSCAR/LIEC (H – O039).

09:00 INFLUENCE OF COBALT ADDITION IN THE PHASE TRANSITION ZrO2 TO ZrSiO4 OBTAINED BY MEANS OF THE POLYMERIC PRECURSORS METHOD
S. Cava, F. A. Santos, S. M. Tebcherani, A. C. Antunes, R. M. Antunes, S. A. Pianaro. UEPG. LIMAC-CIPP, Av. Gal. Carlos Cavalcanti, 4748, CEP 84032-900, Campus Uvaranas, Ponta Grossa-PR, Brasil. C. A. Paskocimas. UFRN. J. A. Varela. UNESP. E. Longo. UFSCar (H – O046).

09:15 MECHANICAL PROPERTIES OF Ce-TZP CERAMICS OBTAINED FROM FREEZE-DRIED POWDER MIXTURES
M. C. A. Nono. INPE - Instituto Nacional de Pesquisas Espaciais (INPE), Laboratório Associado de Sensores e Materiais (LAS), Av. dos Astronautas, 1758, CEP 12227-010, São Jose dos Campos, Brasil. (H – O029).

09:30 NIOBIUM SILICATE GLASSES PREPARED BY CHEMICAL PROCESS
M. J. Bertolini, M. A. Zaghete, R. Gimenes, C. O. Paiva-Santos, J. A. Varela. IQ - UNESP – ARARAQUARA. Laboratório Interdisciplinar de Eletroquímica e Cerâmica, Rua Francisco Degni, s/n, Araraquara, Brazil (H – O011).

09:45	TO BE ANNOUNCED (H – O050).
10:00	COMPARISON OF TEXTURAL, STRUCTURAL AND MAGNETIC PROPERTIES OF NICKEL FERRITE PARTICULATE MATERIALS AND NANOCOMPOSITES J. B. Silva, CDTN/CNEN. TR2 - Rua Prof. Mário Werneck, s/n,Campus UFMG, Pampuha, CEP: 30161970. BH, MG. N. D. S. Mohallem. UFMG (H – O008).
10:10	COFFEE BREAK
Session Chair:	Antonio E. Martinelli (UFRN)
10:30	TO BE ANNOUNCED (H – O032).
10:45	STUDY ON CALCINATION IN SnO2-MnO2 SYSTEMS D. Berger, T. Sequinel, S. M. Tebcherani, S. Cava, J. C. Z. Silva, A. V. C. Andrade, S. A. Pianaro. UEPG. LIMAC-CIPP, Av. Gal. Carlos Cavalcanti, 4748, CEP 84032-900, Campus Uvaranas, Ponta Grossa-PR, Brazil. M. A. L. Nobre. FCT/UNESP. P. D. Spagnol. IQ/UNESP (H – O009).
11:00	CONTRIBUTION TO THE KINETICS OF ZINC FERRITE FORMATION M. C. G. Marroquín. J. C. D\' Abreu. H. M. Kohler. R. N. Rodrigues Filho (H – O026).
11:15	PREPARATION OF ZIRCON PIGMENTS FROM THE ZrSiO4-Co3O4 SYSTEM J. D. Albani, A. C. Antunes, S. Cava, S. A. Pianaro, S. R. M. Antunes. UEPG. Laboratóro Interdisciplinar de Materiais Cerâmicos, Avenida Carlos Cavalcanti, 4748, CEP 84030-900, Ponta Grossa, Paraná, Brasil. T. T. Tominaga. Unicentro. E. Longo. UFSCar (H – O022).
11:30	POROUS CERAMICS FORMING BY “SOYA PROTEIN CONSOLIDATION” J. G. A. Santana, T. G. Cruz, E. Campos, L. R. O. Hein. DMT - FEG – UNESP. Departamento de Materiais e Tecnologia - Rua Ariberto Pereira da Cunha, 333 Pedregulho, Guaratinguetá, São Paulo, Brazil, CEP 12516-410 (H – O034).
11:45	PHOTOLUMINESCENCE IN DISORDERED RUDDLESDEN-POPPER HOMOLOGOUS SERIES L. G. P. Simões, J. A. Varela. CMDMC/LIEC/UNESP. Instituto de Química, UNESP, C.P. 355, 14801-900 Araraquara, SP, Brazil. P. S. Pizani. UFSCar-DF. E. Longo. CMDMC/LIEC/UFSCar (H – O007).

12:00 INFLUENCE OF VANADIUM IN ZINC TITANATE MORPHOLOGIC (Zn2TiO4), STRUCTURAL AND PHOTOLUMINESCENT PROPERTIES
M. S. C. Câmara, A. C. Chaves, L. L. M. Sales, D. M. A. Melo. UFRN. Laboratório de Análise Térmica e Materiais. E. Longo. UFSCar. A. G. Souza, I. M. G. Santos. UFPB (H – O006).

12:15 A STUDY OF THE PHOTOLUMINESCENCE OF Li2TiSiO5 AND Na2TiSiO5 COMPOUNDS
V. C. Albarici, M. T. Escote, E. Orhan, R. C. Lima, E. Longo, E. R. Leite. CMDMC, LIEC. Rodovia Washington Luís, Km 235, CEP: 13565-905, São Carlos, São Paulo, Brasil. N. S. L. S. Vasconcelos, J. Vasconcelos, J. A. Varela. UNESP, CMDMC, LIEC (H – O002).

12:25 LUNCH

Wednesday, October 12

Session Chair: Carlos Alberto A. Cairo (CTA)

08:00 Invited INJECTION MOULDING OF CERAMIC AND METALLIC POWDERS
Rubens M. Nascimento (UFRN) (H – I004).

08:45 THE EFFECTS OF POWDER MILLING ON DENSIFICATION OF BORON CARBIDE
P. A. S. L. Cosentino, C. A. da Costa Neto. PEMM/COPPE. UFRJ, Metallurgical and Materials Engineering Program, University City – Tecnological Center - Bloco F, sala F-210, Ilha do Fundão - Caixa Postal 68505, Rio de Janeiro, RJ - CEP 21941-972 - BRASIL (H – O018).

09:00 EFFECT OF B4C, C AND ZrO2 ADDITIVES ON PRESSURELESS SINTERING OF SILICON CARBIDE
M. S. S. Beltrão, C. A. Costa. UFRJ. COPPE/UFRJ. Programa de Engenharia Metalúrgica e de Materiais. Cid. Universitária-Centro de Tecnologia. Bloco F. Sala F210. Caixa Postal 68505. Rio de Janeiro, RJ. CEP 21941972. Brasil. M. A. P Santos. IpqM. (H – O027).

09:15 MICROSTRUCTURAL EVOLUTION DURING LIQUID PHASE SINTERING OF ALN WITH CaCO3 AND CAO ADDITIONS
H. N. Yoshimura, A. L. Molisani, G. R. Siqueira. IPT. Instituto de Pesquisas Tecnológicas do Estado de São Paulo, Laboratório de Tecnologia Cerâmica, Av. Prof. Almeida Prado, 532, São Paulo, SP, Brazil, 05508-901. H. Goldenstein. EPUSP (H – O033).

09:30 THE APPLICATION OF CLASSIC MODELS OF INITIAL STAGE OF SINTERING
L. Kupchak, M. V. Parizotto, S. R. M. Antunes, C. Antunes, A. Pianaro, S. M. Tebcherani. UEPG. Laboratório Interdisciplinar de Materiais Cerâmicos, Av. carlos Cavalcanti, 4748, Ponta Grossa, PR, Brazil (H – O031).

09:45 EQUILIBRIUM DIHEDRAL ANGLE DETERMINATION OF THE SnO2 SINTERING
S. M. Tebcherani, S. Cava. UEPG. LIMAC-CIPP, Av. Gal. Carlos Cavalcanti, 4748, CEP 84032-900, Campus Uvaranas, Ponta Grossa-PR, Brasil. J. A. Varela, P. D. Spagnol, M. Cilense. UNESP. E. R. Leite, E. Longo. UFSCar (H – O010).

10:00 IN-SITU OBSERVATION OF GLASS PARTICLE SINTERING
M. J. Godinho, R. Aguiar, E. Longo, E. R. Leite. LIEC-UFSCar. R: Candido Padim 699, São Carlos-SP, Brasil. (H – O025).

10:10 COFFEE BREAK

Session Chair: Alexandre Lago (UFSC)

10:30 MECHANICAL BEHAVIOR OF THE CARBON CARBON COMPOSITE COATED WITH CERAMIC CARBIDES BY CVR PROCESS
C. A. A. Cairo, B. S. A. Perez, M. L. A. Graça. IAE/AMR. Pça Mal. Eduardo Gomes, 50, Vila das Acácias, 12228-904, São Jose dos Campos, São Paulo, Brazil. M. Florian. ITA (H – O045).

10:45 MICROESTRUTURAL EVOLUTION OF ALUMINA-NIOBIA COMPOSITES ON SINTERING
J. P. Moreira, M. L. A. Graça, C. A. A.Cairo. IAE/AMR. Pça Mal. Eduardo Gomes, 50, Vila das Acácias, 12228-904, São Jose dos Campos, São Paulo, Brazil. W. Acchar. UFRN. E. M. J. A. Pallone. USF. K. F. Portella. LACTEC (H – O052).

11:00 SiC-SiC COMPOSITE FOR THERMAL PROTECTION SYSTEMS
M. Florian, L. E. Carvalho, K. Iha. ITA. Pça Mal. Eduardo Gomes, 50, Vila das Acácias, 12228-904, São Jose dos Campos, São Paulo, Brazil. M. L. A. Graça, C. A. A. Cairo. IAE/AMR (H – O030).

11:15 TO BE ANNOUNCED (H – O024).

11:30 SOFTENING OF THE TiO2 MEASURED BY ANELASTIC SPECTROSCOPY
C. R. Grandini, R. L. N. Oliveira. UNESP. Laboratório de Relaxações Anelásticas, 17.033-360, Bauru, SP, Brazil. R. P. Mota. UNESP/Guaratinguetá. F. P. Santos, E. Campos. EEAR (H – O049).

11:45 ANALYSIS OF THE PROPERTIES OF Si3N4-Al2O3-CTR2O3-AlN-CeO2 SYSTEM FOR ADVANCED APPLICATIONS
J. V. C. de Souza. FEG-UNESP. Av. Ariberto Pereira da Cunha, 333, cep. 12516-410, Guaratinguetá-SP. C. A. Kelly, M. R. V. Moreira, M. V. Ribeiro. DEMAR-FAENQUIL. M. A. Lanna. AMR-CTA (H – O047).

12:00 PROCESSING AND EVALUATION OF THE POROUS CERAMICS PROPERTIES FOR MICROFILTRATION
S. R.. Daniel, R. S. Vânia, V.C. Pandolfelli. UFSCAR. Edificio Alcoa, Dema, Rodovia Washington Luiz, Km 235, São Carlos, São Paulo, Brazil, CEP: 13.565-905 (H – O041).

12:15 DEVELOPED OF GRADED POROUS CERAMICS
T. G. Cruz, L. J. Silva, L. R. O. Hein. DMT - FEG – UNESP. Rua Ariberto Pereira da Cunha, 333, Pedregulho, Guaratinguetá, São Paulo, Brazil, CEP 12516-410 (H – O005).

12:25 LUNCH

Session Chair: Carlos. A. Paskocimas (UFRN)

14:00 Invited TO BE ANNOUNCED (H – I005).

14:45 DRYING ADDITIVES FOR REFRACTORY CASTABLES
R. Salomão, C. M. Peret, V. C. Pandolfelli. DEMa/UFSCar. Depto Engenharia de Materiais, Universidade Federal de São Carlos, km 235 São Carlos SP Brazil ZIP 13565-905 (H – O016).

15:00 PREPARATION AND CHARACTERIZATION OF CERAMIC TAPES USING POTASH FELDSPAR, SODIUM LGINATE AND POLY(ETHYLENEGLYCOL) IN AQUEOUS MEDIA
P. Lemes, M. Guiotoku, D. Hotza. UFSC. Departamento de Eng. Mecânica - LABMAT - Laboratório de Materiais, Campus Universitário, Trindade, Florianópolis-SC CEP 88040-900. A.P.N. Oliveira. CTCMat/Criciúma (H – O015).

15:15 A NEW METHOD TO DETERMINATE OPEN POROSITY AND PERMEABILITY CONSTANTS OF OPEN-PORE SINTERED GLASSES BY PHOTOACOUSTIC TECHNIQUE.
M. T. Yasuda. DEMa/UFSCar. Departamento de Engenharia de Materiais - DEMa, Rodovia Washington Luís (SP-310), Km 235, São Carlos, São Paulo - Brasil. CEP: 13565-905. W. L. B. Melo. Embrapa Instrumentação Agropecuária. (H – O021).

15:30 MICROSTRUCTURAL CHARACTERIZATION OF Bi2212 SUPERCONDUCTORS SAMPLES TREATED UNDER THE INFLUENCE OF AN APPLIED EXTERNAL MAGNETIC FIELD OF 5T
A. R. Bigansolli, D. Rodrigues Jr. FAENQUIL. Departamento de Engenharia de Materiais, Polo Urbo-industrial, Gleba AI-6, Lorena, SP, Brazil 12600-000. K. Watanabe, K. Takahashi. Tohoku University (H – O048).

15:45 MECHANICAL CHARACTERIZATION OF TiO2 CERAMICS FOR DENTAL APPLICATIONS
F.P. Santos, E. Campos, E. M. Marins, E. F. Lucena. EEAR. Escola de Especialistas de Aeronáutica, Av Brig. Adhemar lyrio s/n , Guaratinguetá, Brasil, 12500-000. C. R. Zacharias. UNESP. F. C. L. Melo. AMR/CTA. (H – O035).

15:55 COFFE-BREAK

16:20 POSTER SESSION

INVITED PRESENTATIONS

H – I001 POWDER METALLURGY PROCESSING OF TITANIUM ALLOYS: RELEVANT MICROSTRUCTURAL ASPECTS

H. R. Z. Sandim (FAENQUIL).

Titanium alloys are attractive materials for many engineering applications including the manufacture of parts for aircraft and automobile industries due to their superior properties such as high strength-to-weight ratio and outstanding corrosion resistance. Its outstanding biocompatibility is also essential to the development of the still-growing market of Ti-based surgical implants. Powder metallurgy (P/M) approaches allow the production of near-net-shape parts lowering their manufacture costs, a mandatory prerequisite to boost titanium industry worldwide. Furthermore, P/M processing of titanium and Ti-based alloys enables the obtainment of components having weak-to-absent textures, uniform grain structure, and higher homogeneity compared to conventional wrought products. In the present paper, the main aspects concerning the processing of titanium alloys by blended elemental (BE) and pre-alloyed (PA) powder methods are presented. Practical examples describing the microstructural development of two commercial alpha-beta alloys (BE-Ti-6Al-7Nb and BE-Ti-5Al-2.5Fe) and other titanium alloys already reported in the literature during P/M processing will be presented and discussed, with emphasis to the dissolution behavior of elemental powders, concurrent alloy formation, segregation, final porosity, and residual contamination with interstitial elements, in particular oxygen and nitrogen.

H – I002 MINERAL PROCESSING AND METALLIC PARTICULATE MATERIALS

U. U. Gomes (UFRN).

The natural resources of the mineral province of Seridó, Northeastern Brazil, include a wide variety of both ceramic and metallic industrial raw materials. Large reserves of kaolin, feldspar and mica as well as refractory metals including tantalum, niobium, tungsten and molybdenum are commonly encountered in the region. In this lecture, technological questions on the processing and sintering of both intermediate and final particulate materials from local minerals will be addressed. In addition, an update on the processing route of high-energy milled composites and nanoparticulate powders of refractory carbides and nitrides from organometallic precursors will also be presented. Finally, the production of ceramics, special devices and micrograined hard metals will also be discussed.

H – I003 SYNTHESIS OF ELECTROCERAMICS BY SOFT CHEMISTRY TECHNIQUES

E. N. S. Muccillo (IPEN).

Over the last fifty years a remarkable advance in materials science and engineering has been experienced. The emphasis on the relation between microstructure and properties has demonstrated to be fruitful. However, the performance of ceramic components in severe conditions is still far below the ideal limits predicted by theory. The ultimate goal in chemical processing of ceramic materials is to control physical and chemical variability to achieve desired properties. In this work, the role, advantages and limits in the use of soft chemistry techniques for the synthesis of ceramics are discussed, and recent results of experimental works on selected materials will be shown.

H – I004 INJECTION MOULDING OF CERAMIC AND METALLIC POWDERS

Rubens M. Nascimento (UFRN).

The injection molding of metallic and ceramic powders has been increasingly used in industrial applications. The technique merges characteristics of powder metallurgy and the geometric diversity of conventional thermoplastics injection, resulting in small intricate components with reduced density. From a general standpoint, 80% of the injected components do not exceed 100 cm3 and depict complex geometries. The process is particularly cost-effective in the production of large batches, as compared to conventional powder technology, machining and microfusion. Both scientific and technological advances have modernized the different stages of the process, contributing to an increase in the market of injected ceramic or metal alloy parts. In the scope of this lecture, an updated insight on new powder injection trends will be focused along with distinct aspects on the injection of ceramics and metal powders, particularly those concerning process parameters, binder selection and removal, and sintering.

H – I005 TO BE ANNOUNCED

ORAL PRESENTATIONS

H – O001

STRUCTURAL AND THERMAL PROPERTIES OF Co-Cu-Fe HYDROTALCITE STUDIED BY 57Fe MOSSBAUER SPECTROSCOPY AND X-RAY DIFFRACTION

D. X. Gouveia, A. G. S. Filho, J. A. C. Paiva, J. Mendes Filho. UFC, Depto de Fisica, Caixa Postal 6030, CEP 60455-900, Fortaleza, Ceara, Brasil; A. H. Iglesias, O. P. Ferreira, O. L. Alves. IQM-UNICAMP.

Layered double hydroxides (LDHs) or hydrotalcite-like compounds (HT) have been widely investigated owing to their potential applications such as ion exchangers, catalysts and catalyst precursors, pharmaceuticals, UV stabilizers, adsorbents, among others. In particular, Cu-based HTs and their decomposition products are known to be active catalysts for oxidation reactions and Cot-based HTs or the oxides obtained after thermal treatment are being continuously developed for their use in the Fischer-Tropsch process. In this work we report an investigation of the ternary (Co1-yCuy)1-xFex(OH)2(CO3)x.mH2O HTs compounds and their thermal decomposition behavior by using short (57Fe Mossbauer spectroscopy) and long-range order techniques. The main focus relies on the investigation of structural and thermal effects of Cu2+ addition (x-ray diffraction) to Co-Fe based HTs aiming to understand the system in order to make a rational use of these modified HTs and potentialize their applications. The Isomer Shift and Quadrupole Splitting values indicate that the probe atom is in the Fe3+ oxidation state with octahedral coordination. By changing the Cu content we observe that both Isomer Shift and Line width are nearly constant. However, the Quadrupolar Splitting values systematically increases as Cu concentration increases. This result tells us that the replacement of Co by Cu induces some distortion in the Fe3+ coordination sphere and this is understood based on the electronegativity of the

H – O002

A STUDY OF THE PHOTOLUMINESCENCE OF Li2TiSiO5 AND Na2TiSiO5 COMPOUNDS

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Some titanates, like for instance ATiO3 perovskites are known for a long time for presenting PL only at low temperatures. However, more recent studies about A2TiBO5 (A=Na, Li: B=Si, Ge) compounds, show that they do possess strong luminescent properties at room temperature. This phenonenon is somehow linked to the presence of pentacoordenated titanium in the crystal structure. The powders were synthesized by a Pechini-type method and analyzed by X ray diffraction and Raman spectroscopy. The photoluminescent properties were measured using excitation wavelengths of 250 and 488nm. Li2TiSiO5 powders do not present luminescent properties when the excitation wavelength is 250nm, but they do when structurally disordered and excited by the 488nm line. On the other hand, Na2TiSiO5 samples, in their crystalline or disordered forms present luminescence with the 250nm excitation although they do not when excited by the 488nm line. The obtained results suggest that the origin of the luminescent behavior lies on different mechanisms and also depends on the crystalline structure and on the excitation wavelength.

H – O003 FABRICATION PROCESS BY POWDER METALLURGY FOR BIOCOMPATIBLE Ti-13Nb-13Zr ALLOY IMPLANTS

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The perfect reconstitution of bone tissues after accidents or illnesses is one of the main challenges of modern science. The trend of the current research in orthopaedical implants is based in the development of titanium alloys with low modulus of elasticity, next to the bone, and aluminum and vanadium free. Among the titanium alloys recently developed, Ti-13Nb-13Zr is distinguished for presenting low modulus of elasticity, high mechanical strength and superior biocompatibility. Samples were produced by mixing of initial metallic powders followed by uniaxial and cold isostatic pressing with subsequent densification by sintering between 900 up 1500 °C, in vacuum. The alloy was characterized by means of scanning electron microscopy, X-ray diffraction, Vickers microhardness measurements and density. As expected, metallic phases surround niobium particles is observed leading to core-shell structures. The microstructure is typically from diffusion-controlled reaction sintering. The microstructure homogenization is improved with the dissolution of the elemental powders by the increase of the sintering temperature.

H – O004 INFLUENCE OF THE PROCESS CONTROL AGENT (PCA) IN THE MECHANICAL ACTIVATION OF Nb75Al POWDER MIXTURES

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NbAl3 intermetallic compound were synthesized by reacting Nb75Al powder mixtures, which were previously mechanically activated in a high-energy ball mill (SPEX®8000) for 1 hour. Different surfactants, known as PCAs (stearic acid, ethanol and methanol), were added to the powder charge in different proportions (1 and 2 wt.%) in order to control the welding and fracture events during the milling of ductile materials. Shape and microstructure of activated powders were analyzed by scanning electron microscopy. Milled powder were uniaxially pressed in cylindrical pellets that were further vacuum reacted at a constant heating rate (30ºC/min). It was found that the shape and the dispersion degree of the milled powders were strongly affected by the kind and quantity of PCAs, therefore changing the reaction behavior and the densification of the produced pellets.

H – O005 DEVELOPED OF GRADED POROUS CERAMICS

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This work proposes methods for production and characterization of graded porous ceramics, formed by successive slices of TiO2. The ceramics bodies were obtained with a novel route of manufacturing based on starch consolidation technique, which uses the starch properties as binding and pores former. TiO2 porous ceramics were manufactured with different number of slices, also changing starch nature and concentration in slurries. The porous ceramics were characterized through quantitative microscopy. Based in digital images obtained by light microscopy, it was noticed the adhesion between slices for all samples. Mean pore volume fraction, porosity distribution along samples and the pores size and shape have been determined. The different pore density and morphology along slices are witnesses of porosity gradient.

H –O006 INFLUENCE OF VANADIUM IN ZINC TITANATE MORPHOLOGIC (Zn2TiO4), STRUCTURAL AND PHOTOLUMINESCENT PROPERTIES

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In this work, the polymeric precursor method was used to obtain Zn2TiO4 doped with V+5. The power was obtained after calcination of the respective precursor powders at different temperatures, and their characterization was accomplished by X ray diffraction (XRD), infrared spectroscopy (IR) and thermal analysis (TG and DTA). It was difficult for vanadium to get into the polymeric net, leading to segregation in the resin and also in the final material. After doping, the thermal decomposition temperature decreased from 438 ºC to 420 ºC. The powders calcined at 400°C and 500°C presented a substitutional solid solution between Ti+4 and V+5 metallic ions, in addition to secondary phase formation. A decrease in the volume of the unit cell after addition of vanadium was observed. Relative crystallinity and crystallite size presented a randomic variation due to the difficult of vanadium substituting titanium. The best results of photoluminescent properties were obtained for the sample with 0.2 mol % of vanadium.

H – O007 PHOTOLUMINESCENCE IN DISORDERED RUDDLESDEN-POPPER HOMOLOGOUS SERIES

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Intense photoluminescence in highly disordered strontium titanate was observed at room temperature. The emission band maximum of the material is in the visible region and depends on the exciting wavelength.

Disordered strontium titanate particles were processed by the polymeric precursor method, which is based on the chelation of cations by citric acid. The titanium citrates were formed by the dissolution of titanium (IV) isopropoxide in a water solution of citric acid under constant stirring. Then, strontium carbonate was added to the solution. After homogenization of the solution, ethylene glycol was added to promote mixed citrate polymerization. The molar ratio between the strontium and titanium cations was 3:2 and the citric acid/ethylene glycol ratio was at 60/40 (mass ratio). After that, the gel was pre-dried at 350°C for 1h. Soon after, different thermal treatments were accomplished. X-ray diffraction analysis showed that, after calcination at low temperatures, an amorphous phase is formed. The photoluminescence spectra the strontium titanate present broad bands. The material maximum peak positions were around 550 nm. The photoluminescence intensity decreases with the increasing the calcination temperature. With the increase in the calcination temperature the system is organized and consequently photoluminescence decreases.

H –O008 COMPARISON OF TEXTURAL, STRUCTURAL AND MAGNETIC PROPERTIES OF NICKEL FERRITE PARTICULATE MATERIALS AND NANOCOMPOSITES

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Nanocrystalline NiFe2O4 particulate material were synthesized using metallic nitrates dispersed in aqueous media precipitated by stoichiometric amount of NH4OH and nanocomposites of NiFe2O4/SiO2 were obtained by sol-gel process from the mixture of tetraethylorthosilicate, TEOS, ethyl alcohol, water and metallic nitrates. The influence of heat treatment on the texture and magnetic properties of the prepared materials was studied. The specific surface area varied with the annealing temperature from 246 to 1 m2g-1 for the NiFe2O4 particulate materials and from 293 to 285 m2g-1 for the NiFe2O4/SiO2 nanocomposites. The nanocomposites presented isotherms characteristics of mesoporous materials in all calcination temperatures and the ferrite particulate materials presented isotherms characteristics of mesoporous materials only at calcination temperature above 700°C. The composites presented different magnetic behavior of the particulate material.

H – O009

STUDY ON CALCINATION IN SnO_2-MnO_2 SYSTEMS

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Tin dioxide based materials doped with 0.5-1.0 mol% manganese oxide were obtained by the physical method of oxide mixture and also by the chemical method of the polymeric precursors. Each composition was analyzed in respect to powder morphology after calcination by means of specific surface area using B.E.T method. Thus, adjustments in the calcination temperature and time can be made for the oxides obtained by chemical method equaling the same superficial conditions of oxides obtained by the physical method. In this way, it was found that the behavior of oxides obtained by the chemical method follows an equation of exponential decay. Such equation can be applied to the calcination of oxides obtained by polymeric precursors from 700°C.

H – O010

EQUILIBRIUM DIHEDRAL ANGLE DETERMINATION OF THE SnO_2 SINTERING

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The kinetic models usually used in the study of the sintered ceramic, are produced by means of indirect physical rehearsals, such as, the results obtained in the linear shrinkage and the mass loss, such fact can be justified due to the difficulty in the determinations of the intrinsic parameters of the ceramic materials along every sintering process. This way, the proposal of this work was to estimate, through statistical data, the evolution of the dihedral angle and to determine the equilibrium dihedral angle in the sintering SnO_2-MnO_2 system obtained by Pechini's method, the atomic force microscopy technique (AFM) was used.

H – O011

NIOBIUM SILICATE GLASSES PREPARED BY CHEMICAL PROCESS

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It's well known that vitreous systems containing Nb_2O_5 have been of great interest due to their structural versatility and useful applications. The insertion of Nb_2O_5 in silicate systems contributes to the development of new materials with higher non-linear optical coefficients and improved radiation optical properties and laser glasses of high-stimulated emission parameters. In this work systems based on composition $4.5SiO_2 – 3Al_2O_3 - X\ Nb_2O_5 – 2CaO$ with X from 0 to 1.5 were synthesized by chemical process Polymeric Precursor Method. This method allowed the preparation of niobium silicate systems at 700ºC/1h. The process of glass formation has been investigated by XRD and DTA. The XRD and DTA results confirmed that the prepared systems are glasses. The structures of systems prepared have been determined by FTIR and 29Si and 27Al MAS NMR. The FTIR results indicated a presence of groups NbO_6. These groups NbO_6 were included in the glass network forming Si-O-Nb bonds. 29Si and 27Al MAS NMR spectra showed that glass network is formed by SiO_4 and AlO_4 linked tetrahedra. Based on these results it is possible to consider that the structures of the systems are composed for a random network formed by SiO_4 and AlO_4 tetrathedra with NbO_6 octahedra.

H – O012 A MODIFIED MASS SPECTROMETRY DIAGNOSTIC TECHNIQUE FOR THE STUDY OF PLASMA-SURFACE INTERACTION WITH HIGH TEMPERATURE SURFACES APPLIED TO POWDER METALLURGY STUDIES.

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This article presents a technique implemented for application of mass spectrometry to surface treatment analysis of metallic materials by dc plasma (abnormal glow discharge) in powder metallurgy, as well as a general description referring to developed and published research which employ this technique. In the adopted configuration, a disk sample, used as cathode, is placed in the head of the quadrupole mass spectrometer probe, and can heated up to 600C by ion bombardment from the plasma process. The chemical species to be investigated during the treatment process are collected directly from the sample surface, through a conical orifice previously made in it. This new experimental arrangement permits the determination of some chemical species formed on the sample surface during the plasma treatment, as well as to correlate them to the micro-structure of the treated material surface. This experimental arrangement has been used to investigate decarburizing process of steels during plasma nitriding (N2-H2) and the plasma assisted nitrocarburizing process (N2-H2-CH4). The obtained results show the contribution of various species in the processes, such as CO2, CH3, CH4 and NH3, in addition to the investigation of formation mechanisms of some of these compounds and the correlation of these mechanisms to the chemical structure of the treated layer.

H – O013 ELECTRODEPOSTION OF CoPd ALLOYS FROM CHLORIDE BATH AND THEIR MAGNETIC PROPERTIES

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CoPd alloys have good mechanical properties, such as hardness, high corrosion resistance and consequently high durability. Also, their magnetic properties are of interest, specially in the magneto-optical recording media field. Electrodeposition is a very simple, inexpensive and versatile technique and has several advantages compared to other deposition technologies, in terms of costs and operating conditions. In this work CoPd alloys were obtained by electrodeposition using an aqueous Cl- bath. Two bath compositions were studied. The influence of solution pH and the current density on the alloy magnetic properties and deposit composition (estimated by EDS) was studied. It was observed that the deposit composition varies with the current density. The pH in the range 6.4-9.3 does not affect alloy composition. At pH 8.9, applying a high current density (100 mAcm-2), Co-rich alloys (80 at%.) were obtained, whilst the use of a low current density (8 mAcm-2), Co/Pd ratio was ~1. High coercivity as much as 1.38 kOe was achieved when a high current density (j = 250 mAcm-2) was used, indicating the formation of considerably hard magnetic materials. The influence of the additive additions in the magnetic properties of CoPd alloys is still under invetigation.

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H – O014 SURFACE CHARACTERIZATION OF PLASMA SINTERING UNALLOYED IRON ENRICHED WITH DEPOSITED NICKEL: CORROSION DEPENDENCE FROM POROSITY AND SURFACE COMPOSITION

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One of the largest limitations of materials achieved from ferrous powder metallurgy is their low corrosion resistance, due to the presence of pores. The plasma sintering thermal treatment is an alternative to improve the corrosion resistance. Two different treatments were used: the deposition of a nobler element as iron, in the surface of the material during sintering and the reduction of the superficial porosity by impact of energy particles, activating the atomic diffusion in the surface. The electrical discharge was generated in gaseous mixture of 80%Ar-20%H2 and the sintering was processed at 11500C during 60 minutes. Ni was deposited in the anode-cathode confined configuration, with the sample placed in the anode, in order to obtain the surface enrichment, since Ni is frequently used to reduction of corrosion. The reduction of the superficial porosity was obtained placing the sample in the cathode, where it suffers highly energetic collisions by ions and fast neutrals, which are strongly accelerated in the cathode sheath. The samples were characterized using electrochemical techniques. The results evidenced a reduction in the corrosion rate due to Ni surface enrichment, obtained during sintering, and due to the decreasing of superficial porosity.

H – O015 PREPARATION AND CHARACTERIZATION OF CERAMIC TAPES USING POTASH FELDSPAR, SODIUM LGINATE AND POLY(ETHYLENEGLYCOL) IN AQUEOUS MEDIA

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The tape casting is a technique widely used to produce flat thin ceramic substrates and multilayered structures, like capacitors and sensors, which are used mainly in the electronic industry. In this work, the rheological behavior of suspensions, properties and microstructures of the green and sintered tapes were studied for aqueous tape casting process. Aqueous slurries using potash feldspar raw material, sodium alginate as binder and poly(ethyleneglycol) as plasticizer were employed. The viscosity of the slurries were measured with respect to changes in the plasticizer and binder concentration. After casting and solvent evaporation, the green densities of the samples were measured. The sintered samples were evaluated to their density and shrinkage. The microstructure of green and sintered samples was evaluated by scanning electron microscopy.

H – O016 DRYING ADDITIVES FOR REFRACTORY CASTABLES

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Refractory castables are low permeability materials and, due to this, their drying process must be carried out carefully in order to avoid explosive spalling. It occurs when the pressure of the water vapor generated reaches the ultimate mechanical strength of the material. To reduce these risks, drying additives have been incorporated in the castables formulations. Their general role is to promote conditions which allow the castables to resist the spalling. Two additives were explored in the present work: aluminum powder and polymeric fibers. The first, known as a gas generator (H2), can increase the porosity of the castable at the same time the gas evolution removes part of the water content. On the other hand, polymeric fibers act as permeability magnifier, creating a net of permeable paths, as well as mechanic reinforcement. Aspects of the drying behavior of the castables and the modifications promoted by the drying additives were revised and a comparison between their performance in drying and the impact in processing was proposed.

H – O017 TO BE ANNOUNCED

H – O018 THE EFFECTS OF POWDER MILLING ON DENSIFICATION OF BORON CARBIDE

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Boron carbide (B4C) is one of the main advanced ceramic engineering materials for structural ends. It is a covalent solid with high melting point (2450oC), extremely hardness (2500Hv), high section of neutrons absorption and has low density (2,4 g/cm3).

Amongst such properties, it is recognized mainly for its extreme hardness and resistance to the abrasion, therefore it is the third harder material, only losing for diamond and boron nitride. Its low density allied to high hardness and modulus of elasticity indicates this material to be used in lightweight armor for military helicopters, airplanes and personal protection. The aim of this work is to obtain densified pieces (over 94%) of boron carbide, sintered at lower temperatures (<2000oC). Normally, sintering temperature is over 2200oC. The difficulties to process this material arises from the covalent bonds and it demands hot isostatic pressing, in inert atmospheres with high tempeature., which is too expensive.

This work presents the result of milling B4C powders in a planetary mill, using steel vessels coated with WC, using zirconium spheres and alcohol as milling agents. The technique used to determine grain size distribution is the LASER difraction and the reactivity measurement uses BET technique. The density, hardness, chemical composition and microstructural characterization of the sintered pieces are also presented.

H – O019 STUDY OF Ni-Al INTERMETALLIC SYNTHESIS MECHANISMS BY TEMPERATURE EVALUATION DURING HIGH ENERGY MILLING

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Ni3Al and NiAl intermetallic compounds in the Ni-Al system were synthesized by mechanical alloying of elemental powder blends in a Spex type mill. Two compositions were used: (75%Ni-25%Al and 50%Ni-50%Al, atomic percentage), in order to produce NiAl and Ni3Al compounds. The milling was carried out in inert atmosphere with a 1:6 powder–to-ball ratio. Measurements of jar temperature during powder mixture processing were done in order to evaluate the two mechanisms of alloying showed by the literature. The formation of Ni3Al and NiAl was examined at different milling times using x-ray diffraction analysis (XRD). MA of elemental blend of Ni50Al50 composition showed a temperature peak around 60min of milling and it indicates the occurrence of a self–propagating reaction and discontinuous additive mode mechanism. On the other hand, no peak was observed during processing of Ni75Al25 composition, indicating a continuous diffusive mode of alloying during the formation of Ni3Al intermetallic compound.

H – O020 TUNGSTEN OXIDE REDUCTION BY METALLIC ALUMINIUM USING HIGH ENERGY MILLING

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High energy ball milling has been used like a new route for processing of materials. In the present paper, the reduction of tungsten oxide by aluminum in order to obtain metallic tungsten was studied using a SPEX type high energy mill. A powdered mixture of WO3 and metallic aluminum weighed, according to the stoichiometric proportion with an excess 10% Al, was processed with hardened steel utensils using a 1:6 powder-to-ball ratio. The processing was carried out with milling jar temperature measurement in order to detect the reaction type. The temperature evaluation indicated the self-propagating reaction occurrence by fast increase of the jar temperature after a short milling time. The tungsten oxide reduction was verified by x-ray diffraction (XDR) analysis and the milling products were characterized by scanning electron microscopy (SEM). The results were slightly different from the literature due the mill type and milling parameters used in this work.

H – O021 A NEW METHOD TO DETERMINATE OPEN POROSITY AND PERMEABILITY CONSTANTS OF OPEN-PORE SINTERED GLASSES BY PHOTOACOUSTIC TECHNIQUE.

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This work deals with a new method to determine the open porosity of the open-pore sintered glasses (filters) based in the photoacoustic (PA) technique. In the PA technique, a modulated beam of light illuminates the sample generating acoustic waves, detected by a microphone inside a PA cell. In the filters, the light beam is partially absorbed and diffusely transmitted. The transmitted light reaches a support where the filter is fixed. In this method, two supports were used: an absorber and a reflector. The relation (R) between the PA signals of the filters, in different supports, gives informations about open porosity and permeability. In order to demonstrate this method, filters had been produced with different porosities through soda lime glass powder and by filler principle, resulting in a set of filters with the following calculated volumetric porosities: 0; 15,1; 28,9; 41,0; 52,6%. The results obtained by this method had been compared with the results of permeability constants by decay pressure technique. The relation between the PA signals versus the opened porosity was adjusted by R/mass=R0/mass+s(eqe–1), where: R0, is the relation R for the sample without pores; s, depends on the surface roughness; q, is a constant of opened porosity and e, open porosity. The results demonstrated that new PA method is able to characterize opened porosity and surface roughness of the filters, making possible to determine their permeability constants. Support: Embrapa, Cnpq, FAPESP.

H – O022 PREPARATION OF ZIRCON PIGMENTS FROM THE ZrSiO4-Co3O4 SYSTEM

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New Co-doped ZrSiO4 based pigments were obtained in this work using conventional ceramic method. The ZrSiO4 samples were doped with different concentration of Co3O4 and calcined for one hour at three differents temperatures: 1150ºC; 1250ºC and 1350ºC. The pigments obtained were characterized using X-Ray Diffraction (XRD) and UV-Visible. Phase analysis by XRD in the investigated system indicated only the one crystalline phase, corresponding to ZrSiO4. The colorimetric coordinates on CIELab method were obtained by reflectance measuring in the UV-visible region. The results shown changes from light blue to dark blue when Co3O4 were added.

H – O023 ELETROCHEMISTRY HYDROGENATION OF Nd2Fe17 AND Nd2Fe17Ga1,5

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The R2Fe17 compounds and their interstitial with hydrogen and nitrogen are good candidates for permanent magnets. Beside high values of the Curie temperatures, saturation magnetization and anisotropy field, the technical application of these materials is limited by their poor thermal stability. Partially substituting Fe by Ga the phase can be stabilised up to temperatures above 1200K. The utilisation of the hydrogen disproportionation desorption recombination process (HDDR) is an effective way of obtaining particulate materials, and consequently high coercive magnets. Unfortunately, the substitution of Fe by Ga, also stabilise others compounds against disproportional by hydrogen. Thus, the improvement of hydrogen treatment of R2Fe17pure phase and with Ga are very necessary. Until now the most of the hydrogen treatment were done at high temperatures and in high hydrogen pressure. However, these conditions, how we describe above, can facilitate the formation of new no desire phase and also oxides. The gas hydrogenation require expensive and complicate equipament. In contrast, eletrochemistry methods are very simple, not expensive and can be performed at room temperatures. We use of electrochemistry method to hydrogenation of metallic alloy Nd2Fe17 and Nd2Fe17Ga1,5. The pure and electrochemistry hydrogenated samples were characterized by Moessbauer spectroscopy and X-Ray Diffraction. Observed similarities and differences with gas hydrogenation are discussed.

H – O024 TO BE ANNOUNCED

H – O025 IN-SITU OBSERVATION OF GLASS PARTICLE SINTERING

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This work introduces the sintering process to students unfamiliar with the subject, showing the viscous flow sintering process of glass particles. Frenkel’s model, which describes the viscous flow process, was applied to demonstrate its validity. Spherical soda-lime glass particles were heat treated at 750oC in an optical microscope equipped with a hot stage. The glass sintering process was recorded using a digital camera coupled to the microscope. During sintering, the particle boundaries moved apart until the interface separating them was flat, subsequently joining to become a single particle as a result of the decrease in superficial free energy. Frenkel’s model was verified for the vitrification process and for ceramic shrinkage during sintering and the two graphs indicate experimental measurements so similar to those in Frenkel’s model that the linear correlation coefficient (R2) was approximately 0.97.

H – O026 CONTRIBUTION TO THE KINETICS OF ZINC FERRITE FORMATION

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The present work includes a kinetic study of the formation of zinc ferrite from an equimolar mixture of pure iron oxide Fe2O3 and pure zinc oxide ZnO, focusing the fact that this substance is often the major constituent in the electric arc furnace dust.

Initially an equimolar mixture was characterized thermally (DTA-TGA) and structurally (XRD). The temperature in which the zinc ferrite began its formation and the quantitative results regarding the synthesis conversion of this compound, were obtained by the software Topas 2.1 Difracc Plus, using the Reitveld XRD method. In this work the following major experimental results were obtained: at low temperatures (650-730 oC) the synthesis phenomena fitted the interface reaction model, or topochemical model, being the chemical reaction the rate control mechanism. The obtained data in this case was: Ea = 65.6 kcal mol -1 and = 2.32 x 10-3 K-1. At high temperatures (750-1000 oC), the modified population growth formalism showed the best fit, being the diffusional mechanism the rate controlling process. In this case the obtained data was: Ea = 16.1 kcal mol -1 and = 570 K-1. Finally, the transition between the two mechanisms was found to happen at approximate 744 oC (mixed control).

H – O027

EFFECT OF B4C, C AND ZrO2 ADDITIVES ON PRESSURELESS SINTERING OF SILICON CARBIDE

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Silicon carbide has been used as a structural ceramic material for high performance. This material combines unique properties, such as oxidation resistance, high wear resistance, low density, high hardness, excellent thermal shock and the maintenance of the most of these properties in high temperatures. However, due to the highly covalent bonding between Si and C atoms, silicon carbide presents a complex and difficult sintering. Specific additives or hot pressing have been used to achieve near full density. In this study, B4C, C and ZrO2 were used as sintering additives in the pressureless sintering of silicon carbide. The admixtures were homogenized using polyethylene bottle and isopropanol media in a ball mill. Sintering was performed in a graphite resistance furnace at 2050 oC for 30 min in a flowing helium atmosphere. Relative density of sintered compacts was determined by the Archimedes method. The phase identification of the starting powder and sintered compacts was investigate by X-ray diffraction and the microstructures were observed by optical microscopy and scanning electronic microscopy. Acknowledgments: CAPES/IPqM/NUCAT

H – O028

KINETICS OF THERMAL DECOMPOSITION OF TITANIUM HYDRIDE POWDER USING IN SITU HIGH-TEMPERATURE X-RAY DIFFRACTION (HTXRD)

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The thermal decomposition of titanium hydride powder (delta-phase) to titanium (alpha-phase) was investigated by means of thermogravimetry analysis (TGA) and high-temperature X-ray diffraction (HTXRD) in high vacuum. The delta-to-alpha phase transformation was followed in situ by HTXRD at temperatures varying from room temperature up to 1000°C. The transformation was analyzed as a function of time with aid of experiments performed at isothermal conditions from 450 to 650oC. The results of TGA show that the decomposition of the titanium hydride becomes significant at about 450°C. Above 500°C the decomposition is completed in times shorter than 50 minutes. The apparent activation energy for hydrogen desorption was found to be 64 ± 6 kJ.mole-1.

H – O029

MECHANICAL PROPERTIES OF Ce-TZP CERAMICS OBTAINED FROM FREEZE-DRIED POWDER MIXTURES

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Chemical methods are frequently used to prepare ultrafine, homogeneous and high sinterable powders. One of most critical steps in these methods is the drying of the precipitated. The initial aggregates can be transformed in hard agglomerates during this processing stage. Though it may be difficult to avoid aggregration entirely, there is some control that can be achivied over the agglomeration state after drying step. In this work, freeze-drying or lyofilization technique was used to dry ceramic suspensions in a gentle way, avoiding agglomeration of the powder particles. In this technique the particle aqueous suspension is rapidly frozen and the water is removed by vaccum sublimation. The influence of freeze-drying process on the physical characteristics of the CeO2-ZrO2 powder mixture is discussed, as well as on the microstructural characteristics, on tetragonal-to-monoclinic stress induced transformation and, finally on some mechanical parameters of the Ce-TZP ceramics (as bending strength, Vickers hardness and fracture toughness). Powders with different CeO2 content were prepared by freeze-drying technique and conventional drying of Ce and Zr coprecipitated hydroxides from initial chloride aqueous solutions. The powder characteristics showed an adequate compaction and sintering behavior. The mechanical parameters reached superior values when they are compared to those corresponding ceramic fabricated from coprecipitated powder mixtures dried by the conventional technique.

H – O030 SiC-SiC COMPOSITE FOR THERMAL PROTECTION SYSTEMS

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Composites based on silicon carbide are potential candidate materials for thermal protection systems due to mainly its good thermal conductivity in fiber direction and very low through-the-thickness (transverse) thermal conductivity, high hardness, corrosion and thermal resistance. SiC-SiC composite like a SiC matrix reinforced with SiC polycrystalline continuous fiber were obtained by conversion reactions in high temperature and controlled atmosphere, from a carbon/carbon composite precursor. The CVR process were used to fabricate SiC/SiC composite with crystalline high-purity β-SiC from a carbon-carbon precursor fabricated with non stabilized carbon fiber and carbon matrix derived from phenolic resin. The converted composite exhibited a bulk density of 1,65 g/cc, 40% open porosity and flexural strength of 80 MPa measured by four-point bending testing. The total specific area was evaluated by BET technique and the thermal diffusivity was measured using the laser flash method. Thermal conductivity was calculated as a product of density, thermal diffusivity and heat capacity.

H – O031 THE APPLICATION OF CLASSIC MODELS OF INITIAL STAGE OF SINTERING

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Tin oxide ceramics have been large studied due its electro-electronics properties. In spite of the research in the last years, definitive theory of this material sintering still has not been reached. This work was developed comparing two different kinetic models for the initial phase of sintering, Woolfrey/Bannister and Arrhenius, in SnO2 compacts doped with ZnO and obtained by physical mixture of oxides. Preparing 0.6, 1.0 and 1.5 (% mol) ZnO concentrations and sintering in dilatometer from the temperature of 25ºC up to 1365ºC, varying the constant heating rates. Using the MEV, the variance in grain size has been determined, when concentration and heating rates varied, obtaining larger grains in compacts with bigger concentration of dopant and smaller heating rate. However, the activation energy was reduced by the ZnO concentration increase.

H – O032 TO BE ANNOUNCED

H – O033 MICROSTRUCTURAL EVOLUTION DURING LIQUID PHASE SINTERING OF ALN WITH CaCO3 AND CAO ADDITIONS

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In this work, the effects of CaO addition in the microstructural evolution during sintering of an AlN powder were investigated. A commercial AlN powder was pressureless sintered with 0,5 to 8% CaO (as CaCO3 or CaO) in nitrogen atmosphere between 1100 and 2000°C for 1 h. The results of X-ray diffraction analysis showed formation of calcium aluminates as second phases. Each composition of additive resulted in a different phase evolution with sintering temperature, which was discussed in terms of the mass loss, CaO content (determined by chemical analysis), and phase stability. The samples with second phases rich in CaO showed lower starting densification temperature in accordance to the lower eutectic temperatures predicted from Al2O3-CaO phase diagram. SEM analysis showed that sintered samples presented large pores, which fraction was proportional to the added CaCO3 or CaO content. These pores were formed during the sintering, in the places occupied originally by the large CaCO3 or CaO particles. Although the increased amount of CaCO3 or CaO has favored the densification of AlN particles by lowering the densification temperature and forming higher amount of liquid phase, it also caused the formation of higher fraction of large pores, which retarded the bulk densification. As a consequence, the relative density showed a maximum in the intermediate range of CaO content. The grain size of the high temperature sintered samples scaled inversely with the added CaO content.

H – O034 POROUS CERAMICS FORMING BY “SOYA PROTEIN CONSOLIDATION”

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Porous ceramics have great potential in many applications in several areas. In this context, the pore structure in the ceramic may vary depending on the type of application. The porosity in terms of volume fraction, size and geometry is important to obtain essential properties in the final product. Therefore, it is extremely interesting to obtain and characterize the porous ceramic with different morphologic characteristics and specific applications. In this work, ceramic of TiO2 was manufactured through the “direct-consolidation” technique using soya protein. The ceramic pores characterization was made through a quantitative analysis using the digital images processing. Several TiO2 porous ceramic were manufactured through the percentage variation of soya protein. The medium porosity, pore medium size and pore size distribution were analyzed statistically. The morphology and the interconnecting pores were analyzed using the Depth from focus. The proposed methodology is viable and reliable in the determination of the parameters related with the porosity, being adapted for analysis of large pores (closed and / or interconnected). The ceramics formed by protein consolidation presented great pores, in your majority isolated and / or agglomerates.

H – O035 MECHANICAL CHARACTERIZATION OF TiO2 CERAMICS FOR DENTAL APPLICATIONS

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The development of biocompatible ceramics date back to century XIX and nowadays, this field of investigation still attracts researches from different areas. The most common applications refer to prosthetics hip joints, pins and so forth to repair damaged bones or teeth. The work here presented deals with the potential application of titanium dioxide (TiO2) ceramics, as a biomaterial for dental repairs. The ceramics were produced through commercial starch consolidation. This technique permits the development of samples with complex shapes, allied with relatively low production costs. The mechanical characterizations were based on ASTM norm, concerning resistance, porosity, apparent density, roughness and surface wettability. In vitro biocompatibility tests were carried out using cultures of B. Streptococcus mutans. A very interesting property of titanium dioxide ceramics is its bactericidal action when photoactivated by UV radiation. The mechanical and biological results confirm the potential application of TiO2 ceramics as dental prosthetics, stimulating more researches on this area.

H – O036 MICROSTRUCTURAL CHARACTERISATION AFTER HEAT TREATMENT OF SINTERED VALVE SEAT INSERTS

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This work presents aspect related to sintered high-speed steels for valve seat inserts application. Six types of materials were evaluated. The materials were made on purpose with high-speed steel AISI M3 mixed with iron powder and carbides. The microstructures of the high-speed steels before and after heat treatment are presented. The results indicate that these materials under development have potential for commercial application.

H – O037 MICROSTRUCTURE OF Ti-35Nb-7Zr-5Ta ALLOY PRODUCED FROM HYDRIDE POWDERS

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Ti-35Nb-7Zr-5Ta alloy, which has a low modulus of elasticity (55 GPa), can be considered as the best one material to be used as a medical implant. The alloy processing by powder metallurgy eases the obtainment of parts with complex geometry and probably, cheaper. The Ti-35Nb-7Zr-5Ta samples were produced by the sintering of the elementary powders, in vacuum, between 900-1200 ºC, with heating rate of 20 ºC/min. All the powders were obtained by HDH process and used in the hydride form, aiming at the reduction of costs and increase of the sintering rate. The objective of this work is the analysis of alloy microstructural evolution from the powders dissolution under the increase of the sintering temperature. For the alloy microstructural characterization, scanning electron microscopy and Vickers microhardness measurements, were used. Density was measured by Archimedes method. In this work an alternative blend (with planetary mill) method was used. The results shows that the blended elemental P/M process is efficient for the alloy production. The samples presented a good densification and adequate microstructure, with complete dissolution of alloying elements in the titanium matrix with the temperature increase.

H – O038 REDUCTION-DIFFUSION PREPARATION OF Nd15Fe77B8, NdFe11Ti, NdFe10.5Mo1.5 AND NdFe10.75Mo1.25 ALLOYS FOR MAGNETS

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The calciothermic reduction-diffusion (CRD) process is a alternative preparation route for Nd15Fe77B8, NdFe11Ti, NdFe10.5Mo1.5 and NdFe10.75Mo1.25 alloys, which eliminates the need for long homogenizing heat treatment; in addition, the resulting alloy is already in powder form. We have examined the effect of various processing variables in the preparation of mother alloys. Compacts made of NdCl3, Fe, Ti, Mo and Fe-B powders and Ca granules were heated under argon for different times and temperatures.

The NdFe11Ti, NdFe10.5Mo1.5 and NdFe10.75Mo1.25 alloys can absorb substantial amounts of nitrogen, producing a potentially suitable for permanents magnets with high uniaxial anisotropy. These compounds have been successfully nitrogenated by heating a mixture of powdered alloys with sodium azide (NaN3) at temperatures between 330 and 450 oC. Structural and magnetic characterization yielded the following results: (a) the ThMn12 structure of the original compounds is retained with the addition of nitrogen by chemical reaction with NaN3; (b) the Curie temperature is enhanced by about 300 oC; (b) particle size is found to be an important parameter for efficient nitrogenation of Mo compound.

H – O039 SYNTHESIS AND STRUCTURE OF THE CRYSTALLINE Li2Zn1-xNixTi3O8

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The development of nanostructured materials has been very active in the last years. This work deals with the preparation of nanometric particles of crystalline Li2ZnTi3O8 powders doped with nickel using the polymeric precursor method. This approach is based on the chelation of cations (metals) by citric acid in aqueous solution. The citrate solution was mixed with ethylene glycol to promote polymerization by polyesterification. This reaction occurred at temperatures ranging from 90 to 1200 C after water had been eliminated. The polymeric precursor for the synthesis of amorphous Li2Zn1-XNiXTi3O8 doped with 10, 20 or 30% nickel was calcinated at 3000 C for 2h in order to promote e pyrolysis. The powder obtained was characterized by thermogravimetric analysis (TGA), X-ray diffraction (XRD), FTIR spectroscopy, MEV and the refinement was carried out by Rietveld method. Microstructural analysis revealed the presence of crystalline phase on powders calcinated.The Rietveld refinament Showed that the system is mono phase.

H – O040 PLASMA DEBINDING AND SINTERING OF PARTS PRODUCED BY POWDER INJECTION MOULDING (PIM)

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This work presents the characterization of an abnormal glow discharge and a debinding cycle, followed by sintering of PIM parts, using a pilot plasma reactor. Plasma debinding is an alternative process to either thermal or catalytic processes, exhibiting a short processing time, elimination of organic wastes as well as an environment clean operation. The process performance is related to the thermal set condition and to glow discharge parameters, such as biased voltage, ratio between the time switched on (ton) and total time, as well as gas pressure, atmosphere. In this work several cycles were carried out varying the following parameters: ratio ton/total time (25, 50, 75 and 92%), pressure (1 to 4Torr) and the biased voltage applied to cathode (440 and 540V). The plasma current and temperature were assessed to the thermal equilibrium condition of the chamber, hence permitting to choose conveniently the parameters of plasma for the debinding step. The results show that using a biased voltage of 440V, a pressure of 2Torr and ton ranging from 15 a 92% the accomplishment of a suitable debinding cycle is obtained. Such conditions were then used in a debinding and sintering cycle, in which AISI 316L steel PIM spheres were used as samples. It was shown that under such conditions the debinding was completed without any residues in the processing chamber and the resulted sintered parts showed parameters such as densities and dimensions in agreement to the industrial specifications.

H – O041 PROCESSING AND EVALUATION OF THE POROUS CERAMICS PROPERTIES FOR MICROFILTRATION

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Porous ceramics are applied as tecnologicamente collecting of solid, isolating particles thermal, acoustic and microfiltration of liquids and emulsions. The objective of this work understands two stages. The first one after understands the production of porous ceramics by colloidal processing and the evaluation of its final properties the burning. The second stage consists of the analysis of the performance of these ceramics During the fuel microfiltration. The porosity of these ceramic bodies meets in the 48% band 65%, the distribution of size of pores of the same ones disclosed the presence of micropores (1μm) and, its constants of Darcy permeability (k1) and non-Darciana (k2) meet in the 10-15 band m2 and 10-14 m, respectively. These properties are similar to the commercial products imported, therefore, it has a great potential of application in microfiltration processes. The carried through tests of microfiltration in the laboratory of Fluid Thermodynamics of the EESC-USP had confirmed the good performance of these porous ceramics.

H – O042 PRODUCTION OF TI-35Nb BY POWDER METALLURGY TECHNIQUES

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Titanium alloys have several advantages over ferrous and non-ferrous metallic materials, such as high strength-to-weight ratio and excellent corrosion resistance. A blended elemental titanium powder metallurgy process has been developed to offer low cost commercial products. Among the advantages of this approach is the higher dimensional precision and low material loss during parts production. In this work, results of the Ti-35Nb alloy production are presented. Due to its high biocompatibility and lower modulus of elasticity this alloy is a promising candidate for surgical implants fabrication. Samples were produced by mixing of initial metallic powders followed by uniaxial and cold isostatic pressing with subsequent densification by sintering between 900 up 1600 °C, in vacuum. Sintering behavior was studied by means of dilatometry. Sintered samples were characterized for phase composition, microstructure and microhardness by X-ray diffraction, scanning electron microscopy and Vickers indentation, respectively. Density was measured by Archimedes method. It was shown that the samples were sintered to high densities and presented homogeneous microstructure. Processing parameters were optimized in order to reduce the interstitial pick-up (O, C, N and H) and to minimize grain growth during sintering.

H – O043 MAGNETIC PROPERTIES OF Gd5Ge2Si2Hx ALLOYS, x  0.1

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Recently a giant magnetic caloric effect was discovered in Gd5(SixGe1-x) alloys x  5, which make them potential candidates for use in magnetic refrigeration in the range of 20 - 276K. Since then many research groups around the world have studied the magnetic properties of those alloys. In this work the magnetic transition temperatures and the magnetic moment of the Gd5Ge2Si2Hx alloys with x  0.1 are investigated by thermomagnetic analysis from 2K to 370K. The specimens were prepared by arc melt, treated at 1873K/48h and then hand ground carefully. Particles with diameter  0.35 mm were heated at 673K under hydrogen atmosphere in a SIEVERTS apparatus. The control of hydrogen absorption was made by monitoring of pressure changes in the reactor chamber. The M(T) curves show a first order magnetic transition at 260K only for the sample with x = 0.1. All the samples presented the second order magnetic transition between 296K (x=0.1) and 212K (x=2.5). For the samples with x  1.2 was also observed a new antiferromagnetic transition at 40K. The M(H) curves revealed that the magnetic saturation (Ms) increase with the hydrogen amount into the alloy. These results prove that the presence of interstitials into the Gd-Ge-Si based alloys can cause substantial changes on their microstructures, and also on their magnetocaloric effect. Work supported by FAPESP, UEM and CNPq.

H – O044 COMBUSTION SYNTHESIS OF NbAl3 MECHANICALLY ACTIVATED IN SHAKE AND PLANETARY BALL MILLS

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Combustion synthesis from elemental powder mixtures is severely affected by mechanical activation carried out in high-energy ball mills. In this work, Nb75Al powder mixtures (for NbAl3 synthesis) were mechanically activated in shake and planetary mills. The milling time and the ball to powder volume ratio were fixed. Balls and vials of steel and tungsten carbide were tested. Milled powder particles characteristics (shape and microstructure), ignition and combustion temperatures, density and microstructure of reacted NbAl3 pellets were compared. Significant differences between the activated powders from both mills resulted in distinct reaction behaviors (e.g. ignition and combustion temperatures). Mechanical activation was higher in the shake mill.

H – O045 MECHANICAL BEHAVIOR OF THE CARBON CARBON COMPOSITE COATED WITH CERAMIC CARBIDES BY CVR PROCESS

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Functionally gradient ceramic coating provides an effective antioxidant protection to carbon carbon composites at intermediate temperatures (Below 1000oC) by a self healing mechanism. A protection layer based on SiC-B4C compositional gradient was obtained by conversion of the superficial carbon of the composite using CVR process. By using this technique, the superficial carbon is converted without cracks or defects at the substrate/coating interface. In this work, strength and modulus properties were measured by four-point bend testing after the conversion of the 2D carbon carbon composite in a ceramic coating. Samples in as coated and oxidized at 800oC in air conditions were tested. These mechanical properties are compared with microestrutural characteristics of the surface fracture. The influence of the converted surface and integrity of the substrate coating interface of the composite on the variations in mechanical properties is discussed.

H – O046 INFLUENCE OF COBALT ADDITION IN THE PHASE TRANSITION ZrO2 TO ZrSiO4 OBTAINED BY MEANS OF THE POLYMERIC PRECURSORS METHOD

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Co-doped ZrSiO4 redish powders have been synthesized by the polymeric precursors method. A study of crystalline phases evolution of obtained powders was accomplished through X-Ray Diffraction and UV-vis spectroscopy. The obtained results allow to identify the ZrO2 to ZrSiO4 phase transition. The single-phase ZrSiO4 powder was obtained after heat treatment at 1100°C for 2 h. Besides, this phase transition is related with the color effect in function of the cromophore cation concentration and calcination temperature. Data of colorimetric coordinates reveals that the powders in which cobalt has been added remain blue in color. The powders turn red shades according to the transition to ZrSiO4. This fact can be explained by the change in the crystal field of cobalt, dislocating from tetrahedral to octahedral sites.

H – O047 ANALYSIS OF THE PROPERTIES OF Si3N4-Al2O3-CTR2O3-AlN-CeO2 SYSTEM FOR ADVANCED APPLICATIONS

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Due to its excellent property, silicon nitride (Si3N4) ceramics has been shown great interest as engineering material. This work was proposed with objective of to analyze Si3N4 additived with CTR2O3, AlN, Ce2O3 ceramics. However, a powder mixture constituted by 77.68 wt % Si3N4, 11.34 wt % AlN, 2.26 wt % Al2O3, 5.20 wt % CTR2O3 and 2.26 % CeO2 was homogenized in ethanol, dry at evaporator rotative and kiln, respectively. Then, uniaxially (100 MPa) and cold isostatically (300 MPa) pressed. The samples were sintered at 1800 e 1900oC for 1h, in graphite element resistive furnace under nitrogen atmosphere. After sintering, samples showed relative density higher to 96 % of theoretical, fracture toughness and hardness higher to 16.4 GPa and 5.72 MPa.m1/2, respectively. The phase analysis by X-ray diffraction and microscopy electron scanning (SEM) showed the presence of a-SiAlON and b-Si3N4. The obtained results generated economical and technological expectations with relationship to the application of liquid phase sintering Si3N4.

H – O048 MICROSTRUCTURAL CHARACTERIZATION OF Bi2212 SUPERCONDUCTORS SAMPLES TREATED UNDER THE INFLUENCE OF AN APPLIED EXTERNAL MAGNETIC FIELD OF 5T

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The use of superconductors of high critical temperatures in applied superconductivity leads to higher operation temperatures and economy of cryogenic fluids. High temperature superconductor materials exhibits limited transport properties due to grain boundary weak-links and anisotropy on the critical currents. The texturization development in these superconductors decreases in an efficient way the number of high-angle grain boundaries, increasing the values of Jc. Besides, the low orientation among the grains, the low density and the presence of non-superconducting residual phases are obstacles to obtain high Jc. In the present work, the starting material was a commercial high-Tc superconductor precursor powder with nominal composition Bi2Sr2CaCu2O8+ (Bi2212) (UniQuest Ltd.). The powder was pressed into pellets of 10mm in diameter and 5mm in thickness under 250 MPa uniaxial pressure. The pellets were heat treated in a vertical tube furnace under pure O2 flow. The vertical tube furnace operates inside a 7T superconducting magnet that works without cryogenic liquids (cryogen-free superconducting magnet). After heat treatment, the samples were microscopically characterized using XRD and SEM, with the purpose to evaluate the influence of an applied external magnetic field of 5T into the texturization process of Bi2212 samples. The critical temperature and the critical current as a function of applied magnetic field were also measured for all samples. Financial Support by FAPESP.

H – O049 SOFTENING OF THE TiO2 MEASURED BY ANELASTIC SPECTROSCOPY

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Titanium dioxide (TiO2) is a material that finds several applications as for example, filters and as a biocompatible material. Its good inertia chemical, low density and good relationship mechanical resistance/density, besides certain porosity levels, turn the TiO2 an option of biocompatible material used in medical or deontological implant. The oxides TiO2 present different characteristics depending as they are produced. The anelastic spectroscopy technique is a great option to study the mechanical properties of this material, because through her we can obtain information regarding the elasticity modulus. We can still obtain, information on the different atomic jumps that can happen in different structural phases. The samples were produced in the Faculdade de Engenharia de Guaratinguetá (UNESP) and they were consolidated using commercial corn starch, with different concentrations of the corn starch. The anelastic relaxation measurements were made using a torsion pendulum operating with frequency around 40 Hz in a temperature interval between 77 and 700 K with heating rate of approximately 1K/min and vacuum better than 10-5 mBar. The results shown that there was a softening of the material when we increases the amount of the corn starch, that give origin to larger pores. It was still observed, a complex relaxation structure that can be associated to the oxygen loss during the heating. (Financial support: CNPq, FAPESP and FUNDUNESP).

H – O050 TO BE ANNOUNCED

H – O051 MICROSTRUCUTURAL DEVELOPMENT OF PLASMA SINTERED NbC- AND TaC-REINFORCED STEEL

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Sintered steel was reinforced by niobium or tantalum carbide. Composite powder mixtures were milled, annealed, uniaxially pressed and plasma sintered at different temperatures and times. Compositions containing 20 wt.% NbC or TaC and either a pre-alloyed steel powder or a liquid phase additive (Fe3P) were sintered in order to study the effect of liquid phase formation in plasma sintering. The hollow cathode configuration was employed and sintering temperatures varied from 750°C to 900°C. The density and microstructure of the sintered composites were evaluated. Correlations between sintering paramerets, nature of starting powders and the resulting microstructure were established. Composite mixtures were plasma sintered to more than 96% of the theoretical density of the material by adjusting not only the sintering temperature and soaking time, but also the heating rate (10 - 100°C/min). Microstructures containing homogeneously distributed carbide particles were obtained by solid state sintering using heating rates of ~10°C/min. Plasma sintering assisted by liquid phase resulted in substantial reaction of Fe3P and consequent formation of liquid phase near the surface of the material due to intensive bombardment and localized heating. The liquid flows towards the bulk due to capillary effect inducing further melting and dragging carbide particles which end up agglometared at the grain boundaries of the ferritic matrix.

H – O052 MICROESTRUTURAL EVOLUTION OF ALUMINA-NIOBIA COMPOSITES ON SINTERING

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High alumina ceramics has been sintered at 1400oC to 98% theoretical density and 330-350 MPa mechanical strength (measured by four-point bend test) through Nb2O5 addition like a sintering promoter. However it requires using of further additives as MgO to achieve grain growth control and SiO2 to promote the formation of AlNbO4 and MgNb2O6 intergranular phases. In this work, the microestrutural changes of the alumina-niobia composites on heating and cooling from sintering temperature were observed by using differential thermal analysis, x-ray diffraction techniques and scanning electronic microscopy. The probably sintering mechanisms were discussed from microestrutural variations related to temperature.

POSTER PRESENTATIONS

H – P001 57Fe MOSSBAUER SPECTROSCOPY IN LiFePO4-BASED COMPOUNDS: THE EFFECTS OF DELITHIZATION AND SUPERVALENT METAL DOPING

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A number of new materials investigated recently were proposed as alternative cathode to the currently employed oxide (LiCoO2, LiMn2O4) , in lithium ion batteries. The properties of these materials strongly influence the global performance of the high energy density rechargeable batteries. Iron-based compounds containing structural units (PO4)n- such as LiFePO4 have been investigated. The main advantages of these materials include abundance, environmental friendly, low costs and excelent thermal stability. Recently, a good improvement in conductivity was obtained, (8 orders of magnitude), by doping the LiFePO4 (olivine structure) with transition metals to Li+. There are controversies on the physical mechanism behind this phenomenon. In this work we present 57Fe Mössbauer spectroscopy studies of pure LiFePO4, delithiated-FePO4, Cr-doped, Ti-doped, Cu-doped, and Al-doped LiFePO4. In the case of the delithiated FePO4 and delithiated Cr-dopped LiFePO4 the quantitative values of Fe2+ and Fe3+ was determined. In addition, information related to the chemical environment of the probe atom Fe for different doping concentrations of the supervalent metal was obtained. A decreasing in Li content was seen to induce a systematic increasing of Fe3+ content. We get a detailed understanding of the local order of LiFePO4 with delithization process and of LiFePO4 with the Cr, Ti, Al and Cu doping

H – P002 SYNTHESIS AND CHARACTERIZATION OF POWDERS OF Na2TiSiO5

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This work presents a systematic study of the synthesis and characterization of Na2TiSiO5 compounds. We studied their structural and morphologic properties using X ray diffraction, Raman spectroscopy and scanning electron microscopy. The samples were produced through two different synthetic routes(called route 1 and route 2), both based on Pechini method. Different heat treatments were applied in order to obtain crystalline and disordered materials. The polymeric resins obtained were submitted to Raman spectroscopy, to verify their influence on the different routes used in the powders preparation. The X ray diffraction and Raman spectroscopy, showed thermal evolution of the phases, as well as, the structural disorder of the samples. X ray diffraction patterns of the crystalline samples were also analyzed by the Rietveld refinement, which allowed a detailed characterization of the crystalline structure of the material. The morphology of the samples was investigated by scanning electron microscopy. Both routes allowed the production of Na2TiSiO5 compound, through the route 2 seems to produce samples with lower amounts of additional phases rather than the route 1.

H – P003 TITANIUM RESEARCHES IN BRAZIL

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Titanium alloys parts are ideally suited for advanced aerospace system because of their unique combination of high specific strength at both room and high temperature, in addition to excellent general corrosion resistance. The research involving the metallic titanium in Brazilian Aerospace Technical Center (CTA) occurred between 1965 and 1990 aimed to reduce the delay of the Brazilian research in relation to the developed countries. All developed technology was transferred to the industry supplying the market with about 30 tons of titanium sponge, in this time, in a process just available to a closed group of industrialized countries. This work has for objective to rescue the importance of a historical period of the Brazilian\'s vacuum metallurgy for the reactive metals production. This work also includes the present researches in titanium powder metallurgy in the CTA and the current state-of-art of the titanium production in Brazil.

H – P004 RHEOLOGICAL BEHAVIOR OF PORTLAND/POLYURETHANE SLURRIES

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Portland has been reinforced by the addition of polyurethane, which is polymerized during the hydration of the slurry. Tensile tests revealed an increase in both UTS and fracture toughness, and a reduction of the Young´s moduli of the composites with respect to plain Portland. Hardened composite slurries are able to withstand greater thermomechanical stresses with improved plastic behavior, yielding improved materials for a variety of structural applications including oilwell cementing and the production of rheodynamic concrete. However, the addition of polyurethane caused a detrimental side effect to the rheological behavior of the material with significant increase in plastic viscosity and yield stress. Therefore, the main objective of the present study was to evaluate the effect of the addition of sodium naphthalene sulfonate as slurry dispersant. The results showed that small additions of sodium naphthalene sulfonate reduced the yield stress in 87% and the plastic viscosity in 27%. In addition, the dispersant used also retarded the polymerization reaction of polyurethane, which allows control over the setting time of the composite slurries. Addition of sodium naphthalene sulfonate did not affect the mechanical strength or the fracture toughness of the composites.

H – P005 PRODUCTION AND CHARACTERIZATION OF CERAMICS Al2O3/TiO2

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A dense slice of Al2O3 was joined to a porous TiO2 ceramic to combine good production and properties, such as Al2O3 toughness associated to TiO2 bacteriological function.
A new experimental conformation procedure was proposed based on the starch consolidation technique, producing samples with different slice thickness, changing proportions and kinds of starch. The adhesion between the slices was investigated using quantitative microscopy. Microscopy and image analysis permitted the identification of areas were the adhesion was almost complete in the contact region between the slices.Porous fraction and porous distribution in different sample were calculated to study the porosity behavior between slices and to verify the influence of pores in adhesion.

H – P006 Zr1-xFexSiO4 PIGMENTS PREPARED BY A CHEMICAL ROUTE

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Ceramic pigments are inorganic compounds, the quality of which depends on its optical and physical properties. In this work Zr1-xFexSiO4 pigments were developed by polymeric precursor method. Iron (III) nitrate was added to the solution in the following molar proportions: x = 0.01; 0.05; 0.25 and 0.50. The material was heat treated between 750 and 1200°C. The resultant powders were investigated by X-ray diffraction (XRD) in order to accompany the evolution of the crystalline phases. The microstructure was observed by scanning electron microscopy (SEM) and also by energy dispersive X-ray microanalysis (EDX). The color of the pigments was measured by CIE - L*a*b colorimetric system. Red pigments were observed by the values of the colorimetric coordinates.

H – P007 PRODUCTION OF STONEWARE PORCELAIN TILES FROM CLAY MATERIALS FROM CASTRO\'S BASIN

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Based on previous studies of clays located in the area of Carambeí-PR (south portion of Castro\\\'s basin), clays which presented white coloration after firing, being probably suitable to be used in stoneware porcelain tiles, were selected. Six compositions with different clays were prepared from a composition previously studied. Linear shrinkage, water absorption, apparent porosity and flexural rupture module were determined for each composition of test bodies after conformation and thermal treatment. Within the six clays analyzed, four presented characteristics that allow their use in porcelain stoneware tiles, presenting water absorption of approximately 0.1% and flexural rupture module ranging from 51 to 70N/mm2.

H – P008 PREPARATION OF La2TiO5 BY POLYMERIC PRECURSOR METHOD

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Lanthanum titanate particles were prepared by the polymeric precursor method. The titanium citrates were formed by the dissolution of titanium (IV) isopropoxide in a water solution of citric acid (~70°C). Then, lanthanum carbonate was added to the solution in the proportion of 2 lanthanum carbonate for 1 titanium citrate. After homogenization of the solution, ethylene glycol was added to promote mixed citrate polymerization. After that, the gel was pre-dried at 300°C for 2h. The powders were analysed by TG/DTA. The specific surface area of the powder was measured by BET. The morphology and crystal structure of the powders were investigated using X-Ray diffraction (XRD) and scanning electron microscopy (SEM).

H – P009 PRECURSOR MATERIAL FOR NiO.OH/Ni(OH)2 ELECTRODES OF THE ALKALINE BATTERIES

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There is a great scientific and technological interest in the study of the nickel hydroxide synthesis, due to its application in positive electrodes of alkaline batteries. Nickel hydroxide can be formed as -Ni(OH)2 or -Ni(OH)2 phases. In the -Ni (OH)2 lattice, intercalated water molecules and alkali metal ions separate the plane (001). -Ni(OH)2 is unstable in the presence of alkali, and by ageing, that is, with water and alkali metal ions elimination it becomes in -Ni(OH)2. Due to -Ni(OH)2 instability in alkaline solutions, -Ni(OH)2 is frequently used as a precursor material in the alkaline battery. The process of synthesis of the precursor material is still little understood. Microstructure control of the nickel hydroxide is being researched to improve the performance of the NiO.OH/Ni(OH)2 electrodes. In previous work show that the presence of the -Ni(OH)2 in the precursor material increase the specific capacity of the NiO.OH/Ni(OH)2. In this work a study about the relation between the chemical synthesis and the microstructure of the nickel hydroxide was carried on. The materials were characterised in different stages of its preparation with help of the following techniques: X-ray diffraction, Fourier transform infrared spectroscopy (FT-IR), thermodifferential (TDA) and thermogravimetric analyses (TGA), specific superficial area (BET) and UV-vis.

H – P010 THE FORMATION OF ZINC GALLATE IN HEAVILY DOPED ZnO:Ga POWDERS

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Gallium-doped zinc oxide (ZnO:Ga) has deserved great deal of attention due to its myriad of applications, especially as a transparent conductor oxide. This work is aimed at preparing ZnO:Ga (1, 2, 3, 4 and 5 at. %) in powder form by modifying the Pechini method and studying its structural and optical properties. The addition of edta to the synthesis helped avoid precipitate formation. Fine powders were achieved after heat treatment at 900ºC for 4 h. Only one phase (zincite) is observed in ZnO:Ga 1 and 2 at. %, whereas in ZnO:Ga 3, 4 and 5 at. % the spinel crystal structure of zinc gallate (ZnGa2O4) is also present. By means of quantitative phase analysis the amount of ZnGa2O4 in the latter samples was determined to be 7.6, 15.8 and 15.1% weight, respectively. The refinement through the Rietveld method revealed an increase in the unit cell volume and microdeformation from ZnO to ZnO:Ga 3 at. %, which is possibly related to the presence of Zn+. Reduction of Zn2+ is a possible way for charge compensation, since the dopant has charge 3+. For dopant concentrations higher than 3 at. % the unit cell volume decreases due to the smaller formation of Zn+. By diffuse reflectance spectroscopy a band gap of 3.3 eV was estimated for ZnO:Ga. The blue emission of ZnGa2O4 was not observed in the photoluminescence spectra of ZnO:Ga. ZnO:Ga can be successfully prepared by modifying the Pechini method up to the solubility of ca. 3 at. %, after which two crystalline phases are present.

H – P011 POROSITY INFLUENCE IN BIOMATERIALS CRYSTALLINITY

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The bioceramics based on calcium phosphate are in among prominence in biomaterials, having shown good biological compatibility with teeth and bones and readily reacting when in contact with natural tissue. Another important factor that biomaterials constituted of Na2O-CaO-SiO2-P2O5 were obtained by the classic method of melt and bioglasses submitted to the thermal treatment to different temperatures, where the porosity of the pieces were controlled by the addition of sawdust and/or glucose.

Samples submitted to temperatures above 800 ºC became completely opaque, indicating a reorganization of the structure confirmed by DRX analysis and,consequent, increase in the hardness of the material. The influence of the sawdust in the process os crystallization of the glass was observed.

Scannig Electron Microscopy was used, in both ceramics, and results showed the increase of the porosity as so much sawdust or corn glucose was additioned. However, the most organized structure and more homogeneous pores were obtained for the mixture glass/corn glucose.

H – P012 INFLUENCE OF MgO AND CaO ADDITION IN THE COLOR AND TRANSPARENCY OF CERAMIC GLAZE OBTAINED FROM CALCAREOUS SHALE

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The magnesium and calcium oxides when used in glazes provide crystallization and consequently their opacification. Calcareous shale is a mineral consisting mainly of dolomite and quartz. Thus, its use allows to understanding the influence of these oxides in the coloration of ceramic glazes. In this work, a ratio of calcareous shale ranging from 0 to 25% were used in order to study transparency and colors by means of spectrophotometry in the visible region. The results shows the influence of calcareous shale in pigmented and no-pigmented glazes. Five pigments types have been used and the results were evaluated by means of reflectance curves and CIELab colorimetrics coordinates. The results of thermal expansion coefficient for the compositions presents a range from 43x10-7 to 58x10-7 oC-1, being that the lower values have been observed for compositions with 10 and 15% of calcareous shale.

H – P013 METAL- FILLED POLYMERS: SYNTHESIS, MECHANICAL BEHAVIOR, ELECTRICAL PROPERTIES AND THEORETICAL MODELING FOR COPPER-PMMA COMPOSITES

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We report on the preparation and characterization of conducting copper-polymethyl methacrylate (PMMA) composites. In order to accomplish a polymer-copper composite, we have investigated the structural, mechanical and electrical properties of conducting polymer composites using copper powder aggregates. The percolation threshold of the composite was found and the properties around this value were measured and modeled. The composites were synthesized using spherical copper particles of different mean particle size embedded in a PMMA matrix, using different volume concentrations of filler metal. The homogenized mixture was introduced in a mold at a pressure of 40 MPa at a temperature of 400 °C, under inert atmosphere, during 15 minutes. SEM examination of the microstructure of copper-PMMA composite of filler metal, have demonstrated the presence of infinite percolation bond between copper particles, distributed randomly into the polymeric matrix with and without contact between themselves forming a conducting paths into the polymer matrix. This behavior was observed in all studied concentrations. The room temperature conductivity was measured using the two-probe technique with parallel silver contacts. The ohmic behavior was checked in each sample. The experimental data fitted using a theoretical model describes the conductivity in composites due to percolation of mobile carriers, in this way, the experimental results are in agreement with the theoretical law.

H – P014 COMPACTION BEHAVIOR X PHYSICAL CHARACTERISTICS OF CLAYS FROM CORUMBATAÍ FORMATION

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In the present work, the relationships among physical characteristics of pre-milled clays from Santa Gertrudes ceramic district and their compaction behavior are evaluated. Compaction of clay-based ceramics is related to raw materials physical, chemical and mineralogical characteristics, besides processing parameters like moisture content and compaction pressure. Ten clays from Corumbataí formation used in floor tile production at Santa Gertrudes ceramic district (São Paulo, Brazil) were characterized to granule size distribution through sieve analysis, particle size distribution through X-ray sedimentometry, surface roughness through optical microscopy and plasticty through determination of methylene blue index. Responses analyzed in order to evaluate compaction behavior were tap density, apparent density after drying of uniaxially pressed test bodies under four compaction pressures, and modulus of rupture on three-point bending test. Comparisons of clays physical characteristics and measured responses were carried out with statistical tools like correlation and regression analysis. Results showed a good agreement between particle crowding index and plasticity related to compaction behavior.

H – P015 STUDY OF THE CHEMICAL STABILITY OF PIGMENTS CHROMIUM DOPED ZIRCONIUM SILICATE BASED

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Research and development of new pigments with potential industrial applicability has been the theme of several studies in the last years. Requirements for a good pigment are related to: 1) thermal inertia; 2) chemical inertia; 3) high color strength and 4) acceptable optic properties. Thus the present work aims to characterize the chemical stability of developed chromium doped zirconium silicate based pigments front the aggressive ways like silicate melts of different characteristics. Compositions had been formulated keeping its molar equivalence between silica and zirconium, and adding 2, 5, 8, 12 and 15% of Cr2O3, beyond a mineraliser agent. These samples had been calcined 1000ºC/3h, disaggregated in mortar and submitted to the stages of: milling, washing and drying. After this, the pigments had been incorporated in three types of glazes: transparent, opaque and matte, in the ratio of 5% (dry basis), applied in engobed BIIb ceramic tiles with a binil and calcined in industrial oven. Characterization of color development was carried out directly on powdered pigments and on pigments applied in three glazes, by diffuse reflection spectroscopy. Through the comparison of the results between the samples (powder and applied in three glazes) a stability relation could be attained, which revealed adequate. Thus one concludes that the developed pigment is chemical inert, a basic requirement for its future application in ceramic coatings industry.

H – P016 CHARACTERIZATION OF OIL SHALE FINES FOR ADSORPTION APPLICATIONS

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There has been a growing interest in the use of cost-effective materials to remove heavy metals from wastewater. New processing techniques and adsorbents including natural clays, synthetic resins, peat and chitosan have been studied aiming at replacing activated coal. In the present investigation, retorted shale, a solid residue of the thermal transformation of oil shale, was characterized with the objective of preparing heavy metals adsorbents. In addition to the direct environmental benefits from the technology to wastewater, Brazil has the second world reserve of oil shale. The fine residue was fully characterized by different techniques including particle size, thermogravimetric analyses, BET, X-ray fluorescence, FTIR, X-ray diffraction, and SEM/EDS. The characteristics of the powder were appropriate for adsorption. Samples were prepared and used in adsorption experiments carried out using Pb2+, Cu2+ and Cr3+ synthetic solutions as well as wastewater. The results are presented and discussed herein.

AUTOMATIC CLASSIFICATION OF LAMELLAR GRAPHITES IN GREY CAST IRON

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The grey cast iron is an industry widely used material by its low cost combined to the fact that in most cases it is ease to machine. It has graphite inclusions with different morphologies, which has direct influence in its mechanical properties. According to the ASTM A247-67 standard, the lamellar graphite inclusions are classified in the following types: A, B, C, D and E. This classification is exposed in optical microscopy acquired images with 100x amplification in samples without chemical etching. Industrially, the classification is realized by visual inspection, where sample images are compared with the standard images. This technique, although, has a high degree of subjectivity, because it depends of the operator’s experience, it is tedious and slow. Analyzing this factor, it is notorious that this technique has a low degree of reproducibility and causes low operator productivity. Several methods have been proposed for solving this problem. Among them, image analysis show a lower operator dependence, greater reproducibility and a high operator productivity. In this research, motivated by a specific industrial problem, we present an method for classification of graphite inclusions types A and C. Several non dimensional morphology-based parameters were used for this classification. It has been implemented a prototype using the IMAGO software and several tests were realized in grey cast iron images, showing its inclusion classification capability.

H – P018 PROPERTIES OF EXPANDED GRAPHITE PRODUCED FROM BRAZILIAM NATURAL CRYSTALLINE GRAPHITE

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Graphite intercalation compounds or expandable graphite is flake graphite stems from nature graphite mine intercalated which species donors or receptors. In the present work, we compared two products of different intercalation processes, chemical and electrochemical synthesis using Brazilian natural graphite. The chemical process was based on the oxi-reduction reaction of the natural graphite with HNO3 and H2SO4. Another process was based on the electrochemical reaction between the natural graphite and H2SO4 as an electrolyte and intercalate. Both intercalated graphite are washed on the proportion 1g:3mL (graphite: water) and expanded on a muffle at 900ºC. These samples were milling to average particle size 150mm and characterized. These materials are characterized by a charge transfer process between intercalate and the graphite layers. A definite number of graphite layers separate intercalate layers, and the number of graphite layers between two intercalate layers is referred to as the stage of a compound. A variety of physical-chemistry properties such as electrical conductivity and lattice dynamics depend on the stage structure. Both processes produced pure and expanded graphites, and after milling, both graphites have the surface area and porosity diminished. All the samples presented good characteristics and properties that are adequate to specific applications.

H – P019 MORPHOLOGICAL AND TEXTURAL CHARACTERIZATION OF FUNCTIONALIZED PARTICULATE SILICA XEROGELS

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Functionalization of the xerogels for use in chromatograph and catalysis was carried out by solubilization of amorphous silica using a soxhlet extractor. Xerogel was prepared by sol-gel method using tetraethoxysilane, TEOS, ethanol and water in a 1/3/10 molar ratio with HCl and HF as catalysts. Xerogel were obtained by hydrolysis and condensation of TEOS. The samples were prepared in monolithic form and dried at 70 ºC and 110ºC both for one hour each step. After functionalization, the changes on morphology and textural characteristics of xerogel have been investigated by means of scanning electron microscopy (SEM), positron annihilation lifetime spectroscopy (PALS) and nitrogen gas adsorption (BET). As the methods of analysis are based on different physical principles, the results are complementary leading to a good knowledge of the texture of the samples studied.

H – P020 POWDERING AND AMORPHOSATION OF LAVES PHASES

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Hydrogen is known enhance the microstructure and properties of a large variety of materials, including alloy. Currently the process of hydrogenation is made using gas reaction at high temperatures and/or at high hydrogen pressure. Looking for others hydrogenation methods for alloys, we propose the use of electrochemistry hydrogenation. In this work we present the results of electrochemical hydrogenation in the laves phase alloys Fe2Ce and Fe2Gd using Moessbauer Spectroscopy and x-Ray Diffraction. The samples were prepared in a arc melting furnace under atmosphere of argon. After fusing, the alloy was submitted an annealing in inert atmosphere. The electrochemistry hydrogenation was performed an electrolytic solution 0.1N NaOH at 50OC with a current density between 2.5A/m2 and 7.0A/m2. During the electrolytic hydrogenation, the samples were pulverized in fine particles. X-Ray Diffraction and Moessbauer Spectrsocopy results on Fe2Ce and Fe2Gd samples shown partial hydrogenation for low current density and for high current density the sample become amorphous. The Mossbauer Spectroscopy presents the onset of the magnetic order for the hydrogenated samples of Fe2Ce. The experimental results show very close similarities between gas hydrogenation and electrochemistry hydrogenation. Thus, the electrochemistry hydrogenation process permits to realize improvements on properties of materials, in special because the process is simple and is realized at room temperature.

H – P021 MECHANICAL ALLOYING AS A PROCESSING ROUTE FOR ULTRAFINE ELECTROLYTIC COPPER POWDER AND COPPER BASE ALLOYS

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Copper is a base material for high electrical and thermal conductivity applications, but it is known the low or moderate tensile and yield strength over 500ºK. To overcome this problem, any inhomogeneity that interacts with dislocation can be used as strengthener. In the present study, it has been shown that mechanical milling and alloying followed by hot pressing is a viable and promising processing route for copper based alloys by using electrolytic powder obtained from electro-winning process.

The production of powder by electro-winning has certain advantages in the cheap sources of raw material exist. In this study the process was adaptable to the production of fine dendritic powder without difficulties. The refining to ultrafine copper particles and strengthening is provided by mechanical milling in a planetary mill during 8 to 30 h with 0,5% (Cr and Cr3C2.) as fine dispersoids.

The alloyed .powders were hot consolidated by uniaxial pressing at 923ºK during 2h.The alloyed powders and consolidated samples were microstructural characterized by X-Ray Diffraction(X-RD), electron microprobe(EPMA)and scanning electron microscopy(SEM). Mechanical properties are measured with satisfactory results.

H – P022 NEW GEOMETRICAL METHOD FOR ULTRASHORT LASER PULSE ABLATION THRESHOLD DETERMINATION AND APPLICATION TO BOVINE TOOTH ENAMEL

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A new geometrical method for determining ultrashort laser pulses ablation threshold is presented. The method requires the sample to be diagonally translated through the laser beam waist resulting in a pattern etched in its surface. The ablation threshold is obtained measuring only one dimension of this pattern, without the need of knowing any other geometrical parameter of the laser beam or of the lens used. The technique was used to determine the ablation threshold of bovine tooth enamel in the 60 femtosecond regime to be 0.6J/cm2. Also, the quantity of material and the depth ablated per pulse were determined to be 15ng and 400nm, respectively. This removal rate makes femtosecond pulses as natural tools for nanomachinig. The patterns etched present a very sharp edge, showing that the Heat Affected Zone is negligible for this regime.

H – P023 SYNTHESIS OF CATHODE LASRMNO3 BY METHOD POLYMERIZABLE COMPLEX FOR SOLID OXIDE FUEL CELL (SOFC)

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The SOFC have showing increasing interest as efficient generators of electric energy. The development of materials for cathode is a complex process that involves microstructural considerations and dynamic relations between the properties of interfaces cathode/electrolyte, cathode/interconector and process of O2 reduction. Factors related to adjacent components, such as compatibility enter thermal expansion coefficients with the electrolyte, thermal stability and microstructural in the operating temperature and tolerance to gaseous impurities also they must be considered. Perovskites structures (ABO3) of lanthanum strontium manganite vastly are used as material of cathode. The sites B are related to the activity of O2 reduction, while the sites A normally are associates to the properties of electric conduction of the porous cathodes. In this work are presented the results gotten through the synthesis of La1-xSrxMnO3 cathodes (x = 0,15; 0,2; 0,3), through the synthesis by polymerizable complex. This synthesis is based on the reaction of citric polyesterification in situ between acid and ethylene glycol 90ºC of ions La,Sr and Mn, resulting in a transparent polymeric resin.The polymeric resin was preheated at 320oC/2 h to give a powder precursor for LaSrMnO3 and was verified the temperature of calcination for the formation of perovskite between 450 and 950oC/4 h in air. The powders obtained were characterized by distribution of size of particles, SBET, FTIR, DRX, MEV, TG/DTG.

H – P024 EVOLUTION OF CRYSTALLOGRAFIC PHASES IN THE SYSTEM (Pb1-XSrX)TiO3 POWDERS PREPARED BY THE SOFT CHEMICAL TECHNIQUE

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Strontium-modified lead titanate (PST) have had a large technological interest due to its excellent ferroelectric properties, wich are very influenced by the ceramic preparation. A polymeric precursor method based on Pechini process was used to synthesize (Pb1-xSrx)TiO3 powders with strontium contents corresponding to x=0.00; 0.10; 0.20; 0.30; 0.40; 0.50; 0.60; 0.70; 0.80; 0.90 and 1.00. This powders was calcined at different temperatures for 2 h and the evolution of crystallografic phases in the system were studied using X-ray diffraction (XRD). The lattice parameters and cell volume of the PST were measured using the least-squares method and shows that the parameters indicated a decrease in lattice tetragonality with the increase in strontium content. This indicates a gradual change from the tetragonal to cubic structure in these powders.

H – P025 EFFECT OF THE ADITTION OF THE TANTALUM CARBIDE IN THE SINTERING AND MECHANICAL PROPERTIES OF THE AlSi 316L

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In this work, it is intended through the technique of powder metallurgy, to explore the effect of the additive TaC in the hardness and microstructure of the AISI 316L sintered. This powder produced in our laboratories (UFRN) is analyzed in its composition, morphology and distribution of size of particles. Later it will be produced compact of the AISI 316L with or without use of the additive TaC, and in this last case the contents used will be up to 10% wt. The compact produced will be sintered in temperatures between 12000C and 14000C and after this, it will be characterized by the X-Ray diffraction(XRD),scanning electron microscopy(SEM), density and hardness. The results will be compared with other finding for the same materials whose additives had not been produced in our laboratories. Two points will be studied: the effect of the deriving contents of additives of the UFRN and another manufacturer in the sintered microstructure and the correlation of the values of hardness with the sintered microstructure.

H – P026 EFFECT OF CHROMOPHORE ION Cr3+ IN THE ZIRCONIUM SILICATE MATRIX FOR CERAMIC PIGMENT ATTAINMENT

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Industrially used zirconium silicate ceramic pigments are mainly of the three types: the yellow of praseodymium, the blue vanadium turquoise and the pink-coral of iron, all discovered and patented in 1948 for SEABRIGHT, C.A.. The insertion of transition metal ions, the so called chromophores agents, in crystalline matrices provides the development of colors, due to the possibility of these elements originating d-d transitions. Thus the present work aims to study the effect of the content of the chromophore ion Cr3+ in the zirconium silicate matrix. Compositions had been formulated keeping its molar equivalence between silica and zirconium, and adding 2, 5, 8, 12 and 15% of Cr2O3, beyond a mineraliser agent. These samples had been calcined 1000ºC/3h and disaggregated in mortar. A fraction was separated and characterized by XRD, UV-Vis and NIR. Another fraction was submitted to the stages of: milling, washing and drying. After this, the pigments had been incorporated in a transparent enamel in the ratio of 5% (dry basis), applied in engobed BIIb ceramic tiles with a binil, calcined in industrial oven and submitted to colorimetric analysis. XRD results showed the formation of zirconium silicate phase in all compositions; however the tonality of the pigment, characterized by colorimetry, was clearly different among samples, varying from light green to dark green.

H – P027 THERMAL BEHAVIOR OF RARE EARTHS MODIFIED PbTiO3

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Rare earths modified lead titanate ceramics were prepared by Pechini method. The materials were calcinated under flowing oxygen at different temperatures and the obtained materials were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM) and infrared spectroscopy (IR). The thermal behavior of Ytterbium (Yb) and Yttrium (Y) using thermogravimetric analysis (TGA), differencial scannig calorimetry (DSC) and differencial thermal analysis (DTA) will be report. The TGA, DTA and DSC curves of the Yb and Y modified lead titanate are similar. The TGA curves of the rare earths modified precursors showed three-stage weight loss, a steady weight loss from room temperature up to 200 ºC due, probably, to the elimination of residual water and the dehydration of the precursors, a sharp fall in the specimen weight after this temperature that is mentioned to the oxidative decomposition of the organic material and little weight loss is observed at temperatures above 500 ºC. The dehydration and decomposition of the organic material processs observed in TG are confirmed through endothermic and exothermic events by DSC and DTA curves.

H – P028 PHOTOLUMINESCENCE STUDIES OF YTTERBIUM MODIFIED LEAD TITANATE CERAMICS

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Ytterbium (Yb) modified lead titanate ceramics (Pb1-xYbx TiO3, with x = 0.01, 0.05 and 0.07) (PYbT) were prepared by the Pechini method. The materials were calcinated under flowing oxygen at different temperatures from 400 to 700 ºC.The PYbT obtained were investigated using X-ray diffraction (XRD), infrared spectroscopy (IR), raman spectroscopy, scanning electron microscopy (SEM) and surface area analysis (BET). The present work describes the Ytterbium effect on luminescence of the lead titanates ceramics. The emission process were investigated under 488 nm (Ar laser) excitation at room temperature. The photoluminescence spectra of these materials present wide bands in visible region. The emission behavior is related to structural morphology of the material as a function of Yb concentration and the submitted thermal treatment.

H – P029 WATER SORPTION OF A POLYURETHANE/BENTONITE COMPOSITE. EFFECTS OF BENTONITE CONTENT AND DRYING CONDITIONS

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Composite properties are dependent on matrix and filler characteristics and interactions, which can be modified by surface treatments, processing and drying conditions. In this work compression moulded polyurethane/bentonite composites were manufactured and their water sorption characteristics determined as a function of filler content and drying conditions. The matrix was a polyurethane derived from castor oil and the filler a natural sodium bentonite from the city of Boa Vista-PB, commercially known as Brasgel. Filler contents varied from 0-10% in weight and filler drying conditions were: 110 and 200ºC for 1h. The dried filler was added to the polyol, stirred at 600 rpm for 5 minutes, the isocyanate prepolymer added to the mixture and the system stirred for another minute and poured on the mould (200x180x3mm) which was closed and the ensembled pressed under 10 ton for 4 hours at room temperature before demoulding. Samples (20x20x3mm) were cut off from the plate, their borders sealed with resin, the composites immersed in water and weighted at pre-determined time intervals. Our results show that the composites´ water sorption were reduced at higher drying temperatures and that although slightly increasing with bentonite content, they remained smaller than that of the matrix in the first 200 hours of immersion. These results were associated with lower void contents with more efficient drying and to the fillers ability to reduce matrix swelling, thus hindering water sorption

H – P030 SYNTHESIS OF FOAM GLASS BY USING THE WASTE OF CRUSHING LAMPS TO OBTAIN AN THERMOACOUSTIC BLOCKS

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The foamed glass is a light porous material. The constitution of this material resembles solid soap foam. The size of cells of foam bubbles varies from shares of milimeter to centimeter. The colour of a material is from light to black, but depending on a glass composition and additives its possible to get any colour. Foam glass, as any glass is water insoluble and corrosion resistant in most acids and organic solvents, evev at high temperatures. The main unique property of foamed glass in comparison with traditional building heat-insulating materials is low heat conductivity combined with high strength and convenience of processing, ecologicaL safety and durability. In Brazil about 300.000 tons of waste lamps appear annually. The utilization of this glass is still an unsolved problem. The present research aims to produce the novel foam glass by using the waste of crushing lamps to obtain a median size 30 micrometers, and mixtured with inorganic additive in a ball-mill for 2 hours. The powders are put in a crucibles of 10 x 15 x 25 centimeters dimension. The crucibles are put in a electric furnace for 30 minutes, at T = 710 - 780 degrees ranges to obtain the foam glass material. The results of characterization shows by Xray diffraction patterns the SiO2 and Calcium-sodium-silicate crystals phases. Analysis by SEM shows the interconnected porous by 100 micrometers, and the density about 0.30 g/cm3, by helium picnometer. These material can be applied as cryogenic isolant.

H – P031 FEATURING OF TiO2 CERAMIC FORMED BY COMMERCIAL PROTEIN CONSOLIDATION IN DIFFERENT GELLING CONDITIONS

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Gelling process is more important in order to improve the ceramic properties formed by protein consolidation. In this stage, there is production of tridimensional gel structure that supplies to material some solid features. This work purposes a comparison of gelling process of these ceramics that were made with different temperatures. Samples were produced in bar forms, according to ASTM C1161/94 norm. The characterization consisted of three-point flexural test, superficial roughness and apparent density measurements and microstructure analysis by light microscopic. The results showed that there isn\'t meaningful modification for all processes and the ambient temperature of gelling is more indicated for forming big quantities. This permits to reduce cost and time of production.

H – P032 STUDY OF THE AMMONIA FORMATION ON METAL SURFACES DURING PLASMA PROCESS TREATMENTS

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Nitrocarburizing and nitriding are termochemical processes used to improve the mechanical properties of metallic components through the enrichment of their surfaces with nitrogen and carbon atoms. Although several studies about the development of these processes aiming industrial applications exist, the information available in the literature concerning the absorption mechanisms of carbon and nitrogen atoms is relatively scarce. Therefore, the selection of optimal parameters for these processes is currently undertaken through different attempts. The main purpose of the present study is to investigate the reaction mechanisms of the plasma nitrocarburizing and nitriding processes. A mass spectrometry technique was used to study the composition of the chemical species formed in the plasma and the interaction region between the plasma and the sample surface. Treatments were realized in sintered and in cast iron samples, in gaseous mixtures of N2/H2/CH4 and N2/H2, and at temperature and pressure, of 500 ºC and 400 Pa, respectively. The presence of NH3 in the gaseous mixtures during the processes was identified and a correlation between the formation of this species and the formation of the diffusion layer in the treated sample was observed. Moreover two different paths of formation for this species could be verified. In the first case the ammonia is formed by catalysis on the surface of the sample while in the second case it is formed as a product of the processes.

H – P033 WET-CHEMICAL PROCESS TO SYNTHESIZE Ce1-xCoxO2 PIGMENT POWDERS

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This work reports the synthesis by polymeric precursor method and characterization of nanosized Ce1-xCoxO2 pigments, wherein is equal to x = 0.01, 0.02, 0.04, 0.08 and 0.16% in mol. The material was first heat treated at 320oC for 1h, in order to eliminate the organic fraction. In the next step, Ce1-xCoxO2 nanoparticles were calcinated in the temperature range from 500 to 1000oC for 2h (at a heating rate of 10oC/min) in air atmosphere. The effects of heat treatment and cobalt concentration on the final product were investigated by X ray diffraction (XRD), UV-visible espectrofotometry (UV-vis), colorimetric coordinated and transmission electronic microscopy (TEM). The XRD results indicated that samples with x< 0.04% are single-phase material with ceranite type structure, whereas the diffraction patterns showed the presence of a secondary phase (CoO) above this percentage. The particle size distribution varied between 10 and 90nm and, it was observed that the color of the pigments changes as function of the material structure ordering.

H – P034 INFLUENCE OF FREEZE-DRYING AND SPRAY DRYING METHODS ON THE MORPHOLOGY OF CHITOSAN MICROPARTICLES MODIFIED WITH PAPAIN

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The use of biopolymers has been growing in the last years, mainly those gotten from natural sources, as chitosan, a deacetylated derivate of chitin, present in crustaceans shells as crab and shrimp. Due to its biocompatibility with human tissues and organs, this material has potential for use in biomedical engineering and pharmaceutical and medical areas. Chitosan presents wound healing properties and the incorporation of other drugs can improve such quality. Papain is an enzyme that presents anti-inflammatory and antibacterial property. For this reason, it can act improving the healing of injured epithelial tissues. The objective of this work was to evaluate the influence of the drying method used to obtain chitosan microparticles containing papain on their morphology and papain released profile. The release of papain is performed “in vitro” in pH 7.4 phosphate buffer. X-ray diffractometry (XRD) and scanning electron microscopy (SEM) were the used techniques.

H – P035 POLYMERIC TANTALUM PHOSPHATES FOR ION EXCHANGE MATERIALS AND THEIR MORPHOLOGICAL CHARACTERIZTION BY SEM

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Some polymeric inorganic materials are suitable for ion exchange procedures and has been studied in literature. Our research group studied a systematically way some of this materials like the hydrous zirconium oxides and the tungsten corresponding material .In the same way two different tantalum phosphate materials was prepared, characterized and their ion exchange behavior studied too. Amorphous materials in both cases were obtained, beside the literature describe a crystalline material prepared in a solid phase reactional system. The morphological characterization of prepared materials was performed by scanning electronic microscopy in order to exploit their grain distribution and the particles geometry, after and before an exhaustive thermal treatment of the two different synthetic routes for the tantalum phosphates preparation. There are no significative differences on crystallinity and of the particle sizes before the reflux thermal treatment.

H – P036 TAGUCHI METHOD\'S APLICATION ON MILLING PROCESS OF NIOBIUM HYDRIDE

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Taguchi method of preform design is a powerful tool for improvement the quality of the processes. In this paper the Taguchi method was used to find the best milling efficiency of the niobium hydride obtained by the hydrogenation–des hydrogenation process (HDH) with particle size inferior to 75 mm. The influence of the following variables in the niobium hydride milling was studied: volumetric fraction of the balls mill; milling speed; milling time; speed in the rheostat and screening time. An orthogonal array was used with process variables operating in 3 levels, a sign to noise ratio for higher-to-better function and a statistical variance analysis to test the significant of the effects. In addition, the morphology of the particles was observed through SEM on the experiments with the better and worse revenue. Experimental results showed that the milling speed and the milling time are the main variables of the process and the combination of the all variables that provided the largest efficient. The characterization of the particles through SEM showed that in the experiment of smaller revenue it was obtained angular particles with low superficial roughness, the cleavage degree were well evidenced and the particle size distribution with significant homogeneity. In the experiment of larger revenue the particles were less angular , larger superficial roughness, larger incidence of cleavage degree and smaller medium size of the particles than another, however more heterogeneous.

H – P037 SYNTHESIS AND CHARACTERIZATION OF PEROVSKITE, La0, 9Gd0, 1NiO3, PREPARED BY POLYMERIC PRECURSORS

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This work described the synthesis and characterization of perovskite, La0, 9Gd0, 1NiO3, prepared by polymeric precursors.

The system was prepared using a 1:3 molar ratio of metal and citric acid (Me:Ac). The prepared resins were calcinated at 300, 500, 700 and 900oC for 2 h at ambient atmosphere. The resulting powders were characterized by TG/DTG, DTA, FTIR spectroscopy and X-ray difractometry (XRD), BET surface area and scanning electron microscopy (MEV). The X-ray difractograms obtained at 300oC are amorphous. The powders calcinated at 500, 700 and 900oC showed that cristalinity depends on the calcinations temperature. The BET results show that the materials calcinated at 900 oC presented a decrease on specific superficial area. The nitrogen adsorption/desorption isotherms for the powders calcinated at 900 oC for 2 h present behavior the type one hysterese indicating that mesopore structures occur. The amorphous and crystalline powders exhibit catalyst properties.

H – P038 EFFECT OF THE ADDITION OF THE NIOBIUM CARBIDE IN THE BEHAVIOR OF THE SINTERING AND MECHANICAL PROPERTIES OF THE STAINLESS STEEL 316L

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In this work, it is intended through the technique of powder metallurgy, to explore the effect of the additive in the mechanical properties and microstructure of the AISI 316L sintered. They will be used as additive preferential, powder of NbC with particles of nanometric size, produced in our laboratories (UFRN) and analyzed in its composition, morphology and distribution of size of particles. Later they will be produced stainless steel compact with or without additive and in the first case the used contents will be of up to 10% wt. The compact produced will be sintered in temperatures between 12000C and 14000C and after characterized through its chemical composition, density and hardness. The gotten results will be compared with other finding for the same materials whose the additives had not been produced our laboratories. Specifically, in this direction, they will be studied mainly: the effect of the deriving contents of additives of the UFRN and another manufacturer in the sintered microstructure and the correlation of this with the gotten values of hardness.

H – P039 STRUCTURAL GLASS-CERAMICS FROM BLAST-FURNACE SLAGS

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The utilization of waste products from industry is an ecological challenge persecute for many countries. Comercial sucess of glass-ceramics materials enables investigations of cheap raw materials sources like blast-furnace slags from metallurgical industries.This present paper investigated and characterizated structural glass-ceramics from blast-furnace slags used in ballistic armour. Glass-ceramics were obtained by sintering and crystallization of the frit of the parent glass. In this paper were prepared three glass compositions: slag, slag with ZrO2 and slag with ZrO2 and Cr2O3 as nucleating agent. Samples of this glass-ceramic were obtained from melting of the parent glass in ZAS crucibles in a temperature of 14500C for 2 hours. The glass specimens were heat-treated to convert them to fine- grained glass-ceramics in a temperature of 8000C for 2 hours and 930C for 30 , 60 and 90 minutes . Then, samples of glass-ceramics were characterized by means of optical and SEM microscopy, X-ray diffraction and Vickers microhardness measurements

H – P040 LUMINESCENCE: STUDY OF DISORDERED BaWO4

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The great majority of the photoluminescent studies are focused on crystalline materials, in which the emission happens at cryogenic temperatures, limiting the application of these materials. On the other hand, several solids with high density of defects (amorphous), such as titanates, zirconates, present photoluminescence at room temperature, similar to amorphous silicon. In this work, BaWO4 was synthesized by polymeric precursor method. The material was heat treated in ranging from 300 to 500oC in O2 atmosphere. Intense photoluminescence was observed in disordered BaWO4 samples at room temperature. It is supposed WO42 - ion and WO3 defects originated by oxygen vacancies are the responsible for the emission, within the visible region of the spectrum, presenting larger intensity between 500 and 600 nm.

H – P041 MANUFACTURE PROCESS OF WOOD PARTICLE BOARDS: MACHINERY SPECIFYCATION

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The wood based composites have showed advantages in relation to sawn wood due to many factors. They can be manufactured from fibers, veneers or particles. In order that your production be efficient that is important choose appropriate machinery, because the steps in the your manfacture process can influence end characteristics, as mechanical as aesthetics. That paper broach the wood based composites production made with particles as much of industrial particleboard as Oriented Strand Board (OSB), emphasising your similarities and differences.

H – P042 Al-Si MATRIX USED IN IONOMER GLASSES PREPARED NON-HYDROLYTIC SOL-GEL ROUTE

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In this work we investigate the preparation of ionomer glasses by a non-hydrolytic sol-gel route as an alternative technique to obtain alumina-silica matrices. The candidate gels were prepared in oven dried glassware in two ways. In the first, AlCl3, SiCl4, CaF2, AlF3, NaF, AlPO4 and ethanol were reacted in reflux under argon atmosphere. In the second, only AlCl3, SiCl4 and ethanol were initially mixed, and the other components were added at the end of the reaction. Europium III chloride was added as a structural probe. The powder mixtures were dried and –heated-treated at 1000oC during 4 hours. The formation process and structure of the powders were studied by means of thermal analysis, photoluminescence and nuclear magnetic resonance. In the first methodology TGA/DTA/DSC analyses showed a constant loss of mass that can be ascribed to structural changes during heating. In the second, the loss of mass occured until 300oC indicating that the matrix stabilized after this temperature. The excitation spectrum presents a maximum at 392 nm corresponding to 5L6 level, and the emission spectra of Eu III ion present characteristic band transitions arising from 5D0 - 7FJ (J = 0, 1, 2, 3 e 4) monifolds excited at their maximum. The magnetic dipole 5D0 - 7F1 and electric dipole 5D0 - 7F2 transition present more bands than allowed, which indicated different Eu III sites in the matrix. We obtained a stable matrix alunino-silicate by sol-gel method, that can be used with base ionomer glass

H – P043 IMPROVEMENT ON THE PARTICLE SIZE DISTRIBUTION ANALYSIS METHODOLOGY

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The high resolution particle size analysis is used to obtain with accurate, particle size distributions from 10 nanometers right up to 100 microns in samples such as ceramics, clays, refractories, ores and metal powders, etc. In this work a X-Ray Disc Centrifuge System was utilized to characterize oxide powders with different densities. The choise between gravitation or centrifugation settling mode was made depending of the size particle. A fluid with appropriated viscosity was used aiming to obtain a stable suspension. It was verified the influence of several aspects, such as: density of the particle, viscosity of the fluid, concenctration of the suspension and time of the analysis in obtaining of reliable results for particle size distribution. It was observed that the best time of analysis is around 20 to 40 minutes. This time depends of the sample and of the settling mode. The concentration of the suspension, the viscosity of the fluid and the density of the particle are related among them. Higher density of the sample, higher the viscosity of the fluid is necessary to obtain a stable suspension. Particle mean size results with low reliability were obtained by using solutions with concentration higher than 20 g/L.

H – P044 PREPARATION OF PIGMENTS IN THE SYSTEM SnO2.ZnO.Cr2O3.CuO

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The system SnO2.ZnO.Cr2O3.CuO to be used as a ceramic pigment was studied in this work. The pigment was obtained through the conventional ceramic process of oxide mixture. The samples obtained were characterized by X-Ray Diffraction and Particle Size Distribution. After the glazing and firing stages, the ceramic pieces were characterized by UV-vis spectroscopy where the colors were determined by colorimetric scale CIELab in which the calculated parameters L, *a, *b are characteristic of a grayish blue tonality.

H – P045 STRUCTURE AND PROPERTIES OF LAYERS PRODUCED BY COMBINED SURFACE ENGINEERING METHODS

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Surface engineering techniques have been developing in a very dynamic way. Many efforts have been made during the past decades, mainly in the controlling, optimising and modelling of the processes and also in developing new methods. A prospective line in the development of surface engineering is the use of new duplex methods (multiplex) which combined processes for producing multicomponent.

This techniques can be satisfied by e.g. applying various surface engineering techniques that permit to modify the microstructure, phase and chemical compositions of the surface layers of the treated parts. The number of possible combinations of the processes is unlimited. The multiplex methods can be used for prolonging the service life of products made of aluminium, titanium and its alloys, stainless steels, because of their specifically advantageous properties, will play an increasing role in industry, provided that the surface engineering techniques will ensure them an improvement of the properties, in particular an increase of their frictional resistance without any deterioration, but even with an improvement, of their good corrosion resistance.

The idea this new methods is to combine one surface treatment, such as thermal spraying, chemical coating deposition, PVD, CVD technique and etc.

In this paper is showed a combination of nitriding with ASP method and laser alloying. This new method gives diffusion layers of various phase composition Ti(ONC) of high resistance.

H – P04 SYNTHESIS AND CHARACTERIZATION OF THE SYSTEM CONTAINING LANTHANUM, CERIUM AND NICKEL WITH PHOTOLUMINESCENCE PROPERTIES

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In This work, the synthesis, characterization and photoluminescence (PL) property of the systems containing lanthanum, cerium and nickel oxides are reported. The systems were prepared from polymeric precursors. The materials were calcined at temperatures ranging from 300 to 800 oC. The formation of crystalline phases upon calcinations was investigated using X-ray diffraction, thermogravimetric analysis, Fourier-transform infrared spectroscopy, and CHN analysis. The results revealed the formation of crystalline phases at 720 C. The photoluminescence (PL) spectra of disordered systems were observed at room temperature. The PL intensity is related to the degree of disorder in the lanthanum, cerium and nickel oxides (S1) caused by the substitution of cerium into lanthanum nickelate system.

H – P047 USING STATISTICAL TECHNIQUES TO DETERMINE THE DEFLOCCULANT EFFICIENCY IN CONCENTRATED CERAMIC SUSPENSIONS

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Ceramic industry has a constant demand for effective dispersants due to their capacity to promote low viscosity at high solids loading. The kind of deflocculant to be used in a particular ceramic system as well as its optimum quantity have to be determined in order to prepare stable suspensions that can produce defect-free bodies. Changes in raw materials or deflocculant compositions lead to adjustments and laboratory tests. Statistical techniques became in the latest years an important tool to decrease the number of experiments involved in products development and reformulation problems. The aim of the present work is to study the effectiveness of sodium tripolyphosphate and sodium polyacrylate as dispersant agent in triaxial

concentrated ceramic compositions using mixture design approach. Suspensions with 60 wt.% were wet ball milled and the deflocculant addition followed industrial procedure. Deflocculants efficiency were analysed using deflocculation curves. Rheological measurements were carried out using coaxial cylinder viscometer, at a shear rate of 42 s-1, at 25.0 ± 0.1ºC. Regression equations, relating the optimum amount of deflocculant and the weight fractions of raw materials were obtained. The results showed that increasing clay mineral amounts lead to increasing deflocculant consumption. Mixture design showed to be a useful tool for planning and analysing experiments and the influence of raw materials on the rheological properties of ceramic suspensions.

H – P048 MAGNETIC AND TRANSPORT STUDIES OF SUPERCONDUCTING RuSr2(GdEu)Cu2O8.

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Superconductivity and magnetism are two different states, which the materials can present at low temperatures and their coexistence has been extensively studied in different kind of compounds. The discovery of the hybrid ruthenocuprates, in which a ferromagnetic-like order at Tm ~133 K precedes a resistive superconducting transition at Tc ~ 45 K, opened a new line of study. In this work it is done a systematic study of the magnetic and superconducting behavior RuSr2(Gd,Eu)Cu2O8 [Ru-(1212)] system. The samples were synthesized by conventional solid state reacting, characterized structurally and micro-structurally by x-ray diffraction and scanning electron microscopy. The magnetic and superconducting properties were studied by magnetization and transport measurements in order to determine the possible role of the magnetic interactions in the conduction and superconducting mechanisms. The possible type of order of the Ru moments is discussed and a mixed valence state was proposed suggests the existence of an intermediate charge state.

H – P049 VITRIFIED RED STONEWARE TILES OBTAINED FROM BY-PRODUCTS AND TAILING OF OIL SHALE MINING

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In the production process of stoneware tiles are used a mixture of quartz and clay minerals in order to obtain a composition that enables conformation by extrusion or pressing. This mixture is made to optimize the firing shrinkage. In the production of red stoneware tiles, some characteristics are very important: low water absorption, high mechanical resistance, high density of sintered compacts and low open porosity. These exigences are reached with a rigorous control in the production process and adequate raw materials selection. Another important factor, in the red ceramic case, is low costs of raw materials. In the oil shale mining and production of main oil shale derived at Petrobrás/SIX-PR are generated a endless number of tailings that can be used in a wide range of industrial segments, such as ceramic products. In this way, the aim of present work is to study some preselected tailings and by-products of oil shale mining and evaluate its viability to apply in red stoneware tiles production. The results shows that is possible to vitrify at a temperature below that ones used in industry, with low water absorption, high mechanical resistance and low linear shrinkage. Besides, a color analysis by means of UV-Vis spectroscopy has allowed to select compositions from lightening index of L coordinate of CIELab system.

H – P050 RELATIONSHIP BETWEEN OPTICAL AND ELECTRICAL PROPERTIES IN Sb-DOPED SnO2

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SnO2 based materials with 5 and 7% Sb2O3 were obtained by physical method of oxide mixture and calcined at temperatures ranging from 800-1500°C. Colorimetric data of CIELab system were deduced from spectroscopy in the UV-vis region. A change from yellow to blue color was found in agreement to higher calcination temperature. Scanning Electron Microscopy of the obtained powders demonstrates an increase in the average grain size associated with an increase in electric conductivity in agreement to higher calcination temperature.

H – P051 STRUCTURAL CHARACTERIZATION OF PHASE TRANSITION OF Cr-DOPED Al2O3 POWDERS OBTAINED BY POLYMERIC PRECURSOR METHOD

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Cr-doped Al2O3 powders have been synthesized by the polymeric precursors method. A study of crystalline phases evolution of obtained powders was accomplished through X-Ray Diffraction, UV-vis spectroscopy and luminescence spectra. The obtained results allows to identify the gamma-Al2O3 to alpha-Al2O3 phase transition. The single-phase alpha-Al2O3 powder was obtained after heat treatment at 1050°C for 2 h. The results relate the color and luminescence effect in function of the cromophore cation concentration and calcination temperature with the phase transition.

H – P052 PHASE TRANSITIONS AND RELATIONSHIP WITH SPECTROSCOPIC ANALYSIS IN CO-DOPED GAMMA-Al2O3 TO ALPHA-Al2O3 + CoAl2O4

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Co-doped alumina powders were synthesized by means of the polymeric precursor method to obtain ceramic pigments. The effect of different contents of Co2+ on phase transition gamma to alpha-Al2O3 and appearing of CoAl2O4 spinel were studied by means of X-Ray Diffraction. A partial phase diagram of the system Co-Al2O3 was proposed from these data by means of determination of the percentages of these phases in according to the calcining temperature. Critical particle size to phase transition was determined by means of calculations of crystallite size and determination of superficial area through of BET method. UV-Vis spectroscopy of the samples allow to compare the band shift with the phase transition. Besides, a study of thermal stability and intensity of the blue coloration of the synthesized powders with the presence of cobalt in relation to the calcining temperature was accomplished and compared to the phase transition. The results shows that the heigher blue color intensity was obtained for the powders with Co-doped gamma-Al2O3 closest of phase transition to alpha-Al2O3 + CoAl2O4.

H – P053 RELATIONSHIP BETWEEN PHASE TRANSITION AND COLOR VARIATIONS IN CERAMIC PIGMENTS OBTAINED BY MEANS OF POLYMERIC PRECURSORS METHOD

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This work aims at studying the color variations in agreement to the calcining temperature and phase transitions of ceramic hosts to which transition metal oxides are added by means of polymeric precursors method. Two systems with different concentrations of transition metals were selected, synthesized (Cr-Al2O3 and Co-SiO2-ZrO2) and submitted to calcining with the temperature ranging from 700 to1200°C. It was observed that Cr-Al2O3 presented a phase transition from g to a-Al2O3 associated with color variations from green to red. On the other hand, Co-SiO2-ZrO2 presented a phase transition from ZrO2 para ZrSiO4 associated with color variations from blue to red. These results have allowed to estimate the phase transition by means of two distinct methods (X-Ray Diffraction and UV-vis spectroscopy) obtaining temperature data compatible for both methods. Besides, it was verified spectroscopic band shifts according to the calcining temperature enabling to predict the cations location in its respective hosts.

H – P054 SILICON NITRIDE CERAMICS OBTAINING FOR STRUCTURAL APPLICATIONS

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This work was developed with objective of to obtain liquid phase sintered silicon nitride ceramics with good physical, mechanical and microstructural properties, seeking to structural applications. Therefore, three mixture powder were prepared: 81.37 wt % Si3N4, 3.20 wt % Al2O3, 10.80 wt % AlN and 4.63 wt % Y2O3, 81.37 wt % Si3N4, 3.20 wt % Al2O3, 10.80 wt % AlN and 4.63 wt % CTR2O3 and 81.37 wt % Si3N4, 3.20 wt % Al2O3, 10.80 wt % AlN and 4.63 wt % Ce2O3. The powder mixtures were homogenized in ethanol, dry at evaporator rotative and kiln, respectively. Then, uniaxially (100 MPa) and cold isostatically (300 MPa) pressed. The samples were sintered at 1900 for 1h, in graphite element resistive furnace under nitrogen atmosphere. After sintering, samples showed relative density equal to 97 % of theoretical, fracture toughness and hardness higher to 16 GPa and 5 MPa.m1/2, respectively. The phase analysis by X-ray diffraction and microscopy electron scanning (SEM) showed only the presence of b-Si3N4.

H – P055

INFLUENCE OF THE SUBSTITUTION OF Y2O3 BY CeO2 IN MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF THE SILICON NITRIDE CERAMICS

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This work was proposed with objective of to analyze feasibility of the substitution of Y2O3 by CeO2 in liquid phase sintering silicon nitride ceramics, seeking to obtain material with good physics, mechanical and microstructural properties, and also to reduce the production costs of the same. Therefore, two powder mixture were prepared: a composed by 78.50 wt % Si3N4, 3.5 wt % Al2O3, 12 wt % AlN and 6 wt % Y2O3; and other by: 78.50 wt % Si3N4, 3.5 wt % Al2O3, 12 wt % AlN and 6 wt % Ce2O3. The powder mixtures were homogenized in ethanol, dry at evaporator rotative and kiln, respectively. Then, uniaxially (100 MPa) and cold isostatically (300 MPa) pressed. The samples were sintered at 1850 for 1h, in graphite element resistive furnace under nitrogen atmosphere. After sintering, samples showed relative density higher to 97 % of theoretical, fracture toughness and hardness higher to 17.12 GPa and 5.28 MPa.m1/2, respectively. The phase analysis by X-ray diffraction and microscopy electron scanning (SEM) showed the presence of a-SiAlON and b-Si3N4.

H – P056

ANALYSIS OF MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF THE SILICON NITRIDE CERAMICS

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This work was proposed with objective of to analyze the mechanical and microstructural properties of liquid phase sintering silicon nitride (Si3N4). Therefore, a powder mixture constituted by 79.90 wt % Si3N4, 11.88 wt % AlN, 4.82 wt % Y2O3 and 3.31 wt % Al2O3 was homogenized in ethanol, dry at evaporator rotative and kiln, respectively. Then, uniaxially (100 MPa) and cold isostatically (300 MPa) pressed. The samples were sintered at 1850 for 0.5h, in graphite element resistive furnace under nitrogen atmosphere. After sintering, samples showed relative density equal to 98 % of theoretical, fracture toughness and hardness higher to 18.90 GPa and 6.24 MPa.m1/2, respectively. The phase analysis by X-ray diffraction and microscopy electron scanning (SEM) showed only the presence of b-Si3N4.

H – P057

ESTIMATE OF THE GRAY CAST IRON MACHINING WITH SILICON NITRIDE CUT TOOLS

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This work was proposed with objetive of to produze and to use silicon nitride (Si3N4) cut tools obtained for the normal sintering process, in the gray cast iron machining. Therefore, Si3N4, AlN, Al2O3, e Y2O3 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 5.5 MP.m1/2 and 20 GPa, respectively. The phase analysis by X-ray diffraction and microscopy electron scanning (SEM) showed the presence of a-SiAlON and b-Si3N4. Before of to be used as cut tools, the samples were grinned in diamond whell in 13x13x4.8 mm, with 20o negative bevel and thickness of the bevel equal to 0.8 mm. Machining tests were realized no cutting fluid, using different conditions. After machining tests, the Si3N4 cut tools pesented suitable results with relationship to machinability of the gray cast iron in different conditions used.

H – P058 MOBILITY OF INTERSTITIAL OXYGEN IN SmBa2Cu3O7 CERAMICS

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Since the discovery of the high Tc superconductors, several works have been made about the different properties of these materials. YBa2Cu3O7-d ceramic showed to have its superconducting properties strongly affected by the oxygen content. For SmBa2Cu3O7 (SBCO), the possibility of variable stoichiometry and the high mobility of oxygen in the CuOx planes gives rise to a rich phase diagram. Anelastic spectroscopy experiments are sensitive tools to the study the mobility of defects in solids. By this technique, we can distinguish the different types of atomic jumps that happen to different temperatures. In order to evaluate the mobility of the oxygen on these oxides, we have prepared ceramic samples and submitted them to several cycles of oxygen charge alternated by anelastic relaxation measurements. The samples were prepared using powder obtained by the conventional method. Structural analyses of the final powder were also made by x-ray diffraction in order to verify the presence of the most stable phase. The anelastic relaxation measurements were performed using a torsion pendulum operating with frequency near 40 Hz in a temperature range between 77 and 700 K, heating rate about 1 K/min. The results shown a thermally activated relaxation peak that was attributed to the jumps of the oxygen atoms in the inter-chains O1 and O5 of the lattice. (Financial support: FAPESP).

H – P059 PHOTOLUMINESCENCE OF Ca0.95Sm0.05TiO3

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Perovskite Ca0.95Sm0.05TiO3 amorphous and crystalline powders were synthesized by polymeric precursor method. Ca0.95Sm0.05TiO3 powder was pre-calcined at 300 0C for 20 hours in an oxygen flow to promote oxidation of the organic matter and pre-pyrolysis without crystallization. After pre-calcined, the materials was submitted a heat treatment at different temperatures for 3 hours in an oxygen flow for study of its photoluminescence (PL) properties. The Ca0.95Sm0.05TiO3 were characterized structurallly using X-ray diffraction, raman and infrared spectroscopy. The PL measurementes were taken at room temperature using 488.0 nm exciting wavelenghts of an argon ion laser.

Our results had shown that the PL properties is sensitive to its thermal treatment, therefore changes in the calcination temperature provoke changes in the intensity of the PL. The increased structural order is responsible for the decrease in the intensity of the PL. The intensity of the PL increased gradually for the powders calcineds at 350 and 400 0C, and for the powders calcined at 450, 500, 550 and 600 0C the intensity of the PL decreased gradually. For the powder calcined at 600 0C only was observed the properties PL of the present Samarium in the structure.

H – P060 STRUCTURAL AND OPTICAL PROPERTIES OF ZnGa2O4 PREPARED BY THE MODIFIED PECHINI METHOD

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Endowed with luminescence properties capable of being tuned by heat treatments under reducing or oxidizing atmosphere, zinc gallate (ZnGa2O4) displays also UV transparency. Despite its variety of applications, ZnGa2O4 has been obtained by the traditional solid-state route, that is, heat treatments of high temperatures and long times. In this work it is reported the synthesis of ZnGa2O4 by modifying the Pechini method with the use of ethylenediaminetetraacetic acid. A fine powder was achieved after firing the resin at 900ºC for only 4 hours under air atmosphere. ZnGa2O4 with the spinel crystalline structure was observed, along with a slight distortion of the Ga-O octahedra unveiled by means of refining its diffraction data through the Rietveld method. A bond length of 1.997 Å was found for Ga-O and 1.961 Å for Zn-O with lattice constant equal to 8.338 Å. This distortion, characteristic of reduced ZnGa2O4, gave rise to an interesting effect on its optical properties. Besides the blue emission in 430 nm assigned to charge transfer between Ga3+ at octahedral sites and O2- ions, an emission in 710 nm is also observed due to the presence of single oxygen vacancies. In spite of its wide band gap (around 5.0 eV as estimated by its diffuse reflectance spectrum), which usually requires excitation at 250 nm, the emissions referred to earlier were observed under 370 nm excitation. Modifying the Pechini method enables a low-temperature synthesis of ZnGa2O4 with novel optical properties.

H – P061 COLORIMETRIC AND MICROSTRUCTURAL CHARACTERIZATION OF PORCELAINIZED STONEWARE TILES USING GRANITE WASTE

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The porcelainized stoneware tiles are a type of ceramic tiles that are differed from the others due to its highly technological production process resulting in products with low porosity (<0.5%) and high technical performances. The use of granite waste has the objective of providing some technical utilization for an industrial waste and also influencing on the product colour. Several compositions were formulated and this colour evaluated using spectroscopy of UV-Vis and CIELab colorometric system in which the best value for colorimetric coordinated L * was 79.92. The microstructure characterization of the optimised composition evidenced the formation of secondary mullite well crystallized. The study of the substitution influence of the feldspar for granite residue showed that the last one could be incorporated up to 15%. In this case, the increment of the granite residue decreases the absorption of water, the apparent porosity, the linear shrinkage, and the colorimetric coordinate L * assumes the value 72.76.

H – P062 ADDITION OF POLYURETHANE RESIDUES FROM THERMO-ACOUSTIC SHINGLE INDUSTRY TO PORTLAND CEMENT

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ASTM class I Portland cement is by far the most widely used material in civil construction and oilwell cementing. Although Portland cement develop compressive strength adequate for structural applications, limited tensile strength and high permeability are normally observed. The former aspect usually implies in reduced fracture toughness and increased brittleness of the cement, whereas the latter characteristic favors the penetration of corrosive species, which may have deleterious effects on the structural rebars. The addition of polymeric materials may potentially improve the plastic behavior of the cement as well as reduce its permeability. In this study, polyurethane residue from the industrial production of thermo-acoustic shingles was added to Portland cement. Composite slurries containing up to 5 wt.% polyurethane were mixed, homogenized, hardened, and characterized. Rheological and mechanical tests were carried out along with permeability measurements. The tensile strength of the composites was evaluated from diametral compressive tests. The results revealed that the addition of up to 3 wt% polyurethane improved both the diametral compressive strength of the cement as well as its fracture toughness. Both permeability and sedimentation were reduced upon the addition of the polymer. Shorter set times were obtained from composite samples compared to plain cement paste. Finally, the addition of polyurethane increased the plastic viscosity and yield stress of the material.

H – P063 NbC- AND TaC-REINFORCED SINTERED STEEL: DENSIFICATION AND MICROSTRUCUTURAL DEVELOPMENT

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Sintering steels reinforced by hard ceramic particles (NbC and TaC) have been manufactured to match the performance of conventional tool steels. Ceramic reinforced steels have been sintered to near full density avoiding the intrinsic processing difficulties encountered when sintering tools steels, such as limited sinterability and narrow densification ranges. The present study shows the effects of the nature of iron powder and contents of reinforcing phase on the dilatometric profiles of Fe-NbC and Fe-TaC composites. Compositions with up to 20 wt.% NbC or TaC were milled, annealed and sintered in a dilatometer under hydrogen. A liquid phase additive was used in compositions prepared from plain iron. Alternatively, the use of pre-alloyed iron powder was also studied. In the latter case, sintering took place by solid state. The results showed that composite particles were preferentially formed in plain iron by the insertion of carbide particles into iron particles resulting in homogenous microstructures, and, therefore better overall mechanical properties. Sintering at 1180°C for 30 min produced densities in excess of 96% TD. Pre-alloyed iron particle are smaller and harder than plain iron limiting the formation of composite particles.

H – P064

CORRELATION BETWEEN MICROESTRUCTURE AND PHYSICAL-MECHANICAL PROPERTIES OF CERAMIC MATERIALS OF CLAY

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The microstructure of materials is an important characteristic in the characterization and in the final properties of a product. By means of the microstructural analysis it is possible to verify porosity, average size of particles, and when processed thermically, growth of grains and transformation of phases. For ceramic materials of clay, the microstructural analysis is still little used, perhaps for the complexity of such materials. In the specific case of this research line, during the characterization of the clay it tries to indicate an ideal temperature of use, or either, temperature in which the sintering provides the development of products inside of the specifications, that normally is made by the physical-mechanical properties. However, some few studies already relate the microstructure to the final properties. In this context, this work has as main objective to contribute for the study of this correlation between microstructures and final properties of ceramic materials. For this, ceramic bodies were developed starting from clay, varying it temperature of sintering, in order to compare its microstructures, for microscopy optics and electronics of scanning. Results indicate that the biggest variation in the properties was also observed in the biggest variation of the microstructures, where are possible with the increase of data of the comment of the microstruture to correlate the temperature of sintering with the ideal microstruture.

H – P065

INFLUENCE OF SiC CONTENT IN MICROSTRUCTURE OF THE ALPHA-SiAlON-SiC COMPOSITE OBTAINED BY NORMAL SINTERING

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In this work were evaluated the influence of SiC content at the relative density and microstructure of the alpha-SiAlON-SiC composite. Aspects microstructurals of the main phase (alpha-SiAlON); as net parameter, morphology, average size and aspect ratio of the grains also were analized due to the SiC content. Therefore, AlN-CTR2O3 (9:1 mol %) system was used as additive, for to obtain alpha-SiAlON as main phase. In the α-SiAlON-SiC composites developed in this work, the SiC content varied of 0 to 20 wt %. The powder mixtures were homogenized, then compacted by uniaxial and isostatic cold pressing, respectively. The samples were sintered at 1950oC for 1h in nitrogen atmosphere (1 MPa N2). Relative density decreases with increase of the SiC content, whereas weight loss shows inverse behavior. The α-SiAlON and β-SiC phases were identified for X-ray diffraction and Scanning Electron Microscopy (SEM). Net parameter of the a’ phase didn’t suffer considerable alterations with increase of SiC content. However, morphoogy, avarege size and aspect ratio of the a’ phase were considerably changed by rise of the SiC content.

H – P066 ON THE PHASE TRANSFORMATIONS DURING THE HEAT TREATMENT OF MECHANICALLY ALLOYED Ti-30Si-5B AND Ti-23.5Si-10B POWDERS

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Binary alloys containing the intermetallic phase Ti5Si3 have been considered for high temperature structural applications due to their physical, chemical, and mechanical properties such as high melting point, oxidation resistance and good strength/weight ratio. The isothermal section at 1250oC of the ternary Ti-Si-B system was recently determined and two-phase alloys Ti6Si2B+Ti5Si3 can be produced. The present work reports on the phase transformations during the heat treatment of mechanically alloyed Ti-30Si-5B and Ti-23.5Si-10B (at-%) powders. The following high purity powders were used in these experiments: Ti (99.9wt-%, spherical, <150mesh), Si (99.99wt-%, irregular, <120mesh) e B (99.5wt-%, irregular, <120mesh). The milling process was carried out in a planetary Fritsch P-5 ball mill under argon atmosphere using stainless steel vials (225mL), rotary speed of 200rpm, hardened steel balls of 19mm diameter, and a ball-to-powder weight ratio of 10:1. In the sequence, mechanically alloyed powders were heat treated at 1100oC for 4h under inert atmosphere. To understand the phase transformation during milling and heat treatment, powder samples were selected and characterized by means of X-ray diffraction (XRD), scanning electron microscopy (MEV) and microanalyses via EDS. Results indicated the dissolution of boron and silicon atoms to form a supersaturated solid solution of titanium after milling for 100h. After heat treatment at 1100oC for 4h, the microstructures of the Ti-30Si

H – P067 HIGH ENERGY BALL MILLING OF POWDER Ti-Nb MIXTURES

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Free-vanadium and aluminum titanium alloys have been adopted for medical and dental applications due to their toxic features. The addition of niobium, tantalum, and/or zirconium elements contributes to enhance the biocompatibility characteristics and reduce elastic module. The present work reports on the preparation of Ti-Nb powders by high-energy ball milling and further heat treatment at 1200oC for 4h, varying the Nb amount between 20 and 50wt-%. The milled and heat-treated powders were characterized by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), and microanalysis via EDS techniques. The computer Rietveld program was used to determine the lattice parameters of phases in milled Ti-Nb powders. It was noted after milling for 20h, an intense cold-welding mechanism of ductile powder particles on the balls and inner vial surfaces, reducing the final amount of remaining powders into the vial. The cold-welding mechanisms were detrimental with higher niobium amount added. As expected, the dissolution of niobium into the Ti lattice to form a supersaturated solid solution has occurred after milling for 20h. The fine and homogeneous microstructures TiSS(Nb) were formed in Ti-Nb (Nb-20-50wt-%) powders after heat treatment at 1200oC for 4h.

H – P068 CHARACTERIZATION OF MECHANICALLY ALLOYED AND HEAT-TREATED Ti-7.5Si-22.5B AND Ti-18Si-6B POWDERS

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Recently, it was noted the existence of a new ternary phase in the Ti-Si-B system with composition near Ti6Si2B. In addition, three-phase alloys TiSS+Ti6Si2B+TiB and TiSS+Ti6Si2B+Ti5Si3 could be produced. The aim of this work is present results on the phase transformations of mechanically alloyed and heat treated Ti-7.5Si-22.5B and Ti-18Si-6B (at-%) powders. High purity starting powders were used in these experiments: Ti (99.9wt-%, spherical, <150mesh), Si (99.99wt-%, irregular, <120mesh) and B (99.5wt-%, irregular, <120mesh). The milling process was carried out in a planetary Fritsch P-5 ball mill under argon atmosphere using stainless steel vials (225mL), hardened steel balls of 19mm diameter, rotary speed of 200rpm, and a ball-to-powder weight ratio of 10:1. In the sequence, the milled powders were heat treated at 1100oC for 4h under inert atmosphere. The mechanically alloyed and heat treated powders were characterized by means of X-ray diffraction (XRD), scanning electron microscopy (SEM) and microanalyses via EDS techniques. It was noted during milling the dissolution of boron and silicon atoms into Ti lattice to form a supersaturated solid solution. The TiSS+Ti6Si2B+TiB and TiSS+Ti6Si2B+Ti5Si3 phases were formed in heat-treated (1100oC for 4h) Ti-7.5Si-22.5B and Ti-18Si-6B powders, respectively.

H – P069 ON THE IRON CONTAMINATION IN MECHANICALLY ALLOYED Cr-Si POWDERS

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Silicides of refractory metal have been considered for high temperature applications due to their physical, chemical, and mechanical properties such as high melting point, oxidation resistance, and good strength-weight ratio. The present work reports on the iron contamination in mechanically alloyed and heat-treated (1200oC for 4h) Cr-25Si, Cr-37.5Si, Cr-50Si, and Cr-66.6Si powders (at-%). High-purity starting powders were used for the preparation of powder Cr-Si mixtures: Cr (99.9, angular, < 120mesh), and Si (99.995, irregular, < 120mesh). The milling process was carried out in a planetary Fritsch P-5 ball mill under argon atmosphere using stainless steel vials (225mL), rotary speed of 200rpm, hardened steel balls of 19mm diameter, and a ball-to-powder weight ratio of 10:1. Cr-Si powders milled for 200h were heat-treated at 1200oC for 4h using inert atmosphere. The milled and heat-treated powders were characterized by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), and microanalysis via EDS techniques. The intensity of Si peaks was reduced during earlier milling stage. After milling for 200h, the Si peaks disappear indicating that the mechanical alloying process was achieved. Binary phases of the Cr-Si system were identified in heat-treated powder samples, but an intense iron contamination lower than 20at-% was also detected, and a ternary phase was formed.

H – P070 HIGH ENERGY BALL MILLING OF POWDER B-C MIXTURES

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Boron carbides have long been known as high temperature thermo-electrics due to their attractive physical and mechanical properties such as very high melting point (2400 K), high carrier density, extreme hardness, and high stiffness. According to the assessed B-C phase, the B4C phase exists in a composition range between B-10C and B-20C (at-%). The present work reports on the preparation of powder B-10C and B-18C (at-%) mixtures by high-energy ball milling and further heat treatment. The milling process was carried out in a planetary Fritsch P-5 ball mill under argon atmosphere using stainless steel vials (225mL), rotary speed of 150rpm, hardened steel balls of 10mm diameter, and a ball-to-powder ratio of 10:1. Powder samples were heat-treated at 1200oC for 2h using inert atmosphere. The milled and heat-treated B-10C and B-18C powders were characterized by means of X-ray diffraction (XRD), and scanning electron microscopy (SEM) techniques. It was noted that the intensity of B and C peaks was reduced after milling for 2h, which can be associated to reduction of particle sizes due to the intense fracture mechanisms during ball milling of brittle starting components. In both the powder B-10C and B-18C mixtures, phase transformations have occurred after milling for 6h, and a metastable phase was formed. After milling for 90h, only peaks of boron and this metastable phase were presents. Results on the heat-treated powders will be also presented.

H – P071 DEVELOPMENT OF CERAMIC PIGMENTS OF THE CuCr2O4 BY PECHINI METHOD

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The ceramic pigments are inorganic substances constituted by a ceramic matrix of crystalline nature and an coloration responsible element that is thermally stable, insoluble in the glazings, resistant to you attack chemical and physical and that doesn\'t produce emission of gases. Most of the ceramic pigments consists of substances that possess a certain crystalline structure, as it is the case of the spinel structure, tends as example the chromite, base of our experiments. With the objective of obtaining ceramic pigments of black color, the method Pechini was used in the preparation of CuCr2O4, being made the thermal treatment in appropriate temperatures. The powders pigments were characterized in the aspects thermal, structural and morphologic. In TG/DTA the thermal evolution of the powders was observed. XRD allowed the observation of the crystalline evolution of the same ones. In the morphologic characterization for Adsorption of Nitrogen for the method BET, was certain the specific superficial area of the powders.

H – P072 SURFACE CHARACTERIZATION OF PLASMA SINTERING UNALLOYED IRON ENRICHED WITH DEPOSITED NICKEL: CORROSION DEPENDENCE FROM POROSITY AND SURFACE COMPOSITION

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One of the largest limitations of materials achieved from ferrous powder metallurgy is their low corrosion resistance, due to the presence of pores. The plasma sintering thermal treatment is an alternative to improve the corrosion resistance. Two different treatments were used: the deposition of a nobler element as iron, in the surface of the material during sintering and the reduction of the superficial porosity by impact of energy particles, activating the atomic diffusion in the surface. The electrical discharge was generated in gaseous mixture of 80%Ar-20%H2 and the sintering was processed at 11500C during 60 minutes. Ni was deposited in the anode-cathode confined configuration, with the sample placed in the anode, in order to obtain the surface enrichment, since Ni is frequently used to reduction of corrosion. The reduction of the superficial porosity was obtained placing the sample in the cathode, where it suffers highly energetic collisions by ions and fast neutrals, which are strongly accelerated in the cathode sheath. The samples were characterized using electrochemical techniques. The results evidenced a reduction in the corrosion rate due to Ni surface enrichment, obtained during sintering, and due to the decreasing of superficial porosity.

H – P073 CHROMATIC AND PHOTOLUMINESCENCE PROPERTIES OF SPINEL Li2ZnTi3O8 NANOMETRIC DOPED WITH Pr3+

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The synthesis of materials ceramic nanometrics with magnetic properties, superconducting, luminescent and chromatic he/she has a lot of relevance for applications in the industry of pigments and electronics. Among these materials they stand out the spinel, because they possess the capacity to accommodate many cations in your structure allowing applications of great technological interest, such as, pigments (CoAl2O4), refractory (MgAl2O4), among others. A modified polymeric precursor route based on the Pechini method synthesized the spinel Li2ZnTi3O8 doped with Pr3+. X ray powder diffraction analysis, Rietveld refinements, Raman spectroscopy, UV-visible spectroscopy and photoluminescence measurements were used to study the chromatic and photoluminescence properties. The experimental results demonstrated that the spinel is nanometric and it presented properties photoluminescence to the ambient temperature and coloration yellows, and that these properties are influenced by the entropy of the system (presence and absence of the species TiO5 and TiO6) and the presence of the ion rare earth in the crystalline structure of the spinel.

H – P074 PHOTOLUMINESCENCE OF Ca0.95Sm0.05TiO3 PEROVSKITE-TYPE

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Perovskite Ca0.95Sm0.05TiO3 amorphous and crystalline powders were synthesized by polymeric precursor method. Ca0.95Sm0.05TiO3 powder was pre-calcined at 300 0C for 20 hours in an oxygen flow to promote oxidation of the organic matter and pre-pyrolysis without crystallization. After pre-calcined, the materials was submitted a heat treatment at different temperatures for 3 hours in an oxygen flow for study of its photoluminescence (PL) properties. The Ca0.95Sm0.05TiO3 were characterized structurallly using X-ray diffraction, raman and infrared spectroscopy. The PL measurementes were taken at room temperature using 488.0 nm exciting wavelenghts of an argon ion laser. Our results had shown that the PL properties is sensitive to its thermal treatment, therefore changes in the calcination temperature provoke changes in the intensity of the PL. The increased structural order is responsible for the decrease in the intensity of the PL. The intensity of the PL increased gradually for the powders calcineds at 350 and 400 0C, and for the powders calcined at 450, 500, 550 and 600 0C the intensity of the PL decreased gradually. For the powder calcined at 600 0C only was observed the properties PL of the present Samarium in the structure.

H – P075 SOL-GEL SYNTHESIS AND PROPERTIES OF Co2+-DOPED SILICA GLASSES

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The present study describes the properties and structure of cobalt-doped silca gel glasses. Silica gels containing 1 mol % cobalt oxide were prepared by hydrolysis of tetraethoxylsilane (TEOS) and two different inorganic precursors: cobalt chloride and nitrate. Formamide, N,N-dimethylformamide, ethylene glycol and propylene carbonate were used as additives on the sol-gel solutions preparation. Starting solutions were prepared with H2O/TEOS molar ratio equal to 3, in acid condition. The dried gels were thermally treated at 400 and 700 oC. The influence of the different drying control chemical additives on the structural evolution of the gels was investigated. The thermal evolution process of the gels were examined by means of DTA, infrared spectroscopy, UV-VIS optical transmission, physical adsorption measurements, and X-ray diffraction technique. The presence of the additives increases the values of the specific area, for the gels heated to 400 oC, to values varying from 400 to 900 m2/g. The thermal evolution of the gels leads to incorporation of Co2+ ions in tetrahedral coordination.

H – P076 LOW TEMPERATURE UP-CONVERSION SPECTROSCOPY OF NANOSIZED Y2O3:Er,Yb PHOSPHORS

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Up-converting phosphor compounds, that emit visible light under near-infrared (NIR) excitation, form an alternative to conventional luminescent reporters and are increasingly used in immunoassays. The use of IR excitation instead of UV one avoids any background luminescence from the assay biochemistry. In this context, nanosized Er/Yb codoped Y2O3 phosphors represent an interesting system to be investigated in order to evaluate its applicability in immunoassays. This work reports on the structural characterization and on the low temperature upconversion spectroscopy of Y2O3:2%Er,1%Yb nano-phosphors prepared by thermal decomposition of polymeric resin (Pechini´s method). The average particle size evaluated from TEM lies in the range of 20-30 nm. The high-resolution upconversion luminescence spectrum at 10 K in the UV-NIR spectral region shows narrow lines characteristic for Er3+ transitions occupying both Y3+ sites with point symmetry C2 and C3i in the oxide cubic system. The excitation spectra at 10 K in the IR region monitoring the green upconversion and IR luminescences show Er and Yb lines. Power dependence measurements at room temperature and 10 K indicate that the main upconversion mechanism is a two-photon excitation process. The temperature dependence of the upconversion luminescence shows a decrease of emission intensity with increasing of temperature, which is much more evident for the emission in the UV than in the visible and the NIR. Acknowledgement: FAPESP

H – P077 MAGNETIC BEHAVIOR OF NANOCRYSTALLINE MnCo2O4 SPINELS

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A serie of powders with different particle sizes of the spinel MnCo2O4 was synthesized by the Pechini method. Manganase citrate and cobalt citrate were used to prepare a polymeric resin with citric acid and glycol ethylene. The resultant precursor was calcined between 300±C and 900 ± ºC and characterized by TGA, XRD and magnetization measurements. TGA analysis showed a non crystalline region from 200 to 650±ºC and crystallinity after this. X-ray diffraction pattern also confirm this results. Magnetization measurements on the crystalline samples shown an unusual behavior of the hysteresis curves and irreversibility of the magnetization with Field Cooled (FC) and Zero Field Cooled (ZFC) procedure. An evolution of this behavior is observed with increasing of the crystal size.

H – P078 PROPERTIES VISCOELASTIC OF THE THERMOREVERSIBLE SOL-GEL TRANSITION OF SULFATE MODIFIED ZIRCONYL CHLORIDE AQUEOUS SOL

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The thermoreversible sol-gel transition in the system prepared by mixing a ZrOCl2 acidified solution to a hot H2SO4 aqueous solution, were studied by dynamic rheological measurements and quasi-elastic light scattering. The effect of the temperature and of the molar ratio [Zr+4]/[SO4-2] on the kinetics of gelation were analyzed. The viscoelastic properties of these suspensions showed a progressive transformation of the sol at the initial gelation step from the Newtonian viscosity to a viscoelastic behavior. The rheological properties were correlated to mass fractal, nearly linear growth models and percolation theory. This study shows that the linear growth of aggregates occurs at the first period of transformation, while the growth of fractal structures occurs at the intermediate stage. The increase of the temperature leads to a reduction of the period in which the particles growth occurs, favoring the experimental observation of the fractal growth regime. Near the gel point these samples have the typical scaling behavior expected from percolation theory.

H – P079 SYNTHESIS, STRUCTURAL AND SPECTROSCOPYC STUDIES CeO2 – ZnO NANOSIZED POWDER SYSTEMS AS SOLAR UV FILTERS

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Fine particles of TiO2 and ZnO have been used as effective inorganic sunscreens, but make the skin look unnaturally white due to their high refractive index. Recently, CeO2 has been proposed as an UV blocking material, especially as a sunscreen in cosmetics. The introduction of ZnO should reduce the oxidation catalytic activity. So, nanosized CeO2-ZnO systems have been investigated in order to improve such application. This work reports on the synthesis from Pechini method and characterization of CeO2:ZnO systems in appropriate ratio (1:4, 2:3, 3:2 and 4:1), as well as the pure oxides, searching for nanosized powders with potential application as UV filters. The compounds were obtained by heating the polymeric precursors under 900oC/4h (Pechini method). The crystalline phase was identificated by X-ray diffraction and the size distribution and shape were observed using transmission and scanning electron microscopies. UV-VIS absorption and diffuse reflectance spectroscopies were performed in order to evaluate the optical behavior of the systems. The results indicate that CeO2–ZnO nanopowder systems with high opacity in the UV region and high transparency in the visible can be successfully prepared by Pechini´s method. Therefore, they are promising candidates to be applied as UV filter cosmetics in personal care products. Acknowledgements: FAPESP

H – P080 MONOLITHIC MESOPOROUS ZIRCONIA PREPARED BY SOL-MESOPHASE-GEL SYSTEM

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In this work we developed an inedited new route for preparation of monolithic mesoporous zirconia, using a sol-mesofase-gel system. Hexagonal-2D swollen liquid crystal (SLC) systems were developed with salt solution as polar phase, surfactant, co-surfactant and organic compound as a micelle-swelling agent. We established the conditions for stabilize the SLC as a function of pH and surfactant\'s nature. In a second time, we studied the formation of the hexagonal-2D SLC prepared with sulfated zirconium oxyhydroxide colloidal suspension as a polar phase. We analyzed the influence of the surfactant\'s nature and the organic compound\'s nature in the stabilization of the SLC and the formation of mesoporous zirconnia. The mesoporosity was studied as a function of gelification conditions, the pore\'s size control and porosity was analyzed as a function of polar phase\'s volume. The SLCs were characterized by SAXS experiments that confirm the formation of hexagonal-2D SLC based salt solution with cell parameter of 290 Å and hexagonal-2D SLC based colloidal suspension with cell parameter of 272 Å. The SAXS experiments and N2 adsorption-dessorption isotherms of the zirconia burned at 400ºC show that mesoporous zirconia was obtained with a hexagonal pore array, narrow pore size distribution, pore diameter between 20 and 55 Å and specific surface between 70 and 233 m2.g-1.

H – P081 STRUCTURE AND SINTERING OF NiZn FERRITE NANOPOWDERS

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The structure of NiZn ferrite (Ni0.5Zn0.5Fe2O4) ultrafine powders and the influence of the particle size on the sinterability of this material have been investigated. The ferrite nanopowders were obtained by coprecipitation and heat treatment varying between 300 and 600oC. X-ray diffraction (XRD) was used to follow the structural evolution of the samples and to determine the average particle size (D). Small-angle X-ray scattering (SAXS) experiments were performed in order to obtain additional information on particle size and surface characteristics. The sintering behavior of ceramic bodies obtained from the nanometric powders was investigated by using scanning electron microscopy (SEM) and helium picnometry. XRD patterns show the evolution of the powders crystallinity with heat treatment. We observed that the average particle size increases with annealing temperature, reaching a maximum value of 31 nm. SAXS results show scattering curves characteristic of smooth particle interfaces. Volume size distribution functions of the scatters have a wide dispersion, centered in values that increase with the heat treatment, indicating the same trend of XRD results. As observed by SEM, sintering at 1250oC for 4h resulted in densification process without appreciable grain growth for sample with D = 22 nm, and remarkable grain growth for D = 16nm. We show that our ferrite nanopowders present significantly better sinterability than conventional powders.

H – P082 EVALUATION OF THE STRUCTURAL PROPERTIES SrTiO3-BASED CERAMIC PIGMENTS

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In the last years has been growing the interest in the syntheses of oxides with perovskite structure of the STO type, aiming at the use in electro-electronic ceramics, and in other industrial applications. STO systems present as the main characteristic a n-type semiconductor what leads to their application in the production of the thin films, DRAM\'s systems (as thy present a high dielectric constant and a good capacitance at the applied voltage) and also in the ceramic pigment area. Strontium titanate doped with cobalt has been shown as a good pigment, mainly for its application in ceramic coating, due to its good thermal stability and easy industrial reproducibility. In this work were obtained, by the polymeric precursor method, samples of MxSr1-xTiO3 and MxSrTi1-xO3 (M= Co and 0£x£20 mol %), with the purpose of evaluating their characteristics as ceramic pigments. All studied powders were heat treated at a temperature ranging from 600 to 1000ºC for 1 h and were characterized by TG/DTA, DRX, colorimetry, Infra-red and SEM. By this synthesis method were produced homogeneous and single phase powders containing up to 10% in mole of cobalt. Based on the XRD results were determined the lattice parameters, the crystallite size and the relative crystallinity. All these results show that the formation of ionic defects of the type of oxygen vacancies and metal vacancies are the main responsible for the alterations of the final properties of each individual sample.

H – P083 VISIBLE PHOTOLUMINESCENCE OF ULTRA FINE POWDER SILICA GLASS

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Intense photoluminescence was observed at room temperature in soda-lime-silica glass powder, mechanically milled in a high-energy attrition mill. The emission band maximum in the visible region shows an interesting dependence on the exciting wavelength, suggesting the possibility of tune the PL emission. These findings, very similar to that reported on highly disordered ferroelectric materials with perovskites structure, indicate that the photoluminescence may be directly related to unsatisfied chemical bonds correlated with the high surface area. The Raman scattering and ultraviolet-visible optical reflectance measurements corroborate this assertion. Transmission electron microscopy measurements indicate that samples milled more than 2 hours present the formation of nanocrystallites with about 5-10 nanometers. Increasing the milling time increases the population of the crystallites, preserving their dimensions.

H – P084 STRUCTURAL AND MORPHOLOGICAL CHARACTERIZATION OF FERRITES OBTAINED BY THE POLYMERIC PRECURSOR METHOD

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In the present work are presented results of the synthesis and characterization of nanometric ceramic pigments, using the polymeric precursor method. These pigments, spinels displaying the formula AB2O4 should present a good stoichiometric control, a controlled particle size, absence of secondary phases, etc. MFe2O4 (M=Zn or Co) ferrites were synthesized. After obtaining the polymeric resins and after the heat treatment at 300ºC for 1h, the precursor powders were ground in a high energy mill and soon after they were heat treated, initially at 300ºC for 12 h and in a second stage at the 600-1000ºC temperature range for 1h. The precursor powders obtained at 300ºC for 1h were characterized by thermogravimetry (TG), presenting two thermal decomposition stages referring to the exit of water, adsorbed gases and loss of organic matter (pyrolysis). They were characterized by differential thermal analysis (DTA), exhibiting exothermic transitions due to the pyrolysis process. All the obtained powders were also characterized by XRD for the correct identification of the crystalline phases, measurement of the crystallite size, relative crystallinity and the lattice parameters. It was noticed from the diffraction patterns that both the cobalt ferrite and the zinc ferrite are single phase. SEM was employed for the morphologic evaluation. The lattice parameters herein obtained were very close to the theoretical values found in JCPDF records. CoFe2O4 presents bigger crystallites than ZnFe2O4.

H – P085

EFFECT OF MECHANICAL ACTIVATION IN THE STRUCTURE AND NANOSTRUCTURE ON SOLID STATE FORMATION OF BaTiO3

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The BaTiO3 (BT) is widely used due to its high dielectric constant, ferroelectric properties and positive temperature coefficient of electrical resistivity (PTCR). This work presents the results of BT synthesis by mechanical activation chemistry and the characterization of the powders of the Rietveld refinement/MEM and the x-ray diffraction (XRD). The powders BaCO3 and TiO2 were mechanically milled and calcined by 2 and 4 hours. Crystal structure, nanostructure and quantitative phase analyses of the crystalline phases were analyzed before and after the calcination. It was verified about 1 wt% of BT after 5 hours of grinding. This proportion practically does not change with the grinding time of 10 and 20 hours. After calcination it is verified that the increasing of the grinding time generates crystallites tending to isotropics. The increase in calcination time causes the decrease of the crystallite size in c-axis direction and the increase in the a and b-axis.

H – P086

THE INFLUENCE OF CLAY CONTENT ON THE MECHANICAL PROPERTIES OF MELT INTERCALATED PP/BENTONITE NANOCOMPOSITES

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Nanocomposites are an important new class of materials being increasingly studied. These materials display superior mechanical and barrier properties at low filler contents. Melt intercalation is one of the most popular methods to obtain polymer nanocomposites. In the present work, the influence of clay content on the mechanical properties of polypropylene/bentonite nanocomposites was determined. A naturally occurring sodium bentonite from Wyoming, provided by BUN-Campina Grande/PB was employed as a filler. This bentonite was chemically modified with na alquil dimethyl benzyl ammonium (Dodigen) salt. The organophilized clay was characterized by FTIR and X-ray diffraction (XRD).The results show that an organophilic clay was effectively manufactured. This clay was added to a polypropylene matrix (PP-H301), provided by Braskem, by melt intercalation in a twin-screw counter rotating extruder coupled to a System 90 Haake Torque Rheometer. Processing conditions were 210ºC and 60 rpm. The samples were air cooled to room temperature, ground-up, compression moulded and tensile tested. These systems were also characterized by XRD which showed that nanocomposites were indeed obtained. Better mechanical properties were achieved at lower clay concentration (1%). Organophilization with the Dodigen salt was efficient in promoting better tensile properties for this nanocomposite.

H – P087

SYNTHESIS AND CHARACTERIZATION OF GdVO4:Tm BLUE PHOSPHOR VIA COMBUSTION METHOD

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Oxide phosphors have recently gained much attention for applications in plasma and field emission displays panels and for white color light-emitting diodes due to their higher chemical stability compared to others phosphors such as sulfides. However, Tm-activated phosphors in powder form are not yet in practical use in any emissive displays and must be more investigated. So, this work reports on the preparation of high-luminance blue-emitting Tm-doped multicomponent oxide phosphor. Tm-doped (0.25 to 1 % w/w) gadolinium vanadate samples were prepared via combustion method using citric acid, glycine or urea as fuels. The obtained multicomponent oxide samples were post annealed in air at 800 to 1100 ºC, 4 hours. X-ray patterns show that in general the GdVO4 lattice is the majority phase, although with the presence of V2O5 and Gd2O3 as impurities. Nevertheless, the oxide mixture phosphors produced exhibit high photoluminescence intensity in the blue (sharp peak at 475 nm) under UV excitation (305 nm). Therefore, XRD and PL results, combined with scanning electron microscopy (SEM) observation can be used to establish the best synthesis condition in order to provide a blue oxide phosphor with size and shape control. Acknowledgements to FAPESP for financial support.

H – P088

PREPARATION OF MESOPOROUS CARBON BY SODIUM ACETATE AS ACTIVATING AGENT FOR COCONUT POWDER FIBER

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Structural and adsorptive properties of carbon (AC) depende on the kind of precurssors, synthesis and activation condition, which result in nonporous, mesoporous, microporous, biporous active materials. Carbon with a larger contribution of mesoporosity can be interest for some applications but their preparation can be a difficult task. The aim of this work had been to preparer mesoporous carbon using powder of coconut fibres and CH3COONa and ZnCl2 solid as activating agent. The materials were pyrolised at 800 oC with a rate heating of 10 ºC/min, under nitrogen, subsequentely changed to CO2 for 2 h. The samples were washed until elimination of ZnCl2 and CH3COONa. To characterize the carbon were used X-RD, TGA/DTA, SEM, porosity and surface area measurements, UV-Vis spectroscopy to the adsorption studies. The AC showed isotherms type IV with hysteresis H2, which incidate a high mesoporosity. The morphology of the AC powder is similar to the natural coconut powder. It means that during the pyrolisis the powder morphology is preserved. The IR spectrum of the AC using sodium acetate as activating forms aromatics compounds, while the AC obtained with ZnCl2 form aromatics and esters. The TGA/DTA of the coconut powder mixed to CH3COONa show a pyrolisis with a higher decomposition temperature, 920-1300 K, while in the pyrolisis using ZnCl2 the maximum decomposition temperature is at 820 K. Adsorption studies of dye compounds confirm the mesoporosity of the AC.

H – P089

SYNTHESIS AND CHARACTERIZATION OF YTTRIA-STABILIZED ZIRCONIA NANOMETRIC POWDER

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In this work yttria-zirconia nanopowder was synthesized by the Pechini process with aim to obtain the tetragonal phase of zirconia. The Pechini method make possible the formation of powders with particles of nanometric sizes, being necessary an effective control during the synthesis and processing stages of the powder. Among the benefits of the use of nanometric powders can be mentioned the decrease of the sintering temperature and ceramic bodies with more homogeneous and dense microstructures. The powder characterizations were done by TG-DTA analyses to determine the mass loss and the formation of the crystalline phases. The BET technique determined the specific area and the BJH method supplied the pore size distribution. Particle size and shape were examined by SEM observations and X-ray diffraction analysis was used for phase identification. The compaction behavior of calcined powder was studied by measuring the changes in density with increased uniaxial pressure.

H – P090 CHARACTERIZATION AND PACKING BEHAVIOR OF NANOSIZED ALUMINA POWDER OBTAINED FROM FREEZE-DRYED SULPHATE SOLUTION

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Novel mechanical, optical, electric, magnetic, thermal, chemical and biological properties are expected occur as compared to bulk behavior because of the small structure size. In the field of technical ceramics na improvement of these properties is expected when nanosized powders are used. However, owing to their high specific surfaces and their high surface-to-volume ratios, nanosized ceramic powders show a strong tendency to agglomeration. One way to obtain, soft agglomerated powders is the use of freeze-drying technique. In this work, the aggregation/agglomeration characteristics of a freeze-dried alumina powder are discussed. Aluminium sulphate solution was separated in small droplets by spraying and rapidly freezed in liquid nitrogen and the water was removed by sublimation. This technique differs from the other water volatilization methods in that particle agglomeration is prevent by a rapid freezing step. The dried and calcinated powders were characterized by scanning electronic microscopy (SEM) and mercury and nitrogen porosimetry. The results showed the freeze dried powder is compound of soft agglomerates of spherical nanoparticles. Due to the high surface-to-volume ratios of this nanosized powder, the calcination temperature was low and did not alter significantly the agglomeration state. Powder packing behavior was also studied. The result discussion was based on the SEM observations and pore size distribution of the powder before and after the compaction step.

H – P091 PHOTOLUMINESCENCE IN AMORPHOUS SrTiO3 POWDERS DOPED WITH Al, Y, Cr, Nb AND V

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The present paper presents the photoluminescence results of strontium titanate powders, obtained by the polymeric precursor method. Were studied undoped powders and powders doped with Al,Y,Cr,Nb and V. It can be affirmed that amorphous strontium titanate is sensible to doping. The Cr dopium tended to diminish the wavelength of the emission peak, whereas the opposite was observed for the V doping. The photoluminescence spectra were shown to be efficiently separated into two gaussians only. The shorter wavelength peak is located within a narrow range from 560 to 570 nm. The longer wavelength peak is also located within another narrow range, in this case from 635 to 650 nm. The doping of amorphous strontium titanate presents a secondary influence the already important photoluminescence of this material, as it presents, without doping, a quenching temperature over 300 K.

H – P092 NANOSIZED Gd2SiO5:Ce3+ SCINTILLATOR SYNTHESIZED BY DISTINCT SOL-GEL ROUTES

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Recently, an intensive research has been carried out on the spectroscopy of doped rare earth oxides nanocrystals because optical and electronic properties of these materials considerably differ from those of the bulk ones. Therefore, nanosized Ce3+-doped Gd2SiO5 has been investigated because it is an attractive scintillator to be applied in medical imaging due to its high absorption coefficient, fast decay time (20-60 ns), excellent hardness and emission in the green to near-UV regions. In this work, Ce3+-doped Gd2SiO5 was prepared by distinct sol-gel routes: the conventional one, which uses rare earth nitrates solutions and TEOS, and a modified one that employs organic polymers as fuel and dispersing medium. The X-ray powder diffraction (XRD) patterns confirmed that both routes lead to the formation of crystalline Gd2SiO5 with monoclinic system, space group P21/c. The FT-IR spectra show the bands corresponding to asymmetric stretching vibration of SiO4 groups and O-Si-O bonds bending vibration, which are in agreement with XRD data. Transmission Electron Microscopy (TEM) was performed to observe and compare the morphology and medium size of the nanoparticles prepared by both routes. Emission spectra (lex = 346 nm) show a broad emission band with maximum in the blue region (430 nm) corresponding to the 5d - 4f Ce3+ transition. Therefore, both routes can be considered adequate to prepare nanosized Ce3+-doped Gd2SiO5 scintillator.

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PHASE ASSIGNMENTS IN SnO2-ZnO SYSTEM

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Resumo: Small ZnO addition to SnO2 systems results in ceramics with high densification. The aim of this work is to study the behavior of zinc addition ranging from 0.5 to 2 mol% SnO2 obtained by means of Pechini method. In this way, addition of 1.5 mol% ZnO favors the ceramic densification. However, further additions inhibit the process reducing linear shrinkage up to 14%. This behavior is justified by the formation of a second phase of zinc stannate in quantities close to 2 mol% when analyzed by X-Ray Diffraction and with the determination of the lattice parameters in SnO2-ZnO system.

H – P094 MECHANICAL AND THERMAL PROPERTIES OF POLYPROPYLENE/BRASGEL BENTONITE NANOCOMPOSITES AS A FUNCTION OF MELT BLENDING PROCESS CONDITIONS

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In the present work the mechanical and thermal properties of Polypropylene/Brasgel bentonite systems were investigated as a function of melt blending process conditions. Two different forms to melt-mix Polypropylene (PP H103 provided by Braskem) and Brasgel bentonite clay were used: a) preparation of master batch. In this case, PP/bentonite was mixed by melt mixing PP with bentonite carried out in a chamber of mixture (Rheomix 600) coupled to a system 90 Haake-buchler torque rheometer operating at 180 ºC and 60 rpm for 10 minutes. The PP/bentonite master batch pellets were incorporated into the PP matrix by melt intercalation in a counter rotating twin screw extruder (tw-100) coupled to a system 90 Haake-buchler torque rheometer and operating at the same processing condition described above and b) PP/bentonite dry blend was preparation by melt intercalation at the same extruder and processing condition described above (180 ºC and 60 rpm). The content of clay incorporated into the polypropylene matrix by melt intercalation was 1 and 3% w/w. The extruded products were air-cooled to room temperature, ground in a rotating knife cutter and dried in an air-circulating oven operating at 110ºC for 6h before being compression molded at 210ºC, 20Kgf/cm2 for 5 min. The samples were thermally (DSC) and mechanically (tensile) characterized. The results indicate that best overall performance was achieved with the PP/Bentonite system with extruded from dry blend.

H – P095 OBTAINMENT AND CHARACTERIZATION OF CERAMIC-METALLIC COMPOSITE OF SiC/Al

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The present work aims at obtaining composites of ceramic matrix of silicon carbide infiltrated with aluminum. Test bodies of 60x13x5mm of a ceramic matrix of carbide were studied. The test bodies were sintered at 1100ºC. After sintered, the samples were characterized and infiltrated with melted aluminum. The particles size distribution of carbide used for obtaining the test bodies presented medium size of 38,02μm. Before infiltration, the ceramic material was characterized by conventional techniques for the determination of water absorption, apparent porosity, apparent specific mass, flexural rupture module at three points, and by optical microscopy. The SiC test bodies analyzed presented na average value of 23,65% of water absorption, 41,27% of apparent porosity, and specific mass of 2,97 g/cm3. The average rupture module of the sintered samples was of 7 MPa. The characteristics of the composite obtained were determined by scanning electronic microscopy (SEM), where the presence of the particles of SiC with aluminum supply in the intergranular spaces was evident, by X-ray diffraction, presenting the Al2O3, Si(metallic), SiC and Al phases, by flexural rupture module at three points being of approximately 244 MPa and for hardness with na average 138 HB. The microestruture studies showed complete infiltration of pores by the melted aluminum in the ceramic matrix and its homogeneity along the test body.

H – P096 A STUDY OF THE INFLUENCE OF ALUMINA (Al2O3) AND CAULIM CAMPO ALEGRE ON SILICON CARBIDE/ALUMINUM COMPOSITES (SiC/Al)

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Nowadays materials are projected to cater for the most varied needs. Thus, the market for composites material has become a highly competitive area where the substitution of traditional materials for engineering materials is one of the goals. The study and improvement of the properties of these materials is of fundamental importance to their applications. The main objective of this work was the study of the influence of alumina (Al2O3) and caulim loads added to a matrix of silicon carbide in the properties of the composites obtained. Bodies of test of ceramic matrix of pure silicon carbide, silicon carbide with alumina and silicon carbide with caulim were estudied. The test bodies were sintered at 1100ºC. After sintered the samples were characterized and infiltrated with casting aluminum under pressure in press which had been specially developed for that purpose. The ceramic powders used for obtaining the samples were analyzed in order to determine the particles size distribution. The ceramic materials, before the infiltration, were characterized through the conventional techniques of determination of water absorption, apparent porosity, apparent specific mass, and flexural rupture module in three points and optical microscopy. The composites characteristics were analyzed by SEM, and x-ray diffraction was used, for flexural rupture module and hardness testing. The alumina or Caulim Campo Alegre addition provided significant increases in the mechanical properties.

H – P097 EFFECT OF THE POWDER SIZE DISTRIBUTION ON THE MICROSTRUCTURE OF SIC CERAMIC DOPED WITH ALUMINA AND RARE EARTH OXIDES

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The use of liquid phase sintering is increasing for densification of silicon carbide. However, a second phase formation can affect high temperatures properties with severe reduction on ceramics performance in creep. In this work the effect of powder size distribution on the microstructure of SiC ceramic doped with alumina and rare earth oxides is discussed. Samples with 80% of SiC, 10% of Al2O3 and 10% of rare earth oxides (all in weight) they were milled in a high energy mill for different times (1, 6 and 24 hours). The powder mixture was analyzed to characterize the grain size distribution, crystalline phases (by diffraction of rays X - XRD) and form and size of particles (by scanning electronic microscopy - SEM). The powder was compacted by uniaxial and isostatic pressing and sintered at 2050ºC in argon atmosphere, in a closed graphite crucible with a BN powder bed. After sintering, crystalline phases were identified by XRD and the microstructures by SEM. The powder and ceramic characteristics were correlated and the result showed the influence of the grain size distribution of the powder on the microstructure of the sintered SiC ceramics.

H – P098 PRODUCTION AND CHARACTERIZATION OF MgO DOPED ALUMINA POWDERS

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Alpha alumina (-Al2O3) is utilized in many areas of modern industry because of its unique mechanical, electrical, and optical properties. Fine and reactive -Al2O3 powders production for sintering is one of the most important steps to obtain alumina ceramic with superior properties. The -Al2O3 powders production was restricted to be defined size, due to the crystal inherent growth resultant of the phase transformation during the processing powder. New synthesis routes of alumina powders were being studied for reach -Al2O3 without crystal growth. The objective of this work, it was to obtain and to characterize fine alumina powders doped with MgO. The doped powders were compared with non-doped powders alumina. It was used two routes of powder synthesis; one derived from “Pechini” method e another using emulsification with oleic acid. The calcinations powders were made in different temperatures for verify the minimum temperature to obtain total transformation to -Al2O3. Differential thermal (DTA) and thermogravimetric analyses (TG) of the precipitates were performed to determine the temperatures where decomposition of organics matter and phase transformations ocurr. The powders, after calcinations, were characterized by X ray diffraction, superficial specific area and scanning electron microscope. The results showed that the dopant and the method to prepare the samples affect the temperature to obtain -Al2O3 and the phase transformations.

H – P099 PHOTOLUMINESCENCE: DISORDERED PBWO4 THIN FILMS

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Stoichiometric PbWO4 thin films were synthesized using a chemical solution processing, called of polymeric precursor method. In this method, soluble precursors such as lead acetate trihydrate and tungstic acid, as starting materials, were mixed in an aqueous solution. The thin films were deposited on silicon, platinum-coated silicon substrates by means of the spinning technique. The morphology and crystal structure of the thin films were investigated using X-Ray diffraction (XRD) and atomic force microscopy (AFM). XRD characterizations of these films showed that PbWO4 phase crystallize at 500 °C from an inorganic amorphous phase. The optical properties were also studied. The luminescence spectra of PbWO4 disordered thin films at room temperature revealed an intense emission band in the visible region. The intensity of photoluminescence is strongly dependent of the done thermal treatment.