3RD BRAZIL MRS MEETING

October 10-13, 2004

SYMPOSIUM E:

PROGRESS ON DEVELOPMENT OF ELECTROCERAMIC MATERIAIS (Joint Symposium: IV Brazilian Symposium on Electroceramics)

Symposium Organizers:

Antonio Eduardo Martinelli (DEME-UFRN)

Antonio Gouveia de Souza (DQ-UFPB)

José Antonio Eiras (DF-UFSCar)

Reginaldo Muccillo (IPEN-SP)

Sidnei Antonio Pianaro (DEMA-UEPG)

SYMPOSIUM E

PROGRESS ON DEVELOPMENT OF ELECTROCERAMIC MATERIALS

Monday, October 11

Session Chair: Jose A. Eiras

08:00 OPENING REMARKS

08:30 PBN FERROELECTRIC SINGLE CRYSTAL PROPERTIES MIMICKED BY TEXTURING OF CERAMIC BODIES
D. Garcia, M. H. Lente, M. Venet, F. L. Zabotto, J. A. Eiras. UFSCar, Grupo de Cerâmicas Ferroelétricas - Departamento de Física - Universidade Federal de São Carlos, Rod. Washington Luiz Km 235, 13565-905 - São Carlos - SP, Brazil.

08:45 BA1-XSRXTIO3 THIN FILMS CRYSTALLIZED USING A LOW POWER MICROWAVE OVEN
T. Mazon, M. A. Zaghete, M. Cilense, J. A. Varela. CMDMC-IQ-UNESP, R. Prof. Francisco Degni, s/n, Araraquara-SP, Brasil.

09:00 ELECTRICAL AND MICROSTRUCTURAL CHARACTERIZATION OF ZRO2_TIO2 CERAMICS PREPARED BY A MODIFIED SOL-GEL TECHNIQUE
I. C. Cosentino, E. N. S. Muccillo, R. Muccillo. IPEN, Travessa R 400, Cidade Universitária, CP 05508-900, S. Paulo, SP-Brasil; F. M. Vichi. IQUSP.

09:15 TEXTURED LaNiO3 FILMS DEPOSITED BY POLIMERIC PERCURSOR METHOD AND ASSISTED BY A MICROWAVE OVEN
G.P. Mambrini, M.T. Escote, F.M.L. Pontes, E.R. Leite, E. Longo, J.A. Varela. CMDMC/LIEC, Rod. Washington Luís (SP-310), Km. 235, CEP. 13565-905, São Carlos, SP, Brasil.

09:30 Invited PREPARATION OF HIGH-DENSITY NANOCRYSTALLINE $BaTiO_3$ CERAMICS SINTERED FROM TAILORED NANOPOWDERS
P. Nanni. DICheP – Dept. of Chemical and Process Engineering, Univ. of Genoa, P.le Kennedy, I-16129 GENOA (Italy).

10:10 COFFEE-BREAK

12:25 LUNCH

15:55 COFFEE-BREAK

16:20 POSTER SESSION

Tuesday, October 12

Session Chair: Dra. Dulcineia Garcia (DF-UFSCar)

08:00 ZrO2 INFLUENCE ON MICROSTRUCTURE AND MICROWAVE CHARACTERISTICS OF Ba2Ti9O20 DIELETRIC RESONATORS FOR TELECOMMUNICATION APPLICATIONS
Y. Koldayeva, M. C. A. Nono, P. J. Castro. INPE, Av. dos Astronautas, 1758, CEP 12227-010, São José dos Campos - SP, Brasil.

08:15 SURFACE MODIFICATIONS OF METALLIC NIOBIUM FOR PRODUCTION OF ELECTROCHEMICAL SENSORS AND THEIR CHARACTERIZATION BY OPTICAL MICROSCOPY
G. L. J. P. Silva, H. J. I. Filho, M. L. C. P. Silva, R. B. Ribeiro, J. L. Rosa. FAENQUIL. Rodovia Itajubá Lorena km 74,5 CP 116, CEP 12600-970, Lorena – SP, Brasil.

08:30 INFLUENCE OF THE MECHANICAL AND ELECTRICAL STRESS ON THE MICROWAVE DIELECTRIC PROPERTIES IN FERROELECTRIC MATERIALS
J. de los Santos Guerra, J. A. Eiras. Universidade Federal de São Carlos - UFSCar, Departamento de Física, Grupo de Cerâmicas Ferroelétricas. Rod. Washington Luiz, km 235, CEP 13565-905, São Carlos-SP, Brazil.

08:45 TO BE ANNOUNCED

09:00 MIXED CONDUCTIVITY IN YTTRIA-STABILIZED ZIRCONIA/NICKEL OXIDE COMPOSITES
F. C. Fonseca, E. N. S. Muccillo, R. Muccillo. IPEN- CMDMC-CCTM, Instituto de Pesquisas Energéticas e Nucleares C.P. 11049, CEP 05422-970, Pinheiros, S. Paulo, Brazil; V. Espósito, E. Traversa. TOR VERGATA; D. Z. de Florio. UNESP.

09:15 THE INFLUENCE OF Cr2O3 ON THE MICROSTRUCTURE AND NON-OHMIC FEATURES OF SnO2.(Co1-x, Mnx)O-BASED VARISTOR SYSTEM
W. K. Bacelar, E. R. Leite, E. Longo. LIEC-UFSCar, rodovia Washington Luis, KM 235 _ São Carlos _ SP; P. R. Bueno. FFCLRP_USP_Ribeirão Preto; J. A. Varela. UNESP_Araraquara .

09:30 Invited NON-NERNSTIAN ELECTROCHEMICAL SENSORS: FROM THE MATERIALS SYNTHESIS TO FIELD TESTS IN ENGINE EXHAUSTS
Enrico Traversa, University of Rome Tor Vergata, Roma, Itália.

10:10 COFFEE-BREAK

10:30 Invited MAGNETIC INTERACTIONS IN MULTIPHASE SYSTEMS
F. Cebollada. EUIT de Telecomunicación, U. Politécnica de Madrid, Spain.

11:00 HIGHLY TRANSPARENT FERROELECTRIC PLZT:ND^{3+} – A NEW AND PROMISING INFRARED CERAMIC LASER
A. S. S. de Camargo, J. A. Eiras, É. R. Botero, M. H. Lente , É. R. M. Andreeta, D. Garcia. Departamento de Física, Universidade Federal de São Carlos – UFSCar, Rod.Washington Luiz Km 235, CEP 13565-905, São Carlos, SP – Brazil; L. A. O. Nunes. Instituto de Física de São Carlos, Universidade de São Paulo – USP CP 369, CEP 13560-970, São Carlos, SP – Brazil; I. A. Santos. Departamento de Física, Universidade Estadual de Maringá – UEM, Av. Colombo 5790, CEP 87020-900, Maringá,PR – Brazil.

11:15 SINTERING AND ELECTRICAL PROPERTIES OF YTTRIA-STABILIZED ZIRCONIA/NICKEL OXIDE COMPOSITES
D. Z. de Florio, J. A. Varela. UNESP, Departamento de Físico-Química, Instituto de Química, UNESP Rua Prof. Francisco Degni s/n, 14801-970, Araraquara, SP, Brazil; V. Espósito, E. Traversa. TOR VERGATA; F. C. Fonseca, E. N. S. Muccillo, R. Muccillo. IPEN.

11:30 HIGH TEMPERATURE THERMODYNAMIC AND TRANSPORT PROPERTIES OF THE SR3FE2O7-D MIXED CONDUCTOR AT 400 < T < 1000 °C
L. Mogni, F. Prado, A. Caneiro. Centro Atómico Bariloche, CNEA, 8400 S. C. de Bariloche, Argentina; J. Fouletier. Laboratoire d’Electrochimie et de Physicochimie des Matériaux et des Interfaces (L.E.P.M.I), E.N.S.E.E.G., I. N. P. Grenoble, BP 75, 38402 Saint Martin d´Heres Cedex, Francia.

11:45 EFFECT OF LSM ON THE ELECTRICAL CONDUCTIVITY OF YTTRIA-DOPED CERIA
G. S. Godoi, H. L. B. Castro, D. P. F. Souza. UFSCar – DEMa. Rod. Washington Luís Km 235 Monjolinho CP 676 São Carlos – SP, Brasil.

12:25 LUNCH

15:55 COFFEE-BREAK

16:20 POSTER SESSION

Wednesday, October 13

Session Chair: Dr. Fabio C. Fonseca IPEN

08:00 STRUCTURAL AND OPTICAL PROPERTIES OF BI1.5ZNNB1.5O7 PYROCHLORE THIN FILMS PREPARED BY CHEMICAL METHOD
S. M. Zanetti, S. A. Silva, S. A. Souza. ITA/CTA, Pça. Mal. Eduardo Gomes, 50 - Vl. das Acácias, CEP 12228-900, S. José dos Campos - SP – Brazil.

08:15 ELECTRODE REACTION IN AIR OF SR1-XLAXCO0.8FE0.2O3 (0.1 < X < 0.7) ON CE0.9GD0.1O2 AT 600 < T < 800 °C
N. Grunbaum, F. Prado, A. Caneiro. Centro Atómico Bariloche, CNEA, 8400 S. C: de Bariloche, Argentina; L. Dessemond, J. Fouletier. Laboratoire dÉlectrochimie et de Physicochimie des Matériaux et des Interfaces (L.E.P.M.I), E. N.S. E. E. G. I. N. P. Grenoble, BP 75, 38402 Saint Martin d\'Heres Cedex, Francia.

08:30 INVESTIGATION OF FERROELECTRIC DOMAIN STRUCTURES BY ATOMIC FORCE AND OPTICAL MICROSCOPIES
M. H. Lente, D. Garcia, J. A. Eiras, S. M. Gheno, P. I. Paulin Filho. UFSCar, Universidade Federal de São Carlos - Departamento de Física - Grupo de Cerâmicas Ferroelétricas - CEP 13565-670 - São Carlos - SP – Brazil; A.C. Hernandes, T. Mazon. IFSC-USP.

08:45 THE ROLE OF OXYGEN ATMOSPHERE ON THE PROPERTIES OF CaBi4Ti4O15 THIN FILMS OBTAINED BY THE SOFT CHEMICAL METHOD.
A. Z. Simões, M. A. Ramirez, M. A. Zaghete, J. A. Varela. CMDMC, Instituto de Química - UNESP/Araraquara, Rua Prof. Francisco Degni s/n, Quitandinha, CEP 14801-970, PO Box 355, Araraquara, São Paulo, Brazil; E. Longo. CMDMC – UFSCar, Rod. Washington Luís (SP-310), Km 235, CEP 13565-905, São Carlos, São Paulo, Brazil.

09:00 SYNTESIS AND CARACTERIZATION OF NANOCRYSTALLINE IN2O3 PRODUCED BY A MODIFIED SOL-GEL ROUTE
J. F. Q. Rey, T. S. Plivelic, L. Torriani. LNLS/UNICAMP, Laboratorio Nacional de Luz Síncrotron, Rua Giuseppe Máximo Scolfaro, 10000, Caixa Postal 6192, Campinas, São Paulo, Brasil; E. N. Muccillo, S. K. Tadokoro, R. A. Rocha. IPEN.

09:15 PREPARATION AND PROPERTIES OF MULTILAYER PBTIO3-(PB,CA)TIO3 FERROELECTRIC THIN FILMS
P.R. de Lucena, F. M. Pontes, Elson Longo, E. R. Leite. UFSCar, Departmento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13560-905, São Carlos, SP, Brazil; José Arana Varela. UNESP.

09:30 Invited GRAIN SIZE AND GRAIN BOUNDARY DEPENDENT PHENOMENA IN $BaTiO_3$ CERAMICS
L.Mitoseriu. DICheP – Dept. of Chemical and Process Engineering, Univ. of Genoa, P.le Kennedy, I-16129 GENOA (Italy).

10:10 COFFEE-BREAK

10:30 PHOTOLUMINESCENCE IN THIN FILMS OF BZT
L.S. Cavalcante, M.R.M.C. Santos, L.S.S. Júnior. UFPI, CCN Department of Chemistry, UFPI, Terezina, PI, Brazil; F.M. Pontes, P.R de Lucena, I. L. V. Rosa, E.C. Paris, E. R. Leite, E. Longo. UFSCar; L.G. P.Simões. UNESP-Araraquara.

10:45 OPTICAL PROPERTIES OF LIN$B_{1-X}TA_XO_3$ THIN FILMS PREPARED FROM POLYMERIC PRECURSORS
A. H. M. González, A. Z. Simões, M. A. Zaghete, J. A. Varela. CMDMC, Instituto de Química - UNESP/Araraquara, Rua Prof. Francisco Degni s/n, Quitandinha, CEP 14801-970, PO Box 355, Araraquara, São Paulo, Brazil; E. Longo. CMDMC – UFSCar, Rod. Washington Luís (SP-310), Km 235, CEP 13565-905, São Carlos, São Paulo, Brazil.

11:00 INFLUENCE OF GRAIN SIZE OF POWDERS ON THE ELECTRICAL PROPERTIES OF SNO2 VARISTORS
M. L Moreira, S. A. Pianaro, A. V. C. Andrade, S. M. Tebcherani, S. R. M. Antunes, A.C. Antunes, A. J. Zara. LIMAC-CIPP, UEPG. Av. Gal. Carlos Cavalcanti, 4748, CEP 84032-900, Campus Uvaranas, Ponta Grossa-PR, Brasil.

11:15		INFLUENCE OF THE SYNTHESIS METHODOLOGY ON THE PHASE FORMATION OF (1-X)PB(MG1/3NB2/3)O3-XPBTIO3 POWDERS J. C. Bruno, J. A. Varela. LIEC-Laboratório Interdisciplinar em Cerâmica, Rua Prof. Francisco Degni, s/n, Araraquara, CEP:14800-900, S.P, Brazil; A. A. Cavalheiro. UFSCar.
11:30		EFFECT OF SECONDARY PHASE ON THE ELECTRICAL CONDUCTIVITY OF BISMUTH GERMANATE CERAMICS Z. S. Macedo, M. E. G. Valério, DFI – UFS, Rod. Marechal Rondon s/n, Jdim Rosa Elze, São Cristovão, CEP 49100-000, SE, Brasil; A.C. Hernandes. IFSC – USP; R. A. Jackson. Keele University.
11:45	Invited	HYPERFINE INTERACTIONS IN FERROELECTRIC COMPOUNDS A.L. Gárcia. DF – UNLP ad IFLP – CONICET, La Plata, Argentina.
12:25		LUNCH
15:55		COFFEE-BREAK
16:20		POSTER SESSION

E-I001 PREPARATION OF HIGH-DENSITY NANOCRYSTALLINE $BaTiO_3$ CERAMICS SINTERED FROM TAILORED NANOPOWDERS

P.Nanni. DICheP – Dept. of Chemical and Process Engineering, Univ. of Genoa, P.le Kennedy, I-16129 GENOA (Italy).

Miniaturisation of components is the present trend in high-tech microelectronic industry. In fact, decoupling capacitors demand thinner dielectrics of high permittivity and high reliable uniform structure, tunable filters require stable non-linear dielectric films, dielectric elements in passive components, like MLCCs and embedded capacitance in printed circuit boards, need reduced layer thickness (less than 1?m) and grain size (below a few hundred of nanometres) to get high volumetric efficiency. This aim can be achieved if two requirements are fulfilled: i) high quality non-agglomerated nanopowders (in the range tenth to hundred of nanometers) with narrow particle size distribution and ii) a suitable densification technique that inhibits the grain growth. Ferrolectric ceramics with perovskite-like structure, such as $BaTiO_3$, are extensively used for the application afore mentioned. In the last few years, an increasing interest has been focused on direct $BaTiO_3$ powder precipitation in aqueous medium. However some critical points such as nuclei formation, crystallite growth, aggregation of crystallites are rather difficult to control and the preparation of nanopowders is very sensible to all synthesis parameters. To get a deeper mastery of the powder preparation the precipitation process was modelled by a discretised population balance approach for simultaneous primary and secondary nucleation and for growth processes at unsteady state. Ultra-fine $BaTiO_3$ non-agglomerated powders with a narrow particle size distribution (30-40 nm; 35 m^2/g; Na ?300ppm, Sr <100ppm; Ba/Ti = 1?0.01) were synthesised by precipitation from an aqueous solution of $TiCl_4$ and $BaCl_2$ at ?80-90°C in the presence of NaOH to keep pH at ?14. To check the efficiency of the chemical process, powders were produced, both in a small batch reactor and in a continuous Segmented Flow Tubular Reactor (SFTR). In this case, the mixture was pushed into a tube where an immiscible organic fluid (carrier) gives rise to a sequence of small drops (liquid "bubbles") of the aqueous phase. In thermostatic conditions, each droplet can be considered as a micro-reactor where the general thermodynamic conditions are identical. SFTR allows an easier control of the critical points of the reaction, strongly reducing the heterogeneity conditions of the batch reactors. Moreover control of temperature, concentration and Ba/Ti ratio in the solution offers the possibility to tailor the particle size in the range 30-1,000nm. Preparation of dense, bulk nanocrystalline ceramics was carried out by Spark Plasma Sintering at 800-1000°C for 2-5 min (relative density: ?97%; grain size: 50 to 300 nm). Crystal structure and phase transitions were studied from room temperature to 250°C by X-ray diffraction, and differential scanning calorimetry. A progressive reduction of the tetragonal distortion of the polar phase with decreasing grain size was observed whereas high tetragonality with low grain size would be desirable. The heat of transition of the tetragonal to cubic transition accordingly decreases. From these experimental trends the critical size for disappearance of ferroelectricity in $BaTiO_3$ has been evaluated to be 10-30 nm.

E-I002 GRAIN SIZE AND GRAIN BOUNDARY DEPENDENT PHENOMENA IN $BaTiO_3$ CERAMICS

L.Mitoseriu. DICheP – Dept. of Chemical and Process Engineering, Univ. of Genoa, P.le Kennedy, I-16129 GENOA (Italy).

Barium titanate $BaTiO_3$ (BT) is one of the most common ferroelectric material with extensive use as dielectric for embedded capacitance in printed circuit boards, multilayer ceramic capacitors, thermal imaging and actuators, due to its high dielectric constant, thermal stability and low losses. The study of the grain size (GS)-induced electrical properties is a very important task for improving the technical performances of the ferroelectric components and as a fundamental aspect of ferroelectricity. The effect of the GS on the properties of BT ceramics was extensively studied only for ceramics with GS above 0.5?m, due to the difficulty of obtaining high dense ceramics at this scale. Recently, very fine non-agglomerated powders have been used to produce high dense ceramics with grain sizes below 100nm by Spark Plasma Sintering. The properties induced by diminishing GS and by high density of grain boundaries (GB) are presented here. The finest obtained structure (50nm GS) shows a broad phase transition with a maximum of permittivity of 800 around 95°C at the frequency f=1kHz. The reduction of the tetragonal distortion and the shift of the Curie temperature towards lower values with reducing GS lead to the conclusion of intrinsic size effects in this system. This behaviour is explained in the frame of Landau theories with GS-dependent parameters. Extrinsic effects due to the large number of GB with low permittivity are described in terms of brick-wall model. The investigation of the Raman activity proved the existence of all the

structural phases of $BaTiO_3$ with critical temperatures slightly different from the single crystal values. A few Raman modes exhibit some peculiarities related to GS and GB effects. The local polarization state was probed *via* converse piezoelectric effect using scanning force microscopy (SFM) combined with a lock-in technique. Regions with low piezoelectric activity (low P_s) beside to islands with strong piezoresponse (high P_s) and typical hysteresis behaviour were found. The various types of piezoresponse observed in the 50nm BT dense ceramics can be explained in terms of a distribution of tetragonality and polarization over the volume of the ceramic and by considering a mixed short and long range dipolar order within the sample. The present results indicate that the possible average critical GS for the ferroelectric behaviour in dense BT nanocrystalline ceramics is below 50nm.

E – I003 NON-NERNSTIAN ELECTROCHEMICAL SENSORS: FROM THE MATERIALS SYNTHESIS TO FIELD TESTS IN ENGINE EXHAUSTS

Enrico Traversa, University of Rome Tor Vergata, Roma, Itália.

E – I004 HYPERFINE INTERACTIONS IN FERROELECTRIC COMPOUNDS

Alberto López-García. Dto. de Física (UNLP) e IFLP (CONICET), La Plata, Argentina.

E – I005 MAGNETIC INTERACTIONS IN MULTIPHASE SYSTEMS

F. Cebollada. EUIT de Telecomunicación, U. Politécnica de Madrid, Spain.

One of the main problems for the control of the extrinsic behaviour of magnetic multiphase systems lies in the role played by the interactions both in the field-assisted and the thermally-assisted magnetization and demagnetization processes. In this paper we present a comparative analysis of the switching mechanisms of two different systems, NdFeB-type alloys with grain sizes in the single domain range, and Fe-SiO_2 composites with Fe concentrations above and below the percolation threshold. For the NdFeB-type system, the experimental analysis of the coercivity adquisition mechanism suggests that the magnetization reversal is nucleated at the spin misalignments present due intergranular exchange interactions. On the other hand, the study of the magnetic viscosity and of the isothermal remanent magnetization (IRM) and direct field demagnetization (DCD) remanence curves, indicates that the dipolar interactions are responsible for the propagation of the switching produced in individual particles.

E – O001 ZrO2 INFLUENCE ON MICROSTRUCTURE AND MICROWAVE CHARACTERISTICS OF Ba2Ti9O20 DIELETRIC RESONATORS FOR TELECOMMUNICATION APPLICATIONS

Y. Koldayeva, M. C. A. Nono, P. J. Castro. INPE, Av. dos Astronautas, 1758, CEP 12227-010, São José dos Campos - SP, Brasil.

Several kinds of special ceramics for applications as dielectric resonators (DRs) in the area of microwave telecommunications have been developed. These DRs should have high value of dielectric constant, high quality factor due to dielectric losses and small coefficient of resonant frequency variation with temperature. Barium nanotitanate ceramic (Ba2Ti9O20) has fulfilled these requirements. The ZrO2 addition has yielded such devices with better microwave dielectric properties. The objectives of the present work is to optimize the powder and ceramic processing for obtaining DRs with Ba2Ti9O20 majority quantities doped with ZrO2. All the powders were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The powders were compacted using a uniaxial and an isostatic pressure to form cylindrical test bodies with a suitable relationship thickness/diameter, so that DRs were produced to be used in the 5.5-7.0 GHz frequency range. Both Ba2Ti9O20 synthesizing and sintering were performed in a single step. The fracture surfaces of the sintered ceramics were characterized by XRD and SEM. The values of the dielectric constant and the quality factor at microwave frequencies were determined. The results showed that the performed ceramics obtained by BaCO3, TiO2 and ZrO2 powder mixture present better densification degree at lower sintering temperature and grain-structure homogeneity in comparison to the data from the international literature.

E – O002 EFFECT OF SECONDARY PHASE ON THE ELECTRICAL CONDUCTIVITY OF BISMUTH GERMANATE CERAMICS

Z. S. MACEDO, M. E. G. Valério, DFI – UFS, Rod. Marechal Rondon s/n, Jdim Rosa Elze, São Cristovão, CEP 49100-000, SE, Brasil; A. C. Hernandes. IFSC – USP; R. A. Jackson. Keele University.

Bismuth germanate (Bi4Ge3O12), which has a cubic crystalline structure known as eulitine, has been demanded a great deal of interest due to its electro-optic, electro-mechanical and scintillator properties. In this work, the electrical and dielectric properties of Bi4Ge3O12 ceramics were determined and compared to that observed for single crystals, aiming the characterization of the conduction mechanisms of this material. The results were compared with computer simulations results in order to determine the defect structure related to the charge transport. Bi4Ge3O12 ceramic was produced by a solid-state route and sintered up to a relative density of 98%. X-ray diffraction (XRD) measurements pointed out to the presence of 1-2% of the secondary phase Bi12GeO20. The electric and dielectric properties were obtained from impedance measurements performed for Bi4Ge3O12 ceramic and single crystal, and also for Bi12GeO20 single crystal, in the temperature range of 200-720 °C and frequency interval from 1 Hz to 10 MHz. The computed electrical conductivities follow the Arrhenius law, with a change in the apparent activation energy at 500 °C. These energies are related to the possible migration and charge transport mechanisms and were used to model the conduction mechanisms of the crystal phases and of the ceramics.

E – O003 SURFACE MODIFICATIONS OF METALLIC NIOBIUM FOR PRODUCTION OF ELECTROCHEMICAL SENSORS AND THEIR CHARACTERIZATION BY OPTICAL MICROSCOPY

G. L. J. P. Silva, H. J. I. Filho, M. L. C. P. Silva, R. B. Ribeiro, J. L. Rosa. FAENQUIL. Rodovia Itajubá Lorena km 74,5 CP 116, CEP 12600-970, Lorena – SP, Brasil.

The refractory metals as Nb or Ta may be used as electrochemical sensors for conductance - potential mesarurements according their surface modifications will be improved. Literature has been described the use of Nb Nb2O5 as a potentiometric sensor1 or in voltammetric measure-ments as na auxiliary electrode2.Our previous works described in literature too shows the suitability of use a conductance sensor made of two fixed metallic niobium sheets2for analy-tical determinations3.This sensors were studied with no modi-fication of the metallic surfaces, other than the chemical cleaning by an HNO3/HF acid mixture.The Nb/Nb2O5 pair sensor has been studied too for acidity measurements following their potentiometric response against a calomel satured reference electrode.Thir analytical performance of this sensor was studied in analytical routines and agree with those of a co-mertial glass pH electrode. This work describe the morphological characterization of the anodized metallic Nb plates and another

surface modification to take account to a metallic carbide layer for use of this electrode as working electrode in voltammetry applications or eletrolytic processes.

E – O004 HIGH TEMPERATURE THERMODYNAMIC AND TRANSPORT PROPERTIES OF THE SR3FE2O7-D MIXED CONDUCTOR AT 400 < T < 1000 °C

L. Mogni, F. Prado, A. Caneiro. Centro Atómico Bariloche, CNEA, 8400 S. C. de Bariloche, Argentina; J. Fouletier. Laboratoire d’Electrochimie et de Physicochimie des Matériaux et des Interfaces (L.E.P.M.I), E.N.S.E.E.G., I. N. P. Grenoble, BP 75, 38402 Saint Martin d´Heres Cedex, Francia.

The mixed conductors are interesting materials in view of their potential applications in solid state electrochemical devices such as membranes for oxygen separation and electrode materials for solid oxide fuel cells. For this type of applications the knowledge of the oxygen non-stoichiometry and transport properties is essential to evaluate the performance of the mixed conductors. In this work we have undertaken a systematic study of the non-stoichiometry and transport properties at high temperatures of the perovskite related mixed conductor Sr3Fe2O7-d. The variation of the oxygen content (7-d) with T and equilibrium oxygen partial pressure (pO2) was obtained by thermogravimetry within the range: 10-5 < pO2 < 1 atm ; 400 < T <1100 °C . From the experimental values of the oxygen chemical potential (pO2), the partial molar enthalpy (hO2) and the partial molar entropy (sO2) were determined for the composition range 6.00 < 7-d < 6.55. A simple defect model based on the mass law action was used to reproduce the termodynamic data close to 6.00. This model takes the stoichiometric compound Sr3Fe2O6 as a reference state. The so-determined DhO2 values show good agreement with the experimental ones. In addition, electrical conductivity of Sr3Fe2O7-d was measured at high temperature as a function of pO2 at constant temperature within the interval 500 < T < 1000ºC. The variation of the conductivity data as a function of log (pO2) is in agreement with the proposed defect model.

E - O005 ELECTRODE REACTION IN AIR OF SR1-XLAXCO0.8FE0.2O3 (0.1 < X < 0.7) ON CE0.9GD0.1O2 AT 600 < T < 800 °C

N. Grunbaum, F. Prado, A. Caneiro. Centro Atómico Bariloche, CNEA, 8400 S. C: de Bariloche, Argentina; L. Dessemond, J. Fouletier. Laboratoire dÉlectrochimie et de Physicochimie des Matériaux et des Interfaces (L.E.P.M.I), E. N.S. E. E. G. I. N. P. Grenoble, BP 75, 38402 Saint Martin d\'Heres Cedex, Francia.

The electrode reaction of perovskite phases Sr1-xLaxCo0.8Fe0.2O3 (0.1 < x < 0.6) on Ce0.9Gd0.1O2 ceramic pellets was investigated in air (pO2 = 0.21 atm) by impedance spectroscopy in the temperature range 600 < T < 800 °C. The perovskite phases were prepared by an acetic acid based gel route. The crystal structure of the samples was analyzed by X-ray diffraction. Thick porous electrodes (20 microns) were deposited on Ce0.9Gd0.1O2 electrolytes by spray and calcinated at 1000 °C during 1 h. The ac impedance spectra were obtained using the symmetrical configuration. From the analysis of the complex impedance diagrams, the variation of the electrode polarization resistance (Rp) and the activation energy of the electrode reaction with the La content were determined. While Rp increases with increasing the La content and reaches a maximum at x = 0.4, the activation energy related to the electrode reaction increases as the La content increases. This behavior is discussed considering the evolution with the La content of other factors such as oxygen non-stoichiometry, electronic and ionic conductivity in air of the Sr1-xLaxCo0.8Fe0.2O3 perovskite phases.

E - O006 EFFECT OF LSM ON THE ELECTRICAL CONDUCTIVITY OF YTTRIA-DOPED CERIA

G. S. Godoi, H. L. B. Castro, D. P. F. Souza. UFSCar – DEMa. Rod. Washington Luís Km 235 Monjolinho CP 676 São Carlos – SP, Brasil.

Interface behavior is an important aspect concerning Solid Oxide Fuel Cells (SOFCs). One common problem observed in these devices is the insulating interphases formation in the electrolyte-cathode interface contributing for the efficiency loss. However, in a recent work performed by the group it was observed that LSM - Yttria Doped Ceria composites have shown an increase in its electrical conductivity values after long time annealing. These composites were prepared in order to increase the points of contacts between LSM and Yttria Doped Ceria making easier to observe any effect in the electrical conductivity values after annealing. In this work samples containing low quantities of LSM in yttria-doped ceria were prepared in order to evaluate the effect of LSM on the electrical conductivity of Yttria-Doped Ceria. Through this procedure it will be possible to confirm if the LSM presence is

responsible for the increase in the electrical conductivity observed in the LSM – Yttria Doped Ceria composites prepared previously. Samples were characterized by XRD, SEM and electrical conductivity measurements were carried out using four probe dc technique and impedance spectroscopy.

E - O007 INFLUENCE OF GRAIN SIZE OF POWDERS ON THE ELECTRICAL PROPERTIES OF SNO2 VARISTORS

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Varistors are polycrystalline ceramics of high density with no-ohmic properties that depend on typical grain boundary phenomena. The electrical properties are highly dependent on the resultant microstructure that is dependent on chemical composition, initial characteristic of the powder, sintering conditions and grain size of the powder. By means of seed grain method, calcined powders were prepared at 950°C for 3 and 10 h in order to optimize the electrical properties. In this way, the powders obtained presented grain size over 100μm. These powders were added in concentrations ranging from 10 to 30 weigh percent to the precursor composition with grain size under 6 μm, conformed and calcined at 1300°C for 3 h with heating rate of 10°C/min. The results demonstrate reduction in the breakdown voltage up to 650V for the sample with 10 weight percent of seed grains and lower reductions for other samples. The α values underwent strong reduction resulting in data between 8 and 11 and densities of 94.1 to 89.5%, respectively. The crystalline phases were characterized by XRD and quantified by Rietveld method. In this way was found that cassiterite is the primary phase with 97% and cobalt stannate is the secondary phase with 3%.

E - O008 INFLUENCE OF THE MECHANICAL AND ELECTRICAL STRESS ON THE MICROWAVE DIELECTRIC PROPERTIES IN FERROELECTRIC MATERIALS

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Electronic devices are always shrinking in space and time. For wireless communication applications, for example, new radar systems, broadcasting and television via satellites and multi-channel means of communication at microwave wavelength are at the present in great demand and are going to increase in the near future. In this work the microwave dielectric response of the lanthanum modified lead titanate (Pb1-xLaxTiO3) ferroelectric ceramics was investigated in the frequency range of 50MHz-2GHz at room temperature. The dielectric dispersion was obtained on unpoled and poled samples in the parallel and perpendicular direction to the dielectric measurement direction. The influence of the mechanical and electrical stress on the microwave dielectric properties was studied in the whole frequency range. The results revealed two dielectric anomalies, originated from apparently different but equal physical phenomena, which were related to an over-damped resonance mechanism.

E - O009 SYNTESIS AND CARACTERIZATION OF NANOCRYSTALLINE IN2O3 PRODUCED BY A MODIFIED SOL-GEL ROUTE

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In2O3 has attracted considerable attention over the last few years due to its high electrical conductivity and good optical transparency. It has been widely used in optoelectronic devices such as solar cells, liquid crystal displays and gas sensors. In particular, this oxide has shown remarkable potential applications in the upcoming nanoelectronic building blocks and nanosensors. Recently many reports have focused on semiconductor type CO sensors, and In2O3 was identified as a base semi-conducting oxide. Among the factors affecting the properties of In2O3 gas sensors, the microstructure of the sensitivity layer is one of the most important. It has been recognized that when the size of the In2O3 particles is very small, the sensitivity and selectivity to CO is significantly improved. On the other hand, nanosized In2O3 powder is difficult to obtain and often has a high degree of agglomeration. In this work In2O3 was synthesized by a modified sol-gel route. The gel obtained was studied by thermal analysis and Fourier transformed infrared spectroscopy to investigate the thermal decomposition and constitution of the gel. Calcined materials (from 440 to 900 0C for 30 min) were studied by small angle x ray scattering and X-ray powder diffraction to study the evolution of the particle size distribution and

the degree of agglomeration with the thermal treatment.

E - O010 INFLUENCE OF THE SYNTHESIS METHODOLOGY ON THE PHASE FORMATION OF (1-X)PB(MG1/3NB2/3)O3-XPBTIO3 POWDERS

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Two different approaches for synthesizing (1-x)Pb(Mg1/3Nb2/3)O3-xPbTiO3 (x= 0.10 0.28, and 0.35) powders were demonstrated: the polymeric precursor method (PPM) and the modified columbite method (MCM). The PPM consists on the mixture of the metal citrates and esterification with a polyalcohol, while the MCM consists on the Ti-modified columbite precursor (MNT) using the polymeric precursor method, followed by the solid state reaction with PbO to form PMN-PT powders. It is difficult to synthesize PMN-PT powders using MPP directly due to the instability of lead citrate. Using MCM this problem is avoided, resulting in less content of secondary phases. The characterization of the columbite-modified precursor and PMN-PT powders were studied by X-ray diffraction and the data were also used for the structural refinement by the Rietveld method. High content of titanium tends to precipitate as MNT phase in the columbite precursor, but this event does not affect significantly the perovskite phase amount in PMN-PT powders. Previous titanium insertion in the columbite precursor provided by MCM leads to the reduction of time and temperature in the reaction between PbO and MNT to synthesize PMN-PT powders, and a large amount of perovskite phase could be reached at 700ºC for 1h. When using the PPM, a higher temperature to obtain the PMN-PT powders was required (800ºC for 2h) despite the powders were more reactive, the perovskite phase was not so stable.

E - O011 THE INFLUENCE OF Cr2O3 ON THE MICROSTRUCTURE AND NON-OHMIC FEATURES OF SnO2.(Co1-x, Mnx)O-BASED VARISTOR SYSTEM

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An investigation was made to discover how the addition of Cr2O3 affects the microstructural heterogeneity and nonohmic features of the SnO2.(Cox,Mn1-x)O-based varistor system, with x varying from 0 to 1. The presence of Cr2O3 strongly increases the nonohmic features when x = 1. However, the nonohmic features of the system decrease when x drops from 1 to 0, a behavior explained by the increase of the junction heterogeneity within the system's microstructure, accompanied by an excess of precipitates at the triple point in the grain boundary region due to modified MnO sintering. The presence of these precipitates causes the leakage current to increase in response to the creation of a non-effective barrier. The effect of heat treating these systems in oxygen- and nitrogen-rich atmospheres suggests that, according to mechanisms previously discussed in the literature, Cr2O3 is more susceptible to oxygen, so that increasing the amount of oxygen in the grain boundary region may improve the system's nonohmic properties.

E - O012 PREPARATION AND PROPERTIES OF MULTILAYER PBTIO3-(PB,CA)TIO3 FERROELECTRIC THIN FILMS

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PCT thin films are promising candidates for application in a variety of electric devices and displays desired properties for piezoelectric response. In this work, PbTiO3-(Pb,Ca)TiO3 multilayer thin films with perovskite structures were deposited onto Pt/Si/SiO2/Si(100) substrates by spin coating and heated in air at 700ºC. These films were synthesized by a soft-chem method and characterized by XRD and AFM. The thin films are tetragonally structured and present a low surface rougness and a low grain diameter. Top electrodes were deposited in vacuum through a mask to form a MFM capacitor configuration for electrical measurements. Capacitance dependence on the voltage is strongly non linear, which confirms the ferroelectric properties of the multilayer films resulting from ferroelectric domains switching. The ferroelectrics properties of the films were confirmed by P-E hysteresis measurements.

E - O013 MIXED CONDUCTIVITY IN YTTRIA-STABILIZED ZIRCONIA/NICKEL OXIDE COMPOSITES

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The yttria-stabilized zirconia/nickel oxide composite (YSZ/NiO) is the precursor of the YSZ/Ni cermet, which is used as SOFC anode material. Thus, in order to fabricate high performance anodes it is necessary to careful control both the microstructural and the electrical properties of the precursor material. In the present study, YSZ/NiO composites were prepared by a modified liquid mixture technique in the concentration range of 0-75 mol% of NiO. This method was found to result in powders with dispersed nanometric NiO particles, as inferred from scanning electron microscopy analysis. Sintering at 1350 °C for 1 h resulted in samples with high relative densities. The phase analysis and electrical characterizations were performed by X-ray diffraction and impedance spectroscopy (IS) measurements, respectively. The IS experiments were performed in the 100-800 °C temperature range with controlled oxygen partial pressure. The main results show that the composite samples are comprised of a homogenous distribution of the two oxides, and no solubilization of the NiO into the YSZ structure was detected. The IS data indicate three NiO content ranges where ionic, mixed, and electronic conduction are predominant. In addition, the temperature dependence of the electrical conductivity shows two Arrhenius-type transport processes with different activation energies in samples with mixed conductivity. This process was associated with a p-type semiconductivity contribution.

E - O014 SINTERING AND ELECTRICAL PROPERTIES OF YTTRIA-STABILIZED ZIRCONIA/NICKEL OXIDE COMPOSITES

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The study of the yttria-stabilized zirconia/nickel oxide composites (YSZ/NiO) is motivated by both the crescent interest in mixed ionic and electronic conductors (MIEC) and the use of the YSZ/Ni cermet as the solid oxide fuel cell (SOFC) anode. Therefore, controlling the microstructure of the YSZ/NiO is an important issue for the study of the electrical transport mechanisms of the composite MIEC and also a key step for the fabrication of high-performance SOFC anodes. In this study the composites were prepared by a modified liquid mixture technique in the concentration range 0-75 mol% of NiO, followed by calcination at 450 °C. The powders were investigated by field emission scanning electron microscopy and X-ray diffraction analysis (XRD). Pellets sintered at 1350 °C for different times (ts) were studied by XRD and impedance spectroscopy (IS). The IS experiments were performed in the 5 Hz-13 MHz frequency range and 100-800 °C temperature range. The IS data show that there is a strong dependence of the electrical conductivity on the sintering time. The 28 mol% of NiO composite sintered for ts = 4 h exhibits an activated behavior of the electrical conductivity. For decreasing ts, two slopes in the Arrhenius plots and higher values of the electrical conductivity are observed. These results suggest that the relative grain size and possible reactions of the oxides can be controlled by sintering parameters in order to optimize the electrical properties of the YSZ/NiO composite.

E - O015 STRUCTURAL AND OPTICAL PROPERTIES OF BI1.5ZNNB1.5O7 PYROCHLORE THIN FILMS PREPARED BY CHEMICAL METHOD

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The Bi1.5ZnNb1.5O7 pyrochlore thin films were prepared by the polymeric precursor method. The films were deposited by dip coating onto FTO/glass, Si(100) and Pt/Ti/SiO2/Si(100) substrates. These films were annealed at temperatures ranging from 400 to 550 oC for FTO substrates and from 500 to 800 oC for Si(100) and Pt/Ti/SiO2/Si(100) substrates. Atomic force microscopy images revealed the surface morphology of films as function of the annealing temperature. X-ray diffraction detected the cubic pyrochlore in films treated above 450 oC. Films were fully crystallized at 700 oC. The films deposited onto Pt/Ti/SiO2/Si(100) and Si(100) showed a high 222 orientation. Optical band gap calculate from the transmission measurements is 4.0 eV.

E - O016 TEXTURED LaNiO3 FILMS DEPOSITED BY POLIMERIC PERCURSOR METHOD AND ASSISTED BY A MICROWAVE OVEN

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Lanthanum nickelate is an interesting material for applications as electrodes in DRAM memories that utilizes peroviskite type dielectrics, because of its metallic behavior and improvements on fatigue properties of these devices. The microwave frequency source of energy is being developed as a new way to process materials. The microwave processing method has several advantages over conventional processes, among then rapid and uniform heating, lower sintering temperatures and cost savings in terms of energy and time. In this work, LaNiO3 thin films were deposited by the polymeric precursor method on SrTiO3(100) substrate. A pre-heating treatment was made in a conventional furnace to eliminate organic materials and after that the films were crystallized in a microwave oven at 700C for ten minutes. These films were characterized by X ray diffraction, AFM and electrical transport measurements. The XRD analysis showed a single-phase film with (100) preferred orientation. The AFM images revealed a smooth and crack free surface, with roughness of 4.6nm. A near metallic behavior was observed in the temperature dependence of the electrical resistivity between 20 and 300K, and its value at room temperature is 500(micro-ohms).cm.

E - O017 ELECTRICAL AND MICROSTRUCTURAL CHARACTERIZATION OF ZRO2_TIO2 CERAMICS PREPARED BY A MODIFIED SOL-GEL TECHNIQUE

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ZrO2-TiO2 nanometric ceramic powders were prepared by a modified sol-gel method. The raw materials were zirconium isopropoxide and titanium oxichloride. The calcination was performed at 450 oC/1h resulting in nanosized ZrTiO4 orthorhombic powders. After pressing and firing at 300 oC/3h and 400 oC/3h, a small amount of monoclinic ZrO2 was observed by X-ray diffraction. The open porosities determined by mercury porosimetry are close to 45% for all samples. The electrical characterization was carried out at room temperature by electrochemical impedance spectroscopy under 60% and 84% relative humidity. The electrical conductivity of zirconia-titania ceramics increases as a function of the relative humidity due to the water adsorption on the porous surface. The behavior of the conductivity with humidity is found to be advantageous for application as humidity sensors.

E - O018 INVESTIGATION OF FERROELECTRIC DOMAIN STRUCTURES BY ATOMIC FORCE AND OPTICAL MICROSCOPIES

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Ferroelectric materials have offered a tantalizing potential for technological applications since their discovery more than 80 years ago. However, the miniaturization of electrical devices due to the nanotechnology advances as well as the development of domain engineering devoted to creation of domain structures with desirable parameters have demanded in situ characterization of ferroelectric domain structures. Such investigation is fundamental to understand the macroscopic behavior of ferroelectric materials for practical applications. In this context, ferroelectric domain image assisted by contact mode atomic force microscopy (AFM) and optical microscopy (OM) have extensively been used to investigate ferroelectric domain patterns even at nanometer scale. The objective of this work is to investigate the ferroelectric domain structures of Ba1-xCaxTiO3 (BCT) fibers produced by laser heated pedestal growth technique by using atomic force and optical microscopies. The results revealed that the domain patterns are strongly influenced by Nd-doping and mechanical stress introduced by polishing process. It was seen that domains for as-grown non-doped BCT fibers are bended between pinning sites, which are released after annealing at high temperatures or by Nd-doping. In addition, the ferroelectric domain patterns of BCT are compared to (1-x)Pb(Mg1/3Nb2/3)O3-xPbTiO3 single crystals.

E - O019 BA1-XSRXTIO3 THIN FILMS CRYSTALLIZED USING A LOW POWER MICROWAVE OVEN

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Ba1-xSrxTiO3 thin films were produced by the polymeric precursor method using an aqueous solution. The crystallization of the films was carried out using a domestic microwave oven by means of a SiC susceptor in order to absorb the microwave energy and rapidly transfer the heat to the film. Low microwave power and short time have been used. The films obtained are well-adhered, homogeneous and with good specularity, even when treated at 600 degreesC for 10 min. The microstructure and the

structure of the films can be tuned by adjusting the crystallization conditions. The microstructure presented a polycrystalline nature with spheroid small mean grain size.

E - O020 PBN FERROELECTRIC SINGLE CRYSTAL PROPERTIES MIMICKED BY TEXTURING OF CERAMIC BODIES

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Electroceramics with anisometric grains as plates or needles can be textured under certain processing conditions. Ferroelectric systems with tungsten bronze structure can show columnar shape grains with the length axis along [001] direction in the case of the tetragonal symmetry. In this work, textured lead barium niobate (PBN) tungsten-bronze structure type ceramics were obtained using hot forging as texturing and densification processing. The studied composition (Pb/Ba= 56/44) was close to the morphotropic phase boundary, but at the tetragonal side of the PBN phase diagram. The dielectric and ferroelectric properties of the undoped and lanthanum doped ceramics were characterized as a function of the parallel and perpendicular cuts related to the forging axis. Pre-forming was made by cold uniaxial and isostat pressing of PBN powders. The hot-forging, performed at different temperatures and dye assemblies, resulted in fully dense ceramics. SEM analysis showed preferential growth of PBN grains along the plane perpendicular to the forging axis. The lanthanum doped sample showed high spontaneous polarization (>20mC/cm2) for the perpendicular cut without hysteresis behaviour. Strong dielectric anisotropy was found between the different ceramic cuts confirming the potentiality of the processing for obtaining ceramics that mimic single crystal properties.

E – O021 THE ROLE OF OXYGEN ATMOSPHERE ON THE PROPERTIES OF CaBi4Ti4O15 THIN FILMS OBTAINED BY THE SOFT CHEMICAL METHOD.

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Thin films of calcium bismuth titanate (CaBi4Ti4O15) were deposited on the Pt/Ti/SiO2 (111) substrates by spin coating from the polymeric precursor method (Pechini process). Annealing in static air and oxygen atmosphere was performed at 700°C for 2 hours. The films obtained were characterized by X-ray diffraction, atomic force microscopy and electric properties. The dielectric properties of CaBi4Ti4O15 films were found to be remarkably sensitive to the annealing treatment atmosphere. This study demonstrates that annealing in oxygen atmosphere increases the dielectric relaxation phenomenon. Such dependence of the dielectric relaxation was related both to oxygen vacancies and to the presence of negatively charged oxygen, trapped at the grain boundary and/or at the electrode/dielectric film interface.

E – O022 OPTICAL PROPERTIES OF $LINB_{1-X}TA_XO_3$ THIN FILMS PREPARED FROM POLYMERIC PRECURSORS

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Lithium niobate-tantalate ($LiNb_{1-x}Ta_xO_3$) thin films were deposited on sapphire (0001) substrates by dip coating process using polymeric organic solution. The coated substrates were thermally treated at 600°C for 3 h under oxygen atmosphere. In order to study the influence of tantalum composition (x) on the crystallinity, morphology and optical properties, films with x = 0, 0.5 and 1 were deposited. X-ray diffraction results showed that preferentially (006)-oriented $LiNb_{1-x}Ta_xO_3$ films were obtained. Atomic force microscopy (AFM) analyses revealed that the surfaces are not only crack-free but also appear relatively dense. The multilayer $LiNb_{1-x}Ta_xO_3$ films displayed spherical grain structures with a superficial roughness of approximately 1-3 nm. Modified envelope method was used for obtaining optical properties and the thickness of the films by using the transmission spectra. The packing density for the $LiTaO_3$ film was 0,9337, indicating that this film presented a best morphologic quality.

E – O023 HIGHLY TRANSPARENT FERROELECTRIC PLZT:ND^{3+} – A NEW AND PROMISING INFRARED CERAMIC LASER

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Lead lanthanum zirconate titanate ferroelectric transparent ceramic (PLZT) presents very interesting properties for electro-optical applications. Although some spectroscopic studies, in the near infrared spectral region, have also been done in rare-earth ions doped samples, a perspective that has not been explored up to now, is to use these materials as laser active media, to the example of what has been done for YAG:Nd^{3+} ceramics, and others. Ceramic lasers have attracted considerable interest in the past four years, due to their numerous advantages over crystal lasers. Among these advantages are the faster and lower cost production, the large variety of shapes and sizes with which ceramics can be obtained, high laser output powers, low probability of non-radiative losses, etc. In this work, we propose the use of PLZT:Nd^{3+} as a promising material for generation of laser emission at 1.06 ?m. Samples were obtained via solid state reaction of oxide precursors, followed by uniaxially hot pressing, and investigated from the structural, microstructural and spectroscopic points of view. Results are discussed in terms of the ceramic properties, and of the employment of optical spectroscopic as a useful tool, that allowed optimization of obtainment processes, leading to a great improvement in the material optical quality. Proof of its potentiality is given by the observation of stimulated emission at 1.06 ?m, with fairly high cross sections. Furthermore, by combining electrical and optical properties, PLZT:Nd^{3+} might become an interesting integrated device such as a self-switching laser active medium.

E – O024 PHOTOLUMINESCENCE IN THIN FILMS OF BZT

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In perovskite-type crystals, a broad luminescence band is usually observed at low temperatures and is associated to the presence of imperfections or defects. The luminescence discovery in the amorphous thin films of BZT is an interesting property for technological applications in optical devices. In this work, it was synthesized, characterized and analyzed the photoluminescent properties of the thin films of Ba(ZrxTi1-x)O3(BZT) where x=0,50. This material was submitted to heat treatment in oxygen atmosphere by different calcination times. It is interesting to synthesize thin films of Ba(Zr0,50Ti0,50)O3 since they are obtained by low production cost trough the polymeric precursors method and are a new ceramic materials free from heavy metals. The thin films of Ba(Zr0,50Ti0,50)O3 were obtained by the polymeric precursors method and calcinated to 473 K for 2 hours to losing the organic matter. After that, the films were carried to heat treated to 573 K in oxygen atmosphere for different calcination times of 8, 16, 24, 48, 96 and 192 hours. The AFM analysis revealed the growth of the BZT grains in the films, with the increase of the calcination time in the oxygen atmosphere. The thickness of the films was measured trough thin film cross-section analysis by SEM. Intense photoluminescence(PL) at room temperature was observed for all the amorphous thin films of BZT. The films calcinated at 573 K for 192 h presented a cubic crystalline structure and didn´t present luminescence.

E – P001 SPECIAL ERBIUM DOPED FIBERS FOR OPTICAL AMPLIFIER WITH VERY LARGE BANDWIDTH OPTICAL AMPLIFIER

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The tellurite glass thermophysical properties define if optical fibers are viable. The luminescence lifetimes define the amplifier efficiency. Environement around the rare ions, the optical amplification bandwith and energy transfer process will be dicussed in this work.

E – P002 THE INFLUENCE OF THE SrO2 ON THE Ba2Ti9O20 CERAMIC MICROSTRUCTURE FOR DIELECTRIC RESONATOR APPLICATIONS

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This work deals with the influence of strontium oxide (SrO2) on the dielectric properties of the barium nanotitanate (Ba2Ti9O20) ceramics as dielectric resonator for telecommunications applications. The main requirements of these dielectric resonators are the high selectivity and frequency stability, that are accomplished through a high value of both the dielectric constant (E) and of the quality factor (Q), as well as a low temperature coefficient of resonance frequency (tf). The ceramics were prepared using a suitable powder mixtures, without and with SrO2 addition, varying the content from 0.1 to 1.0 mol %. The powders were mixed, compacted by uniaxial and isostatic pressing. Finally, they were sintered at 1300ºC for 3 hours. The ceramics characterization was carried out using X-ray diffraction technique, and scanning electron microscopy – SEM. The microwave parameters (E, Q and tf) were measured using a suitable microwave system. SEM analyses showed a high densification degree with the presence of some pores. The increased Q due both to dielectric losses and E is directly proportional with the amount of SrO2 added to the ceramics, as expected. The thermal coefficient presented good result, thus providing a good frequency stability for dielectric resonators.

E - P003 CALCIUM BORATE GLASSES AND GLASS CERAMICS FOR MEDICAL AND ENVIRONMENT DOSIMETERS

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Borates glasses have been widely studied mainly due to its characteristic as glass-former material. Moreover, these glasses present an absorption coefficient, which is equivalent to that of the tissue. This fact makes some borate glasses ideal materials to develop medical and environment dosimeters. These compounds usually have higher sensitivities when compared to LiF:Mg,Ti (TLD-100) materials. However, its hygroscopicity represents a serious drawback on the performance of the thermoluminescence (TL) property. Among the tetraborates, calcium tetraborate is one of the most attractive system because it presents a high chemical durability. In the present investigation, we have studied the thermoluminescence characteristic of the xCaB4O7-(100-x) CaB2O4 system with x ranging from 0 to 100. Glassy samples have been obtained from melting/molding in air atmosphere. According to the XRD data, glass ceramic samples obtained after the heating process present only the CaB2O4 crystalline phase. The glass transition temperatures were found to be 636 ºC for x = 20 and 646 ºC for x = 80. During the TGA measurements no loss mass was detected. For the thermoluminescence measurements (TL), the samples were irradiated with a UV light (500 W Hg lamp) during 30 min. The temperatures of TL emission peaks were at 142 ºC and 200 ºC, respectively. The best TL results were found for the x=20 and x=80 wt% samples. The dielectric property of the samples has been determined and will be presented herein.

E - P004 HIGHLY ANOMALOUS LOW TEMPERATURE MAGNETORESISTANCE IN La2/3Sr1/3MnO3

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The electrical resistance (R) and the magnetoresistance (MR) of a manganite sample prepared by a Pechini method were investigated in this work. The La2/3Sr1/3MnO3 manganite was obtained by first calcinating the dried powder at 973 K. Next, it was compacted at 3 tons and sintered at 1373 K. Both the powder and the sintered sample were characterized by X-ray diffraction and by ac magnetic susceptibility. The ferromagnetic to paramagnetic phase transition temperature TC (=343 K) obtained for the sintered sample was 5 K higher then the TC of the calcinated powder and 20 K smaller than the values obtained for samples calcinated at higher temperatures. R was measured from room temperature down to 1.5 K and for applied magnetic fields H up 7 T. An anomalous deep minimum was observed near 30 K. The value of the R measured at the lowest temperature was found to be as high as the one obtained at the metal-insulator transition (TP = 185 K). The sample did also show colossal MR whose value decreases monotonically from 55% at 1.5 K to 23% at room temperature. We also found two magnetic field regimes in MR data. Nearly half of the magnetoresistance occurs for H below 0.1 T and half of it above 0.1 T. The highly anomalous minimum, the large low-field magnetoresistance and the anomalous high MR observed at low T are signatures that the large number of defects and grain boundaries are indeed determining the transport and magnetic properties in this material.

E - P005 ELECTRICAL AGING PARAMETERS OF THE HIGH VOLTAGE PORCELAIN INSULATORS

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The understanding of the processes that dominate the aging and the consequent flaw of the porcelain used in the high voltage insulators in power systems is subject of technological importance for the industry of electric equipments and for the power companies. In this work, new and aged insulator ceramics were compared, aiming to correlate the changes in composition and microstructure with the aging process. Field aged samples as well as new porcelain pieces were provided by the local energy company (Energipe). X-ray powder diffraction was performed to verify the crystalline phases in the material, whereas optical and atomic force microscopy were used to investigate topography changes due to the aging process. The electrical behaviour of the samples was followed by impedance spectroscopy measurements in the temperature range of 200-720 °C and frequency interval from 100 Hz to 1 MHz. X-ray diffraction patterns presented an amorphous band which seems to enhance with aging, but no significant changes were observed in the topography of the samples up to the moment. The complex impedance diagram presented two semicircular arcs, with a clear change in the apparent activation energy. Variable frequency Partial Discharge (PD) analysis and radio frequency emission PD analysis are being performed as a function of the artificial aging to investigate the processes that trigger the dielectric breakdown of the porcelain insulator.

E - P006 CHARACTERIZATION OF WATER PERMEABILITY OF ZRO2 - TIO2 CERAMIC ELEMENTS BY PHOTOACOUSTIC TECHNIQUE FOR APPLICATION IN HUMIDITY SENSORS

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The objective of this work is the characterization of ZrO2 - TiO2 ceramic elements for the application as air humidity sensor. The characterization is made through measures of permeability of the sensor element to the water vapour, using the photoacoustic technique. The photoacoustic effect is obtained through the incidence of a beam light modulated on the sample in study, coupled to a photoacoustic cell. This device consists of a small closed volume to which a microphone is coupled. The incident light is absorbed by the sample generating pulses of heat that cause a periodic changing of temperature in the adjacent layer of gas the sample with the same modulation frequency that the incident light. The temperature changing causes the expansion and compression of that layer of air, creating pressures waves inside of the camera, that they are detected by the microphone. In this work, several porous ceramics with different porosity was analysed. Each sample was fixed in the camera photoacoustic, with the external side gone back to an atmosphere where the humidity was controlled. Measures in function of several parameters as time, transient among one humidity and other, allow to obtain the diffusion time and permeability of water vapour in the sample. Recipients partially filled out with

saturated saline solution, simulating atmospheres with relatives humidities of 11, 23, 33, 43, 54, 75, 84 and 97% were used for the control of the relative humidity inside of the cell.

E - P007 EFFECTS OF PR6O11 ON THE GRAIN SIZE AND ELECTRICAL PROPERTIES OF SNO2 BASED VARISTORS

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The effects of Pr6O11 on the SnO2Co2O3Ta2O5 varistor system sintered at 1350oC for 120 min were investigated. It was found that respectively Co2O3 and Pr6O11 significantly affect the grain size and electrical properties of the SnO2 – based varistor. The grain size rises from 3,0 to 7,0 microns, the breakdown electrical field increase from 800 to 1485 V/cm with an increasing in Pr6O11 concentration from de 0,05 mol% to 0,5 mol% . The analysis of experimental results shows that the sample with 0,1 mol% Pr6O11 presents the best nonlinear electrical property and the highest nonlinear coefficient (alpha=11) among all samples. The nonlinearity decreases as a function of Pr6O11 concentration. This behavior is due to the microstuctures defects, which are responsible for potential barrier (Schottky).

E - P008 MORPHOLOGIC PROPERTIES OF SNO2 FILMS PREPARED BY MODIFIED SOL-GEL PROCESS

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The tin dioxide (SnO2) is used in several technological applications, such as sensors, catalysts, and electro-optical equipments. Many of these applications involve the use of SnO2 in the form of ceramic thin film. The preparation of SnO2 films has been widely studied and different methods have been proposed. In this work, pure or doped SnO2 films produced by the sol-gel method, using tin tartrate as a precursor are presented. The films were deposited by “dip coating”. The sol stability was verified using UV-visible spectroscopy. The obtained films were characterized by X-ray diffraction (XRD), optical microscopy, scanning electron microscopy (SEM) and UV-visible spectroscopy. The obtained films presented a thickness below 1 mm. The X-ray low angle analysis showed the small characteristic peaks for SnO2. Showed a low cristalinity of this film, probably, it was due to the low calcination temperature. In the Nb-doped film, the scanning electron microscopy analysis showed the presence of small grains with uniform size. Experimental conditions can be readily controlled to ensure reproducibility.

E - P009 STUDY OF INCLUSIONS MORPHOLOGY IN THE COMPOSITES OF P(VDF-TRFE) COPOLYMER AND PZT CERAMICS.

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The PZT ceramics have a high piezoelectric coefficient but are brittle. The P(VD-TrFE) copolymer is flexible and easy to fabricate. By integrating the superior properties of ceramic and copolymer, one can develop a composite with high processability, good piezoelectric constants, mechanical flexibility and high compliance. The connectivity between ceramics particles and polymer matrix is of utmost importance. This important variable depends on the distribution of ceramic particles in the matrix. The ceramic particle size distribution and the morphology of inclusions are reported in this work. Also, a study of microstructure as function of sample manufacturing methods is described. PZT powders were obtained by organic precursors method and characterized by SEM. The composite samples were manufactured by three methods: solvent cast (SC); dip coating method (DC); fast solvent extraction combined with hot pressing method (HP). For both methods the ceramic agglomerates are formed and the particle morphology is different from that found for the raw materials. Regular size particle distribution was found for the samples obtained by HP method. The densities of HP samples for all ceramic content were close than theoretical density. The SC and SP methods produce heterogeneity microstructures with the presence of rich regions in ceramic. The microstructural results reveals that the

HP method is a simple and alternative method for manufacturing of 0-3 or 1-3 connectivity composites.

E - P010 INFLUENCE OF PREPARATION METHODS ON FERROELECTRIC BEHAVIOR OF P(VDF-TrFE)/BaTiO3 COMPOSITES.

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Electroactive composites are technological interesting due to flexibility, high mechanical compliance, and good piezoelectric properties. The selection of adequate raw materials is not sufficient condition to assure the good piezoelectrical performance of composite. Connectivity of the individual phases is of highest important role, because this controls the electrical flux pattern in the sample. Connectivity depends on the sample preparation method. In this work the influence of P(VDF-TrFE)/BT composite preparation on the ferroelectric hysteresis is reported. The samples were prepared using two methods: (a) solvent cast method; (b) solvent extraction by water method. For the a-method the films were deposited on to a glass substrate. After crystallization at 100oC for 12 hours the samples were characterized by SEM. Ferroelectric hysteresis measurements are reported as function of ceramic volume. The SEM analysis showed that the a-method produce 0-3 composites while for the b-method was observed the 3-1 connectivity. The increase of ceramic content increases the remanant polarization for both methods; however the largest values of polarization were obtained for the samples prepared by b-method. The composite P(VDF-TrFE)/BT 45/55vol% prepared by b-method presented the best value of remanent polarization (5,8micronC/cm2).

E - P011 CAPACITANCE ANALYSIS OF PD-DOPED SNO2 THICK FILMS SENSORS EXPOSED TO CO ATMOSPHERES

M. A. Ponce, J. Fenoglio, M. S. Castro, C. M. Aldao, INTEMA, J. B. Justo 4302, Mar del Plata, Argentina, B7608FDQ.

It was found that resistance and capacitance of SnO2 thick-films are modified by CO adsorption at the grain surface. In particular, the capacitance response to a CO atmosphere was studied. In the analysis, the presence of Schottky potential barriers at the grain boundaries was considered to be responsible for the obseved results. An increasing of the capacitance with time can be related to the reaction of CO with the previous oxygen adsorbed at the grains surface. Also, we found that other mechanisms affect the sensor response.

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E - P012 SYNTHESIS AND CHARACTERIZATION OF LANTHANUM BETA ALUMINA

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LaB-Al2O3 solid electrolyte powders were synthesized by solid state reaction of oxides and by the polymeric precursor method. The aim of this study was to obtain the lanthanum B-alumina phase, which could be used as solid electrolytes in oxygen sensors for determination of ultra-low oxygen contents in molten steel at high temperatures (>1773 K). The ceramic powders have been characterized by scanning electron microscopy and X-ray diffraction for structural analysis, and the sintered pellets by impedance spectroscopy for electrical response analysis. The X-ray diffraction results show that the LaAl11O18 phase has been synthesized. The dependence of the electrical conductivity of the LaB-Al2O3 sintered pellets upon temperature and the use of magnesium oxide as sintering aid for obtaining dense electrolytes were studied.

E - P013 MULTIFERROIC PROPERTIES OF BIFEO3 COMPOUNDS PRODUCED BY POLYMERIC PRECURSOR ROUTE

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The perovskite BiFeO3 presents both a spontaneous magnetization polarization. We have produced polymeric precursor route. The powder were heat-treated in the temperature range of 350 to 800°C in air. The structure, magnetic and electric properties were investigated by X-ray diffraction, magnetization measurements. The XRD pattern revealed that the BiFeO3 samples crystallize in a rhombohedrally distorted perovskite structure, space group R3c. The evaluated unit cell parameters are a » 53,58 and c » 6,93 Å, which are close to those described in literature. Although, small peaks at 2q » 27 and 33° were attributed to an additional phase Bi25FeO40. The ferromagnetoeletric properties of BiFeO3 compounds, show the influence of magnetic and electric field on the polarization and magnetization properties, respectively.

E - P014 MICROSTRUCTURAL DEVELOPMENT OF TRIVALENT OXIDES AND ZNO-DOPED TIN TITANATE ZIRCONATE

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This work consisted of the preparation of pure and trivalent oxides, besides ZnO-doped tin titanate zirconate by oxide mixture. Samples were sintered at temperatures ranging from 1200 to 1450oC. Analyses were made of the influence of sintering on the microstructural development and dielectric properties of these materials at high frequencies. The liquid phase composition present during sintering was shown to be dependent on the ZnO percentage. This percentage determines grain composition and, hence, dielectric performance at high frequencies. A maximum unloaded quality factor, Ql, was found for 1.0%wt ZnO-doped ZTS sintered at 1400oC.

E - P015 SELECTION OF LIQUID PHASE FORMING ADDITIVES AT SINTERIZATION OF POTASSIUM STRONTIUM NIOBATE CERAMICS

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Development of KSr2Nb5O15 ceramic of type tetragonal bronze tungsten (TTB) has attracted attention because of low or free-fatigue and lead-free features. The main drawback of these ceramics is a high processing-sintering temperature between 1450 and 1550 oC. The objective of this study was to analyze several additives former of liquid phase that might result in the decreasing of the sintering temperature of KSr2Nb5O15. Then, some potential sintering additives were studied, as CuO, WO3, V2O5 and H3BO3. Physical and chemical transformations of these oxides and the stability of KSr2Nb5O15 in the presence of additives were investigated by differential thermal analysis (DTA up to 1300 oC) and differential scanning calorimetry (DSC up to 600 oC). All materials were studied in air, with a heating rate of 10 oC.min-1. CuO showed oxidation-reduction phenomenon at 1049 oC and melting point at 1153 oC. WO3 showed similar oxidation-reduction phenomenon at 1200 oC, structural transformations in the range from 220 oC to 1104 oC and sublimation at 1275 oC. V2O5 showed

melting point at 684 oC and an oxidation-reduction phenomenon at 800 oC, as well as the vaporization at 1100 oC. H3BO3 exhibits gradual lost water changing to metaboric acid (HBO2) at low temperatures between 100 and 236 oC and melting at 170 oC. The results are discussed from the view point of sintering mechanisms in according to each additive, such former flux or eutectic liquids.

E - P016 ANALYSIS OF THE DENSIFICATION PROCESS OF THE KSR2NB5O15 CERAMIC PREPARED BY CHEMICAL ROUTE

S. Lanfredi, D. C. Vieira, M. A. L. Nobre. FCT/UNESP, Laboratório de Cerâmicas Funcionais, Rua Roberto Simonsen, 305, C.P. 467, CEP: 19060-900, Presidente Prudente - SP, Brazil.

KSr2Nb5O15 ceramic with tetragonal tungsten bronze type structure (TTB) presents a potential of application as actuators, transducers and capacitors (wireless comunication). Technological research on the next generation of wireless telecommunications devices has revealed a lack of materials with permittivity parameters and dielectric loss appropriate. In this work the sintering of the KSr2Nb5O15 system via constant heating rate was investigated. KSr2Nb5O15 nanostructured powders were obtained by chemical synthesis, based on the polymeric precursor method, using as starting reagents potassium oxalate, strontium nitrate, niobium ammonium oxalate NH4H2[NbO(C2O4)3]3H2O, citric acid and ethylene glycol. The precursor powder obtained was calcined at 1150 oC during 10 hours, in O2 atmosphere. KSr2Nb5O15 powder was characterized by X-ray diffraction. The sintering of the KSr2Nb5O15 was investigated from room temperature to 1300 oC. Both relative linear shrinkage (L-Lo)/Lo and the derivate curve of relative linear shrinkage d((L-Lo)/Lo)/dT as a function of temperature, as well as the derivate curve of relative linear shrinkage as a function of relative linear shrinkage were used to analyze the densification process. The densification phenomenon and the linear expansion coefficient of the sintered ceramic were discussed.

E - P017 PERFORMANCE CHARACTERIZATION OF AMORPHOUS NIO FILMS AS ACTIVE MATERIALS FOR RECHARGEABLE LITHIUM BATTERIES

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It is well known that lithium (Li) batteries are one of the most promising devices for compact and light power sources. This fact improved the interest in new materials for secondary Li cells. In this work we studied the performance of amorphous NiO films as active layer for secondary Li cells. The NiO films forming a: GLASS/ITO(200 nm)/NiO(150 nm) structure. The characterization was made by cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and low-angle X-Ray Diffraction (XDR). The electrochemical measurements were performed in a sealed electrochemical cell with a N2 atmosphere. The used solvent/electrolyte system was a 0.1 mol.L-1 LiAsF6/DMF solution, the counter-electrode was graphite and the potentials were referenced against a Ag/Ag+ quasi-reference electrode. The NiO voltammograms show two redox processes that demonstrated an high reversibility, maintaining unchanged after 100 cycles. This processes presented Epa and Epc values of .0.37/0.12 V and 0.74/0.56 V and the ip varies linearly with the scan rate. This indicates that the process of lithium insertion is not diffusion dependent. The EIS results corroborate with the CV results showing a no diffusional behavior possibly caused by the short thickness of the NiO films. The XRD spectra obtained before CV showed the amorphous character of the films, however after CV the XRD spectra showed that the film develop some crystallinity, indicating that the Li insertion occurs as NiO + 2Li+ -- Ni + LiO2.

E - P018 PHYSICAL CHARACTERIZATION STUDY AND MICROSCTRUCTURAL OF THE CERAMIC SYSTEMS CABI4TI4O15 AND SRBI4TI4O15

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Calcium bismuth titanate (CaBi4Ti4O15) and Strontium bismuth titanate (SrBi4Ti4O15) crystal powders were obtained by the polimerics precursors method, calcinated at 750 ºC for 3 hours. The powders were characterized by X-ray diffraction (XRD) and infrared spectroscopy (IR). The superficial area measurements were made by the (B.E.T.) method. The decomposition of the organic material and verification of the initial crystallization temperature were followed by (TG/DSC) curves. The resulting

powders were compacted in a bulk forms and sintered for the density verification (Arquimedes method) and micro structural analysis. The micro structural analysis was made by scanning electronic microscopy (SEM). The results obtained by (XRD) showed a crystal phase for both ceramics, with a formation of the a little quantity for the Bi2O3 secondary phase. The (IR) analysis showed the same behavior as (XRD) results except for the carbonates existence and with a metal-oxygen (M-O) bond well defined. Through the (TG/DSC) curves were able to follow the temperature range of the organic material decomposition and the beginning of the crystallization process. By the (SEM) results was able to analyze the bulk porosity, boundary and average grain size of the sintered ceramics.

E - P019 PRODUCTION OF TEXTURED SAMPLES OF YBA2CU3O7-D BY INFILTRATION AND GROWTH TECHNIQUE THROUGH SOL-GEL METHOD.

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This work is about the production of superconducting textured samples of YBa2Cu3O7-d, Y123, with uniform distribution of micro inclusions of Y2BaCuO5, Y211, using the infiltration and growth technique, IG. The powders were obtained using the chemical route Sol-gel. This method produces an extremely homogeneous, quite fine ceramic powder, free from agglomerates, reactive in temperatures 30% lower than the powders obtained by the traditional ceramic routes. This process involves the production of a Y211 sample, that is sinterized and placed in contact with a Y123 sample. This sample will act as a continuous source of liquid phase. This group, Y123/Y211, it is heated above the peritetic temperature of the Y123. The liquid of the compacted source will infiltrate in the porous sample of Y211 and the peritetic reaction it will happen during a slow cooling through the peritetic temperature, resulting in the growth of the Y123 grains and an uniform distribution of small particles of Y211. Measures of levitation force and determination of the critical current, Jc, through the method of finite elements, MEF will be done. With those measurement, we hoped to obtain results that show a significant improvement of the transport properties in comparison to superconductors samples of Y123 produced by conventional methods.

E - P020 SYNTHESIS, SINTERING AND IMPEDANCE SPECTROSCOPY OF Bi12TiO20 SILLENITE CERAMICS

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Bi12TiO20 (BTO) is a photorefractive material with potential application in holography, image processing and recording media. A number of publications deal with electrical properties of BTO single crystals, but up to the moment there is not much information about successful production and characterization of BTO dense ceramics. In this work, we report the preparation details, sintering and the electrical characterization of BTO ceramics. The single-phase powder was obtained by solid-state reaction of the precursor Bi2O3 and TiO2, which were milled and calcined at 700°C for 8 hours. The ceramic powder was characterized by differential thermal analysis (DTA) and X-ray diffraction (XRD) to confirm the presence of a unique phase. After the calcination, the powder was conformed by uniaxial pressing and sintered up to a relative density of 95 %, determined by Archimedes method. The electric and dielectric properties were obtained from impedance data in the temperature range of 200-720 °C and frequency interval from 1 Hz to 10 MHz. The dense ceramics presented AC properties very similar to that reported in literature for the single crystal. The electrical conductivity follows the Arrhenius law with activation energy of (1.03±0.03) eV. Real permittivity was 30 for high frequencies. As a single semicircle was observed in the Nyquist diagrams, we are performing impedance measurements in more porous ceramics to enhance the contribution of the grain boundaries.

E - P021 PREPARATION OF TUNGSTEN-DOPED SRBI2NB2O9 THIN FILMS BY SOFT CHEMICAL ROUTE

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Varela. LIEC/UNESP-Ar.

Tungsten-doped SrBi2Nb2O9 (W-SBN) solutions (1 and 5 mol%) were prepared by the polymeric precursor method and were used for deposition of thin films by spin-coating on Pt/Ti/SiO2/Si substrate. The films were treated in a domestic microwave oven, using a SiC susceptor placed under the substrate. The susceptor absorbs the microwave energy and transfers the heat to the film. It was verified a decrease in the crystallization temperature of tungsten-doped SrBi2Nb2O9 films from 600 ºC to 700 ºC in microwave processing, when compared to undoped films treated in conventional oven. Moreover, the crystallization was reached in a reduced time - 10 min. The films presented the perovskite SrBi2Nb2O9 phase as verified by X-ray diffraction using Cu Kα radiation. 1 mol% W-doped film is polycrystalline, conversely the 5 mol% W-doped one revealed a preferential orientation in 00c direction. Rounded and homogeneous grains were observed by atomic force microscopy for all samples. The concentration of dopant associated with the microwave treatment process influence the orientation of the samples studied. Furthermore, the microwave process reduces the time of thermal treatment.

E - P022 FERROELECTRIC TI-DOPED SRBI2NB2O9 THIN FILMS OBTAINED USING A MICROWAVE OVEN

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Ferroelectric SrBi2Nb2O9 (SBN) thin films doped with titanium were prepared by the polymeric precursor method. The films were deposited by spin coating onto Pt/Ti/SiO2/Si substrate and crystallized using a domestic microwave oven. The doped-SBN solutions were prepared by adding 10 mol% of Ti to the SBN precursor solution. The films were treated in a microwave oven for 10 min at 600, 650 and 700 ºC with a 230 ºC/min heat rate and the SiC susceptor placed below the substrate. Structural and microstructural characterizations were performed by X-ray diffraction (XRD) and atomic force microscopy, respectively. A fluorite phase was observed for the SBN film treated at 600 ºC. However, when it was treated at 650 and 700 ºC, the perovskite SBN phase was verified. A preferential orientation in the 00c direction was observed as the temperature increased, verified by XRD and electron backscattered diffraction. The C-V measurements for film treated at 600 ºC showed a typical butterfly-like curve, characteristic of a ferroelectric material. The remanent polarization and coercive field at 60 Hz were 2.8 μC/cm² and 63 KV/cm, respectively. Films treated at 650 ºC presented dielectric constant and coercive field of 42 and 0.05, respectively, at 100 KHz. The study of the electric characteristics for the films treated at 700 ºC was not possible, probably due to the orientation and the degradation of the interfaces substrate / film or electrode / film.

E - P023 ELECTRICAL BEHAVIOR OF ZIRCONIA-YTTRIA/ZIRCONIA-MAGNESIA COMPOSITE MATERIALS UNDER OXYGEN AT HIGH TEMPERATURES

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(ZrO2: 3 mol% Y2O3)0.5 (ZrO2: 8 mol% MgO)0.5 composite ceramic materials have been prepared by solid state synthesis, pressing and sintering at 1500 oC. Phase identification has been done by X-ray diffraction and the electrical properties have been studied by impedance spectroscopy in the 5 Hz – 13 MHz frequency range at 460 oC. An experimental setup was used inside a tubular furnace for measurement the electrical signal (emf) generated to monitoring oxygen activity in gases at temperatures in the 500oC - 1200oC range. The responses of the tubular composite pellets to oxygen and argon were measured as a function of time to evaluate the response time of the sensor. The main results show that this composite is partially stabilized, the emf stabilizes at approximately 1000 oC, and the electrical signal at that temperature depends on the amount of oxygen.

E - P024 PREPARATION AND CHARACTERIZATION OF SNO2.NB2O5.FE2O3 AND SENSOR PROPERTIES

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The chemical synthesis of inorganic materials has become an important study in materials science. Numerous researches show that material properties can change drastically as a function of changing reaction parameters. In the present work we have investigated the preparation of ceramic SnO2.Nb2O5.Fe2O3 powders for the development of liquefied petroleum gas sensor. These powders were prepared by Pechini\'s method, morphologically characterized by X-ray diffraction, the specific surface area was determined by BET and scanning electronic microscopy and submitted to tests of sensitivity for liquefied petroleum gas. A correlation was established taking into account the microstructure of the material, the effects of the dopant and the response of the sensor. The main results show that the precise control of synthesis and processing technique allows obtaining homogeneous ceramic powders with reproducible and optimized properties.

E - P025 STRUCTURAL AND MAGNETIC PROPERTIERS OF LA0,5SR0,5COO3-γ THIN FILMS DEPOSITED BY SPIN COATING

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In conductive oxides, La0.5Sr0.5CoO3 (LSCO) films are widely used because of their isotropic, low electrical resistivity (about 100μ.cm at room temperature) and good chemical stability. LSCO belongs to the pseudocubic perovskite La1−xSrxCoO3(0<x≤0.5) family. The amount of distortion and the electrical resistivity decrease with the increase of Sr contents. LSCO has cubic perovskite structure with lattice constant matched to many ferroelectric materials. In ferroelectric devices, the use of LSCO electrodes results in superior fatigue and retention characteristics compared with the use of conventional Pt electrodes. Therefore, it is often adopted as thin film electrodes. In this work, the thin films of LaxSr1-xCoO3-γ (LSCO), where x=0.50, was synthesized by the polymeric precursor method (PPM). The films were deposited in not-conduction substrate for obtain material as electrode. The thin films were characterized for Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction were used to investigate the formation of the LSCO phase. XRD revealed that the film showed good crystallinity and were crystallized the 700 ºC, treated in oxygen atmosphere. The analysis of AFM data showed a well-developed dense grain structure, after of deposition of layers six of material was measured by a film cross-section analysis made by SEM. The electrode of LSCO obtained by PPM, presented electric resistivity a high little in relation with the electrode of LSCO prepared for other methods.

E - P026 DIELECTRIC RESPONSE OF BaTiO3-FILLED EPOXY RESIN PROCESSED BY DIPPING

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The dielectric behaviour of composite materials (epoxy resin – barium titanate) was analysed as a function of ceramic content. Epoxy and Nb2O5-doped BaTiO3 were chosen because of their good dielectric properties. The ceramic was milled in isopropilic medium using a planetary mill with ZrO2 balls for 90 min to obtain better dispersion during the mixing. Particles size distributions were determined and lattice parameters were characterised by X-Ray Diffraction, while the resin was characterised by scanning differential calorimetry (DSC). Epoxy was diluted using tetrahydrofuran (THF, 60% wt) as solvent in order to reduce matrix viscosity and to facilitate the mixing step. Once the ceramic particles were mixed with the resin, dipping technique was used to obtain the composite films onto glass plates on which gold electrodes were deposited by sputtering. After that, the system was cured at 100ºC during 2 hours. Dielectric measurements were performed from 25 Hz to 1 MHz and 25ºC to 120ºC. It was found that the final materials had high permittivities and a strong dependence with the filler concentration. The peaks (a) were shifted to higher frequencies because relaxation processes were influenced by interfacial polarisation effect, known as Maxwell-Wagner-Sillar. Electrical relaxation a generated by resin dominions was detected in the composites and it was influenced by temperature (principally near the resin glass transition temperature, Tg).

E - P027 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.

E - P028 ELASTIC CHARACTERIZATION OF FERRO-PARAELECTRIC PHASE TRANSITION IN SPT (SR0.75PB0.25)TIO3 CERAMICS

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Phase transitions may be characterized through optical, dielectric or elastic measurements. However, there are no many communication concerning elastic characterization ferro-paraelectric phase transition. Lead strontium titanate (SPT) ferroelectrics ceramics have been the focus of studies because show a ferro-paraelectric phase transition around room temperature, suggesting a high potencial for application as room temperature tunable capacitor. In this work, the ferro-paraelectric phase transition of the SPT 75/25 ceramics has been investigated by the ultrasonic pulse echo technique and dielectric measurements. Ultrasonic attenuation and velocities longitudinal or transversal were simultaneously measured. The temperature dependence of the elastic modulus (Shear Modulus, Bulk Modulus, Young Modulus and Poisson Ratio), calculated from the ultrasonic velocities, was determined around the phase transition temperature (TC»228 K). The results obtained from dielectric and elastic measurement are compared.

E – P029 PHOTOLUMINESCENCE OF MN DOPED BARIUM TITANATE POWDERS

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The nature of visible photoluminescence in BaTiO3:Mn powders prepared by the Precursores Polimeric Method, in the light of the results of experimental and theoretical studies. From the experimental side, BT-Mn powder samples have been synthesized following a soft chemical processing, their crystal structure has been confirmed by X-Ray data and Retvield data and the corresponding photoluminescence (PL) properties have been measured. Only the structurally disordered samples present PL at room temperature. From the theoretical side, first principles quantum mechanical techniques, based on density functional theory at B3LYP level, have been employed to study the electronic structure of a crystalline (BT-Mn-c) and an asymmetric (BT-Mn-a) models These results are observed in the band struture, DOS and electronic density . Our investigation of the electronic structure involved the use CRYSTAL98 to simulate the variation of the electronic structure in the BaTiO3:Mn crystalline and assimetric phases. Correlations between results experimentals (DRX and PL) and theoreticals (calculation periodic) can bring to light clarification about this structure.

E - P030 INFLUENCE OF POROUS MICROSTRUCTURE ON HUMIDITY SENSING PROPERTIES OF ZrO2-TiO2 CERAMICS

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In the present time of automation, sensors form an integral part of any control system. Since water is highly a polar molecule and it influences almost all phenomena in the atmosphere, therefore, it is necessary to obtain cheap and a reliable humidity sensor. Porous ceramic materials, which are useful as humidity sensor, exhibits change in their electrical properties on exposure to humid atmosphere. The conductivity changes with the ambient humidity since the variation in the amount of physisorbed water on grain surface or condensed water in capillaries changes the surface conductivity. Although, ceramic materials, in particular metal oxides, have advantage in terms of thermal stability, mechanical strength and resistance to chemical attack, they rely on the surface related properties which are closely dependent on its microstructure. Therefore, it is necessary a understanding of the correlation between the microstructure and humidity sensing properties of the porous ceramics. In the present study, ZrO2-TiO2 ceramic sensor, which has shown good humidity sensing characteristic, are selected for in a investigation of microstructural correlation with humidity sensing. The sensitivity as well as response and recovery time are found strongly dependant on porous structure and volume. The results showed that the ZrO2-TiO2 ceramic has great potential to be used as relative humidity sensor.

E – P031 PHASE TRANSITION OF THE NA0.80K0.20NBO3 CERAMIC: AN ANALYSIS BY DILATOMETRY VIA CONSTANT HEATING RATE

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Akaline niobate type (Na,K)NbO3 system have been studied due its piezoelectric and dielectric properties. Although antiferroelectric character of the NaNbO3 the addition of small quantity of K+ cations in its structure result in the development of ferroelectric properties at room temperature. In this work was investigated the linear coefficient dilatation of the Na0.80K0.20NbO3 prepared by chemical synthesis via evaporation solution method. After evaporation of solutions a precursor salt was obtained. This precursor was calcined at 700 oC by 5 h obtaining a crystalline and single phase powder of Na0.80K0.20NbO3. Dense ceramics (98 % of relative density) of Na0.80K0.20NbO3 were investigated by dilatometry via constant heating rate from 25 up to 1200 oC. The linear shrinkage curve as a function of temperature showed the presence of two anomalies between 100 oC and 700 oC. These phenomena occur in the same temperatures where are identified anomalies in the dielectric behavior of the Na0.80K0.20NbO3 at high temperature. The first anomaly positioned at 200 oC is associated to the orthorhombic-tetragonal phase transition and the second one, positioned at around 400 oC is attributed to the tetragonal-cubic phase transition, which is associated with its Curie temperature. From dilatometry via constant heating rate was possible to derive the linear expansion coefficient and to investigate the phase transitions of Na0.80K0.20NbO3 correlating with its dielectrical behavior.

E - P032 THE TiO2 INFLUENCE ON THE MICROSTRUCTURE AND ELECTRIC PROPERTIES OF VARISTOR SYSTEM SnO2.CoO.Nb2O5.Cr2O3

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The objective of this work is to investigate the TiO2 influence, in different concentrations, on the microstructure and electric properties of varistor system SnO2.CoO.Nb2O5.Cr2O3 obtained from conventional ceramic processing and sintered at 1300ºC/1h. The microstructure characterization of the sintered samples was carried out through a scanning electronic microscopy. The ceramic phases were determined through X-ray diffraction, which showed a single-phase system, regardless of the amount of TiO2 added. In electric characterization, the measurements I x V were carried out by stabilized voltage source and two digital multimeters. The non-linearity coefficient (α) value was obtained through a linear regression of points in logarithmic scale from 1mA/cm2. From measurements I x V at different temperatures, the values of the potential barrier height fb and the constant b, which is related to the potential barrier width, were determined. The potential barrier width (b) remained practically constant; however, the height (fb) presented a small reduction in some samples. The non-linearity coefficients, in terms of temperature, had a similar behavior up to 100ºC. For higher temperatures, the sample with 2.0% of TiO2 had a better result for greater temperature variations. Due to its resistance to higher temperatures, this varistor emerges as the best option for the protection of electrical equipment among the TiO2 added samples.

E - P033 CHARGES DENSITY ALTERATIONS IN HUMAN HAIR FIBERS

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Nowadays, technological development in products of hair care has been researched. However, some subjects still were not answered completely. Electrostatic phenomena in insulators have still possesses several phenomena to be answered for instance: which is the distribution of electric potentials across an organic polymer or ionic non-conducting material, and how does it contribute to mechanical, optical, adhesion and electrically insulating properties of the solid. Electrostatic force microscope (EFM) is the new purpose to evaluation of charges density on hair surfaces submitted or not to poly dimetil dialil cloruro amonico (polyquaternium 6) treatments. It was evaluated hair fibers submitted to different treatments: virgin hair, virgin hair washed with polyquaternium 1%, and bleached hair without treatment and washed with polyquaternium 1%. The virgin hair presented a homogeneous charge distribution on hair surface. The virgin hair treated with polyquaternium shown that domains with excess electric charge on surface decreased with the polymer application, remaining charges just in cuticles edges. Already the bleached hair treated with the polyquaternium present a decrease of the charges on hair surface. These results indicate that the polyquaternium promoted the neutralization of hair surface charges. It can be concluded that EFM technique allowed the verification of hair surface charges alterations that caused by the polyquaternium interaction with the hair fiber surface.

E - P034 LINBO3 THIN FILMS ON SI (100) CRYSTALLIZED IN A MICROWAVE OVEN

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The effects of thermal treatment using microwave and conventional ovens on LiNbO3 thin films deposited on Si (100) substrate were investigated. The deposition solution was prepared by the polymeric precursor method. The obtained films were crystallized from 400 ºC to 700 ºC for 2 h with a 5 ºC/min heat rate in a conventional oven, and for 10 min with a 230 ºC/min heat rate in a microwave oven by hybrid heating with a SiC susceptor. The scanning electronic microscopy of these films showed a good adherence to the substrates. X-ray diffraction and atomic force microscopy (AFM) images revealed that the microwave-treated films are polycrystalline at temperature as low as 400 ºC. Conversely, films were not crystallized when treated at this same temperature in the conventional oven. AFM images revealed a homogeneous crack-free surface of spherical grains for all films. Furthermore, it was observed an increase in the grain size with the microwave process. The use of microwave energy makes possible to obtain thin films at low crystallization temperatures and reduced costs in terms of time and energy.

E - P035 INFLUENCE OF NB IONS ON THE PROPERTIES OF BIT BASED CERAMICS

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Pure and niobium bismuth titanate ceramics (BIT) present attractive ferroelectric and dielectric properties promising for memory applications. In this work, the ceramics were prepared by the polymeric precursor method in the composition range Bi4Ti1-xNbxTiO12 with x equal to 0; 0,1; 0,2 and 0.4. X-ray analyses of the diverse ceramic compositions indicated the presence of a single perovskite phase. It was also verified that the addition of niobium leaded to a nonlinear particle size distribution. The grains present a plate-like morphology which is typical for this system. They electric condutivity was measured as a function of the temperature; in general, it was verified that the increase in niobium concentration diminished the condutivity of those ceramics.

E - P036 RAMAN SCATTERING AND X-RAY DIFFRACTION STUDIES OF POLYCRYSTALLINE CACU3TI4O12 UNDER HIGH-PRESSURE

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The complex perovskite CaCu3Ti4O12 (CCTO) has been recently reported as a material having the largest dielectric constant (16000-18000) ever measured in the laboratory. In addition to this striking property, the dielectric constant is nearly constant over a wide temperature range (from 100 to 600 K). The effects of pressure on vibrational and structural poperties of CCTO have not yet been reported. The knowledge of the pressure dependence of the Raman spectra of polycrystalline CCTO can be the basis for probing and interpreting built-in stress in CCTO films. We studied CCTO under high pressures. Both Raman and x-ray results indicated no evidence for pressure-induced phase transition in the 0-46 GPa pressure range. The frequencies of all Raman modes exhibit a linear dependence on pressure. We also determined the pressure coefficients dw/dP for all modes. This set of parameters was used for evaluating the built-in stress in CCTO thin films prepared by pulsed laser deposition. The pressure-volume data at room temperature has been well described by the Birch\'s equation of state. The experimental value of the zero pressure bulk modulus was determined as Bo = 212 GPa. The dw/dP values along with the Bulk modulus were used for calculating the Gruneisen parameters for CCTO. This work improved our knowledge of both vibrational and structural properties of CCTO that will be useful for feeding back theoretical models and allowed to better understanding the Raman spectra of thin films.

E - P037 LaNiO3 THIN FILMS DEPOSITED BY THE POLIMERIC PRECURSOR METHOD ON DIFFERENT SUBSTRATES

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This work reports a systematic study on the synthesis and characterization of LaNiO3 thin films. This material is a ceramic conductor and presents a near metallic behavior, and is interesting for capacitor electrodes application in memories systems, because of the reduction of fatigue behavior. They were produced by the polymeric precursor method and were deposited on five different singlecrystall substrates: Si(100), Al2O3(0001), MgO(100), LaAlO3(100) and SrTiO3(100). These films were crystallized at 700ºC for two hours in a conventional furnace. The effects of substrate on the physical properties of the samples were studied by X ray diffraction, SEM, AFM and electrical resistivity measurements. Such characterization revealed that all samples are single-phase, and some of them are epitaxial. In the case of the films LaNiO3/SrTiO3 and LaNiO3/LaAlO3, it was observed only the Bragg reflections belong to the (100) plane. The morphological images revealed smooth and crack free surfaces. The temperature dependence of the electrical resistivity showed that all films present a conductor behavior. Although, the lower electric resistivity were observed for the LaNiO3/SrTiO3 sample. Thus, a simple methodology was used resulting in films with good characteristics and promising for the suggested application.

E - P038 EFFECTS OF ADDITION OF LI/SC AND SC/MO DOPANT PAIRS ON THE PROPERTIES OF THE RELAXOR PMN

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PMN (Pb3MgNb2O9) is good relaxor ferroelectric and have a great volumetric efficiency (Km >20.000) and low firing temperature (~1000°C). Columbite associated to the co-precipitation methods allows the obtaining of PMN powders with good homogeneity. The effect of 1 and 2-mol% of Li/Sc and Sc/Mo dopant pairs on the microstructure and phase formation of columbite MN and perovskite PMN have been investigated. The use of the Rietveld method permitted an adequate structure refinement of the found phases allows investigating the changes in crystal parameters and phase amounts. Phase formation superficial area was determined for the samples, showing conclusive results under additives and methodological variations.

E - P039 LUMINESCENCE PROPERTIES OF EU-DOPED LITHIUM TANTALATE POWDERS

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Recently, materials with photoluminescence (PL), electroluminescence (EL), non linear optics properties have been attracted much attention due to several applications in optical-electronics devices. Lithium tantalate (LiTaO3) is currently one of the most widely used electrooptic materials because of its characteristic ferroelectric, piezoelectric and pyroelectric properties, besides of its large non linear optical (NLO) coefficients and photorefractive damage resistance. Pure and 0.1 to 1 mol% europium doped LiTaO3 powders were prepared by polymeric precursor method. The powders were characterized by X- ray diffraction (XRD) aiming to accompany the phase formation; infrared spectroscopy to verify the presence of residual organics compounds; and luminescence spectroscopy. The powders calcined at 650 oC for 3 h showed LiTaO3 crystalline phase without secondary phases. The presence of residual organic compound was not observed in infrared spectrum in these temperature and time conditions of calcination. It was observed in the excitation spectra a higher absorption, around 270 nm, which was attributed to Eu-O charge transfer (TC). In the emission spectra, it was possible to verify that the 5D0 ®7F2 transition is more intensive than 5D0 ®7F1 transition. This suggests that the Eu+3 ions occupy in the crystal lattice a site with no inversion symmetry.

E - P040 PHYSICAL AND ELECTRICAL PROPERTIES OF NTC THERMISTORS BASED ON MANGANITE CERAMICS

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In this work, results on physical and electrical properties of Negative Temperature Coefficient thermistors based on manganite ceramics are reported. Several compositions with partial substitutions by Zr, Co, Cu and Fe in nickel manganite were prepared by solid state reactions as well as by the polymeric precursor techniques. Thermal analysis results evidenced the main reactions occurring up to 1300 ºC. Phase characterization was performed by X-ray diffraction experiments. Resistance measurements were carried out to verify the thermistor behavior and to determine the specific temperature coefficient in the studied compositions.

E - P041 CHARACTERIZATION OF THE BA2IN2O5 PHASE OBTAINED BY THERMAL CRYSTALLIZATION

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The Ba2In2O5 compound was synthesized by thermal crystallization of a precursor solution. The thermal decomposition of the precursor solution and that of individual cation solutions was studied by thermogravimetry and differential thermogravimetry. The composition and structure of precursor materials and calcined powders were investigated by Fourier transform infrared spectroscopy. X-ray diffraction experiments were carried out for phase analysis and lattice parameters determination. It was found that barium nitrate is a suitable starting material for the synthesis of this compound. The use of this barium precursor allowed for obtaining a single brownmillerite phase of Ba2In2O5 with a reduction of the sintering time or temperature.

E - P042 SYNTHESIS AND PROPERTIES OF NANOSIZED INDIUM OXIDE

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Indium oxide is an n-type semiconductor widely used in optoelectronic devices and in the gas sensor field. Over the last few years, great attention has been given to the synthesis of ultrafine In2O3 powders, because its sensing properties were found to be greatly improved with reduction of the particle size. In this work, nanosized powders of indium oxide were synthesized by a homogeneous precipitation method using two precursor materials. Average crystallite size values of powders calcined at 400 ºC

lower than 20 nm, and exhibiting a bixbyite-type cubic structure were obtained. The crystallite size was found to be dependent on the precursor material.

E - P043 INFLUENCE OF MICROWAVE ANNEALING PROPERTIES OF THE SnO2 BASED VARISTORS

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Microwave and conventional furnaces were used to sintering SnO2 + 1.0%CoO + 0.05%Nb2O5 + 0.05% Cr2O3. The samples were prepared by the mixed oxide method and sintered at 1200°C with different annealing times ranging from 10 to 80 min. In order to investigate the influence of the microwave energy on the microstructural and electrical properties of the SnO2 based varistors, the samples were treated at 1200ºC for 60 min. It was observed that all the samples sintered in the microwave furnace presented good density (values higher than 95%). The sample sintered for 60 min presented excellent electrical properties with non-linear coeffcient () equal to 35. Ceramics sintered for periods of time ranging from 10 to 50 min presented a resistive characterisitic while for times higher than 60 min presented a conductor characteristic. The grain sizes for the samples sintered in the microwave oven were smaller than conventional oven ones.

E - P044 OPTICAL AND ELECTROCHEMICAL PROPERTIES OF SNO2:SB THIN FILMS PREPARED BY POLYMERIC ROUTE

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Thin solid film of SnO2:Sb (7 % mol of Sb) have been prepared by the polymeric precursor method using the dip-coating technique. The solution is based on metallic citrate polymerization using ethylene glycol. The thickness of the multiplayer film is typically 500 nm after a final heat treatment at 500 oC during 2hour. The electrochemical property of the film was studied in a 1M LiClO4/propylene carbonate electrolyte using cyclic voltammetry at different scan rates. In Situ UV-Vis spectroelectrochemcial measurements of SnO2:Sb film indicated that the film shows a weakly cathodic coloration during the intercalation process. The feasibility for use of this electrode as an ions storage for electrochromic devices was investigated.

E - P045 MICROESTRUTURAL AND DIELECTRIC PROPRIETIES OF CA(ZR0,05TI0,95)O3 THIN FILMS

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In this work, Ca(Zr0,05Ti0,95)O3 (CZT) thin films were prepared with success by the polymeric precursor method (PPM). The formation of the CZT perovskite phase was confirmed by Differential scanning calorimetry (DSC), Thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD). The film showed good structures crystalline and no presence of secondary phase was identified by XDR. This indicates that the film was crystallized in a single phase when calcinated at 640 ºC. The roughness and size of the grains were analyzed by AFM. SEM was used to obtained the thickness of the film. The electric properties of the (CZT) film was measured in the Pt–CZT–Au sandwiched configuration. The ferroelectric hysteresis was determined using Radiant Technologies RT6000HVS ferroelectric test system. These loops were traced using the \"charge\" program included in the software of the RT6000HVS in a virtual ground mode test device. The capacitance–voltage (C–V) properties were measured using a Hewlett-Packard (4194A) impedance/gain phase analyzer, in which the capacitance value was taken using a small AC signal of 10 mV at 100 kHz. The dielectric constant and dissipation factor were measured as a function of frequency by using a frequency, in the 100 Hz–10 MHz range. The leakage current–voltage (I–V) characteristic was determined with a voltage source measuring unit (Keithley237). All the measurements were taken at room temperature.

E - P046 INFLUENCE OF ADDITION OF SEEDS ON THE MICROSTRUCTURE AND ELECTRICAL

PROPERTIES OF SNO2 BASED LOW VOLTAGE VARISTOR

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Tin dioxide based ceramics have been extensively studied in the last years aiming at its applications in high voltage varistor. However, the application of these ceramics as low or high voltage varistor is directly associated to the formation of the potential barrier and the number of then. The varistor's nominal voltage or breakdown voltage (Eb) is proportional to the number of these barriers, i.e., the increasing of grain size decrease the number of barriers and consequently the Eb. The goal of this work was to present a SnO2 based low voltage varistor obtained by addition of seeds. The seeds were obtained of systems contend SnO2 doped with 1 mol % of CuO, 1 mol % of CoO or 1 mol % of Cr2O3. The varistor compositions were obtained by homogenization after addition of seeds, pressing and sintering at 1500oC for 4 hours in water vapor atmosphere. The micrographs of samples presented high density and mean grain size of 13-18 m. The electric characterization of ceramics with seeds of Cu and Co shows nonlinear coefficient () values of 6.0 and 6.7 respectively. The Eb values for these systems are 350 and 360 V/cm respectively, resulting in low voltage varistor. However for system with Cr was observed  = 10.1 and Eb = 820 V/cm, behaviour due to Cr increase the number of potential barriers and decrease the grain size.

E - P047 INFLUENCE OF THE OXIDES ZINC AND MOLIBDENIUM, IN THE PROPERTIES VARISTORS OF SYSTEMS SnO2.CoO.Nb2O5

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The practical interests in the properties of the tin dioxide have been being a lot investigated last decade. In this work was analyzed the influence of the zinc and molybdenum oxides in the system SnO2.CoO.Nb2O5 in the densification mechanisms (CoO/ZnO), electric conductibility (Nb2O5), formation of the potential barrier (MoO3).The systems were prepared by varying the concentration of the zinc and molybdenum oxides, keeping constant the others cations. The results showed that for the concentration superior to 25% of ZnO the materials present low densification and a temperature lower temperature of maximum retraction rate. The increase of ZnO concentration implied in the decrease of the density of the systems and consequently, in the degradation of microstructure associated to the reactions between ZnO and the other oxides, favoring, in the region of the grain bondary, the appearence of secondary phases deleterious to the densification mechanism and caused the degradation of the electric property. Analyses of electric conductivity and complex impedance confirmed a decrease of the conductivity in function of the increase of the concentration of ZnO and MoO3, related to the limitation of the current through the grain boundary.

E - P048 (TA, CR, CO, PR) DOPED TIO2 BASED ELECTROCERAMICS

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The addition of different dopants, as well as the processing parameters, influences the densification, the mean grain size and the electrical properties of the TiO2-based varistor ceramics. Dopants like Ta2O5 have an especial hole over the barrier formation at the grain boundary in the TiO2 varistors, increasing the nonlinear coefficient and decreasing the breakdown electric field. In this paper, will presented the microstructural and electrical properties of (Ta, Cr, Co, Pr) doped TiO2 systems. It will be demonstrate that some of this systems exhibit electrical properties that possibility their use like low voltage varistors.

E - P049 RETICULATED CERAMIC OBTAINED FROM NATURAL RAW MATERIALS: PRODUCTION AND OPTIMIZATION

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In this work, were studied mixtures of the natural raw materials: feldspar, kaolin and quartz, with the objective of producing reticulated ceramic, using the impregnation technique in polymeric foam. The simplex centroid experimental design was employed, associate to the factorial design 2^2, aiming at investigating the factors: temperature (1100-1200 ºC) and heat treatment plateau (0-2 h). The raw materials were characterized by means of XRD, XRF, AAS, and Hg porosimetry. The TG and DTA curves of the polyurethane foam, the raw materials and the mixtures were obtained. The mixtures were characterized by water absorption, apparent porosity and bulk density. Polyurethane foams were impregnated with suspensions with 50% v/v containing 0.50% w/w of PAA-Na. The apparent porosity results of the mixtures heat treated at 1200 ºC for 2 h indicated for such experimental condition the best sintering results. The reticulate ceramics produced were analyzed by SEM photomicrographs and XRD. With the intersection of the contour plots of the answer allowed to identify an experimental area with pre-established properties: viscosity of the suspensions lower than 250 mPa.s, apparent porosity lower than 5.0%, volumetric retraction lower than 30% and mechanical strength of the reticulated ceramic higher than 1.0 MPa. The optimized composition obtained to product reticulated ceramic, at 1200 oC/2 h of landing, was of approximately 4242.6% feldspar;29.3 <% kaolin <34.7; and 22.7 <% quartz < 28.1.

E - P050 MICROSTRUCTURAL CHARACTERIZATION OF PZT THIN FILMS PREPARED BY PECHINI METHOD AND ANNEALED IN DOMESTIC MICROWAVE OVEN

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In this paper we studied pure and Nb doped PbZr0.4Ti0.6O3 thin films prepared by the Pechini method and deposited by spin coating on Pt/Ti/SiO2/Si (100) substrates and annealed at different temperatures in a domestic microwave oven modified to control the temperature. The obtained films present preferential orientation in the crystallographic plane (100). The predominant phase is tetragonal perovskite. The AFM images show a smooth, dense and uniform surface with well-developed grain structures with medium-sized grains of 200 nm for the pure PZT thin films annealed at 600ºC for 15 min. The 1.0 mol % Nb doped PZT thin films showed typical rosette structures consisting of two phases: a rosette phase and a phase surrounding the rosettes known as pyrochlore particles. Considering that the perovskite phase is formed from the pyrochlore phase, the inhibition of crystallization caused the Nb doping in PZT thin films is the main cause of the appearing of pyrochlore containing matrix.

E - P051 ELECTRICAL BEHAVIOR OF TIN OXIDE BASED VARISTOR HEAT TREATED IN VACUUM

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Tin oxide varistor is very sensitive to the oxygen partial pressure used during sintering or heat treatment. In this work tin oxide based varistor with composition of 99.15% SnO2, 0.75% TiO2, 1.0% Co2O3, 0.05% Nb2O5, 0.05% Cr2O3 all in mol were sintered at 1250C during 90 minutes and the microstructure and electrical properties were characterized by SEM, JxE curves and impedance spectroscopy. To verify the effect of oxygen on the voltage barrier height the varistor sample was heat treated at 900C for 1 h in vacuum and its electrical properties was characterized again. The results showed that sintered samples submitted to heat treatment in vacuum showed a significant decrease in all varistor properties. This suggests that adsorbed oxygen at grain boundaries are very important for voltage barrier formation and its desorption during heat treatment in vacuum is responsible for the decrease of the varistor properties.

E - P052 MOLECULAR-LEVEL MANIPULATION OF V2O5 LAYER-BY-LAYER FILMS TO CONTROL ELECTROCHROMOGENIC AND ELECTROCHEMICAL PROPERTIES

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This work describes how manipulation at the molecular level of V2O5/polymer nanocomposites built with the layer-by-layer (LBL) technique promotes enhancement of charge storage capability, new

electrochromic effects, and control of ionic flux. By changing the film architecture we control the amount of PANI participating in the redox processes of the LBL films. As a consequence, the films display the electrochemical profile of V2O5 and the chromogenic properties of PANI. Further control of the properties of the nanoarchitectures is achieved by adsorbing V2O5/PANI LBL films onto a cast PANI film. By changing the time of immersion of the PANI-V2O5/PANI system into a solution of LiClO4/propylene carbonate (PC), we were able to monitor the mass gain/loss with an electrochemical quartz crystal microbalance (EQCM) as a function of charge (q) and control the intercalating/deintercalating species. The electrochemical and electrochromic properties of layer-by-layer nanoarchitectures of V2O5 alternated with a blend of poly(ethylene oxide) (PEO) and chitosan have also been examined. Using a blend was important since multilayers of PEO/V2O5 could not be built. A pronounced effect from PEO is observed in the migration/diffusion process, according to cyclic voltammetry and impedance spectroscopy data. The charge injected was 8.02 mC cm-2 at 20mVs-1. The importance of these results for the production of Li secondary microbatteries and electrochromic devices is discussed.

E - P053 DEVELOPMENT OF ZrO2-TiO2 CERAMIC AS SOIL HUMIDITY SENSOR FOR APLICATION IN ENVIRONMENTAL MONITORING

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Considering the need for acquaintance and follow up of the risk-areas, specifically hillside landslide, efforts have been made to develop more versatile, reliable sensors and sensor systems with smaller cost. In the sense, search of new materials, modeling study of sensor and development of new measure techniques and processing of signals have been orientating the progress in this area. In this work are shown and discussed the results of the characterization analyses of sensor elements of ZrO2-TiO2 porous ceramic for application as soil humidity sensor. These ceramics were obtained from the mechanical mixture of ZrO2-TiO2 powders and sintered at 1000, 1100 and 1200 ºC, to obtain different pore size distributions. The characterization of the ceramic was carried out using measurements of B.E.T. (specific surface area), nitrogen and mercury porosimetry (distribution of nano, meso and micropore size), scanning electronic microscopy (microstructure) and X-rays diffraction (crystalline phases). The porous ceramic characterization as soil humidity sensor element was accomplished through impedance and capacitance measurements using a RLC bridge. The ceramics were immersed in the selected, previously characterized soils. The results obtained for specific area, pore size distribution curves, microstructure, crystalline phases and sensibility to the soil humidity showed that the ZrO2-TiO2 ceramic presents a great potential to be applied as sensor element for soil humidity monitoring.

E - P054 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.

E - P055 INFLUENCE OF THE EXCESS OF PB ON THE MICROSTRUCTURAL PROPERTIES OF LEAD LANTHANUM TITANATE CERAMICS OBTAINED BY COMBUSTION SYNTHESIS

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Lanthanum-modified lead titanate (PLT) has become popular because it possesses interesting properties such as a lower Curie temperature, a lower coercive field, and because of its great potential for nonlinear optical and electro-optical applications. In this work, ferroelectric ceramic powder of lanthanum-modified lead titanate Pb1-1.5xLaxTiO3 (plt), with x = 0.17, 0.20 and 0.23, was prepared by combustion synthesis reaction. An analysis was made to identify the effect produced by the excessive addition of varying concentrations of lead to the precursor mixtures on the microstructural properties of the resulting PLT ceramics. The precursors used here, lead nitrate, lanthanum nitrate, and tetraethylorthotitanate, were weighed according to the desired stoichiometric ratio, mixed with a fuel, and ignited on a preheated hot plate. An X-ray diffraction analysis (XRD) revealed the presence of perovskite and lead oxide phases in all the ceramics obtained. Scanning and transmission electronic microscopy (SEM and TEM) revealed nanometric grains in all the ceramics to which different amounts of excess lead had been added. X-ray photoelectron spectroscopy (XPS) was employed to investigate the surface chemical composition, indicating the presence of La3+, Ti4+, and two Pb species.

E - P056 ANALYSIS OF THE CRYSTAL STRUCTURE OF COBALT DOPED LANTHANUM CHROMITES

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Lanthanum chromite (LaCrO3) is one of the materials most suitable for use in solid oxide fuel cells (SOFC), as it is very resistant to oxidant as well as to reducer environments at high temperatures (about 10000C). It presents an adequate (p-type) electric conductivity at the operation temperatures of SOFC. For this reason, this material may act as a spacer as well as an interconnect, that is, as a receptor and a conductor of electric charge. LaCrO3 structure is considered to be a pseudo-perovskite, or distorted perovskite. At some temperature between 200 and 2600C it undergoes a crystallographic transformation from orthorhombic to rhombohedric structure, in which volume change occurs. In the present contribution we present the results of refinements of crystallographic structures of lanthanum chromite doped with cobalt (LaCr1-yCoyO3, y = 0.10; 0.15; 0.20; 0.25). The crystalline structures were analyzed by means of the Rietveld method for structure refinement. The powder X ray diffraction data were obtained from anomalous diffraction at the chromium absorption edge (E = 5.85 KeV). Samples were sinthetized at IPEN (Instituto de Pesquisas Energéticas e Nucleares), in CCTM (Centro de Ciência e Tecnologia de Materiais), by combustion reaction using nitrates and urea as fuels. The X ray diffraction was performed at LNLS (Laboratório Nacional de Luz Síncrotron). Variations on the lattice parameter values caused by partial replacement of cobalt at chromium sites were observed.

E - P057 THE CHARACTERIZATION OF SNO2- FILMS DEPOSITED ON DIFFERENT SUBSTRATES

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In this work, a study of SnO2 films preparation by the polymeric precursors method was presented. The films were deposited by dip coating on glass and alumina substrates. The samples were prepared controlling the immersion and emersion speed and temperature. The obtained films were characterized by scanning electron microscopy, X-ray diffraction, electrical measurements and absorption spectrum in the UV-Visible region. The resultant film on glass substrate presented a tickness of 0,46 micrometre.

E – P058 MIXED CONDUCTION MEMBRANES: PANI IN SITU POLYMERIZATION IN SPEEK MATRIX

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Mixed ion and electron conductive ceramic materials, like perovskite, have gained relevance in membrane technology. In this work we describe the preparation and characterization of a mixed

conduction membrane by the aniline in situ polymerization, in a sulfonated poly(ether ether ketone) [SPEEK] film. The doping of polyaniline [PANI] with SPEEK was investigated by infrared spectroscopy and it was observed an intense broad band at 1152 cm-1. This is a characteristic band of doped PANI, due to an interaction of sulfonic with imine groups. Scanning electronic micrographs show a fine colloidal dispersion of PANI.SPEEK particles, which coalesces and form aggregates, with elongated structures, percolated through the membrane. Oxidant/aniline molar ratio variation originated electronic conductivity values (four-probe method) with the same order of magnitude. Membranes prepared by PANI in situ polymeriazation with 80 wt. % of SPEEK presented an electronic conductivity of 9.8 10-6 S cm-1 and, by redoping with 1 molar HCl, the conductivity was 4.9 10-5 S cm-1. The membrane thermal stability at high temperatures was investigated by thermogravimetry and infrared spectroscopy. It described a first weight-loss at approximately 300 oC assigned to sulfonic group weight loss. The PANI doping efficiency with SPEEK, the conducting path formation at the percolation threshold and a good thermal stability of the system makes possible to obtain mixed conduction membranes.

E – P059 MICROSTRUCTURAL AND OPTICAL CHARACTERIZATION OF BAMOO4 PREPARED BY CHEMICAL SOLUTION METHOD

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Recently crystal structures based on the scheelite-type structure, such as BaMoO4, have attracted much interest, which is related to their potential application as scintillating medium and use in electro-optic applications (i.e. solid-state laser and in optical fibre applications). In this sense, numerous investigations on the luminescent properties of these materials have been carried out and have shown that, these compounds produce green luminescence. A variety of preparation techniques have been proposed to produce these materials, among these are solid state reaction, hydrothermal synthesis, sputtering and the sol-gel rocesses. In addition, considering the importance of BaMoO4 system, thin films prepared by soft chemical processing are difficult to find in the literature.Hence, in this work, we will report some experimental data based on a soft chemical method, the so-called polymeric precursor method, to produce thin films of BaMoO4 compounds on a Si substrate. For this, we have deposited the polymeric resin of BaMoO4 by using a spin coater. After spinning onto the substrates, the films were annealed at 200°C between 2 and 16h and at 600°C for 2h in air. In order to study the structural, morphological and optical properties, these films were characterized by XRD, AFM and photoluminescence spectroscopy. This method is very promising alternative for better three-dimensional, molecular-scale control of nanostructured materials and for being environmentally friendly.

E - P060 INFRARED TO VISIBLE FREQUENCY UPCONVERSION-BASED TEMPERATURE SENSING IN ER3+ DOPED PLZT TRANSPARENT CERAMICS

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Rare-earth ions-doped PLZT transparent ferroelectric ceramics are new materials that have been calling much attention due to a variety of possible device applications, based not only on their electrical and structural properties, but also on their interesting optical characteristics. Neodymium doped samples, for instance, have proven to be potential media for laser generation at 1064 nm, and erbium doped samples lead to the same perspective at around 1550 and 2800 nm. Besides being of utmost technological importance nowadays, these emissions can be pumped by low cost, high-power, diode lasers at 800 and 980 nm. In the visible portion of the spectrum, and while pumped at 980 nm, PLZT:Er3+ also presents intense green emission due to infrared to visible upconversion. Because the two emitting levels (2H11/2 and 4S3/2) lie very close in energy, they are thermally coupled and their emission lines intensity ratio is very sensitive to temperature changes. Since the ceramic presents excellent stability at high temperatures (an advantage over glasses used for this purpose), a low cost, optical sensor can be developed. In order to get a full insight of the radiative and non radiative properties of the system, samples of PLZT:Er3+ were prepared through a co-precipitation method and sintered by uniaxial hot pressing, in O2 atmosphere. The green upconversion signal was analyzed as a function of temperature (up to 500 C). A detailed spectroscopic and structural investigation is presented.

E - P061 ELECTROCHEMICAL BEHAVIOR OF RUPIC COMPLEX IMMOBILIZED USING DIFFERENT APPROACHES

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The electrochemical property of the RuCl3(PP)(Mepy) >(PP=Ph2P(CH2)4PPh2, Mepy=CH3C5H4N), Rupic, in CHCl3 is governed by the >formation of species Ru2Cl5(PP)2, Ru2(PP)2Cl4(Mepy) in the reduction to RuCl2(PP). However, this behavior may change if Rupic is in the form of cast, spin-coated or Langmuir-Blodgett (LB) films or incorporated into a carbon paste electrode (CPE). This work describes the electrochemical response of immobilized Rupic using different methods and its possible use in electrocatalysis. Y-type LB films were produced onto ITO using a KSV5000 Langmuir trough. The cyclic voltammograms (CV) were obtained with a MQPG-01 potentiostat. The reference was a SCE or an Ag/AgCl, Pt was used as counter electrode, ITO or CPE (25:1 carbon/Rupic) was employed as working electrode, immersed in a HCl/KCl 0.1 M solution. The CV for cast and spin films presented no redox processes but for the LB films and for CPE/Rupic one redox process was observed, at Epa=0.45V, Epc=0.25V vs SCE, and Epa=0.27V, Epc=0.20V vs Ag/AgCl, respectively. The difference is attributed to the lack of order in the cast and spin-coated films, in contrast to the LB films. For LB films and CPE/Rupic, the redox processes were ascribed to the Ru3+/2+ charge transfer. These results indicate that immobilization of Rupic could impair the formation of species occurring in solution. Furthermore, immobilization in an ordered structure or in a conductive matrix opens up the possibility of using Rupic in electrocatalysis.

E – P062 STUDY OF CERAMIC DEVICES SnO_2-BASED FOR OBTAINING OF LOW TENSION VARISTOR.

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SnO_2 is n-type semiconductor material, it has crystalline structure similar of the rutilo and it has high electronic mobility that qualifies it potentially for using as varistor. Many varistors were developed for applications of high tension, today the attentions are on the varistors of low tension, it operates typically in the area from 3 to 12V. With the objective of producing dense ceramic systems with high values of non linear coefficient and low values of rupture tension and escape current, SnO_2 was dopped through the conventional method of mixture of oxides in mill of balls, with 1,0mol% ZnO, 0,01mol% WO3, 0,025 mol% and 0,050mol% of the following doppings: Nb_2O_5, Al_2O_5, VCl_3. The resulting powder was pressed in the form of tablets and sintering in tubular oven for 3h to 1400^{o}C. It was obtained the densities and the medium sizes of grains of the samples, it was obtained values up to 98% and 9? m, respectively. And through studies of electric characterization, it was obtained values of non linear coefficient (?) getting 51, rupture tension (E_r) of 127V/0,94mm and escape current (I_f) equal the 0,08mA/cm^2. The conclusion was that the studied ceramic systems presents varistors characteristics, with excellent non linear coefficient and escape current values, however the rupture tension values are still elevated, characterizing an average tension varistor

E – P063 SYNTHESIS AND CHARACTERIZATION OF COPOLYMERS WITH DIFFERENT RATIOS OF PPV AND DCNPPV UNITS

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The use of PPVs type polymers (*poly-p-phenylenevinylene*) as active layer in organic light emitting diodes (OLEDs), has received much of attention in the last two decades, resulting in commercial projects developed by great companies like DuPont, Kodak and Philips. However some intrinsic problems related to the poor efficiency and short display life impossibility the utilization of this kind of technology. For this mentioned reasons, the search for PPV like polymers with CN connected to the backbone is justified by the diminishment of the electron injection barrier and by the improvement in the electron mobility in the polymer matrix. Nevertheless the synthesis of this kind of materials has been to much complicate and the obtained polymer showed a sequence of problems. Facing theses problems, this work describe the

preparation of a series of copolymers prepared with different ratio of PPV and DCVPPV units using the Wessling methodology. These copolymers, after purification and thermal treatment were characterized by FTIR, UV-vis, GPC and cyclic voltammetry. The obtained results showed that the copolymers films presented a low conjugation defects degree, a high molecular mass and a ratio of PPV and DCNPPV unit correspondent to the monomeric proportion using to the polymers preparation.

E – P064 ELECTRONIC CONDUCTIVITY IN NANOSTRUCTURED TIN DIOXIDE: INFLUENCE OF OXYGEN SPECIES AND ANTIMONY OXIDATION STATE

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Tin dioxide is a wide bandgap semiconductor with very many application such as gas sensors and optoelectronic devices. When undoped, it presents n-type conduction due to oxygen vacancies and interstitial tin atoms. Adsorbed oxygen at grain boundary may also contribute to conduction mechanisms. Sb doping increases significantly the conduction if incorporated as +5 oxidation state. In this work we present electrical characterization data modeled according to a nanostructured material model where grain boundary scattering is the dominant mechanism even though intragrain scattering mechanisms are considered as well. It becomes clear when the nanoscopic dimensions of the material is taken into account. The deposition by sol-gel dip-coating process produces material within 5 to 15 nm large grains. Then, the material presents a large amount of crystallites and, besides, the grain boundary depletion layer may be as large as half of the grain width, concomitant with high potential barrier. EXAFS and XANES measurements, carried out at LURE (France) and Hasylab (Hamburg, Germany), indicates that Sb is mostly in the 5+ oxidation state. Considering that our sol-gel process has some intermediate firing steps, we are now investigating when the antimony change from Sb3+ to Sb5+ actually takes place. Besides, synchrotron radiation data also indicate a substitutional position of antimony at a tin lattice site. These data bring new light to the conduction model.