

3RD BRAZIL MRS MEETING

October 10-13, 2004

<u>SYMPOSIUM</u>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)

the degree of agglomeration with the thermal treatment.

E - 0010 INFLUENCE OF THE SYNTHESIS METHODOLOGY ON THE PHASE FORMATION OF (1-X)PB(MG1/3NB2/3)03-XPBTI03 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 700°C for 1h. When using the PPM, a higher temperature to obtain the PMN-PT powders was required (800°C for 2h) despite the powders were more reactive, the perovskite phase was not so stable.

E - 0011 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 _

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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 - 0012 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 700°C. 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 - 0013 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.

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 - 0014 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 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 - 0015 STRUCTURAL AND OPTICAL PROPERTIES OF BI1.5ZNNB1.507 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 - 0016 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.