ICAM2009
11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25

Effect of atmosphere on stability of PLMN-13PT powder.

F. A. Londoño(1)*, J. A. Eiras(1) and D. Garcia(1)

(1) Grupo de Cerâmicas Ferroelétricas, Departamento de Física/Universidade Federal de São Carlos, Rodovia Washington Luís, km 235 - SP-310, São Carlos - São Paulo – Brasil, CEP 13565-905. e-mail: flondono@df.ufscar.br

Abstract –Materials of lead based magnesium niobate ceramics are difficult to synthesize single phase. It is important to obtain ceramics with good optical and electrical properties. In this study, powder de PLMN-13PT was obtained by two stage process. The phase formation was investigated by X-ray diffraction. The thermal behavior was studied by simultaneous TG/DTA measurements. The microstructural features of the powders were investigated by SEM. Little influence of the atmosphere was observed on the stability of PLMN-13PT powder, except for it calcined in N atmosphere. This study is important for our knowing, in the fabrication of transparent PLMN-PT ceramics.

Materials of lead based magnesium niobate ceramics are relaxor ferroelectric materials, that exhibit good dielectric and electrostictive properties [1,2], but they are difficult to synthesize single phase without coexisting pyrochlore-type compound and other phases. Various synthetic techniques for suppressing the formation of pyrochlore-type compound have been reported, for example, two-step process via PbO and $MgNb_2O_6$ columbite method [3]. It is important to obtain ceramics with good optical and electrical properties.
In this study, powder de (1-x)(Pb,La)(Mg,Nb)O_3-x$PbTiO_3$ with x=0.13 was obtained by two stage process, the columbite method, proposed by Swartz and Shrout [3]. In the firs stage, MgO and Nb_2O_5 powders were prereacted at 1000^oC in air to form columbite. In the second stage, the prefabricated $MgNb_2O_6$ was reacted with appropriate amounts of PbO, TiO_2 and La_2O_3 at 900 ^oC in different atmospheres (O_2, Ar, N, vacuum and air). The thermal behavior was studied by simultaneous TG/DTA measurements. The phase formation was investigated by X-ray diffraction and the microstructural features of the powders were investigated by scanining electron microscopy (SEM).
The thermal behavior of PLMN-13PT powders are quite similar to reported in the literature [4], below 400 oC two endothermic effects due to the volatiles elimination (water from $La(OH)_3$ dehydration and organic residuals originated from solvent). The broad exothermic effect situated at 680 ^oC, is probably determined by the perovskite formation. The slow and continuous mass loss over 800 ^oC, is probably due to the PbO volatilization. X-ray diffraction patterns for PLMN-13PT powders calcined in different atmospheres presented the perovskite as phase majority (JCPDS 81-861), a small amount of pyrochlore phase (JCPDS 33-769) and other phases were detected.
The diffraction data for the powders obtained emphasized the little influence of the atmosphere on the stability of PLMN-13PT powder (except the powder calcined in N atmosphere) as can be seen in the table 1.
This study is important for the best of our knowing, in the fabrication of transparent pore-free, homogeneous microstructure and structure of the PLMN-PT ceramics.

Table 1: Unwanted phases in PLMN-13PT.

Material and atmosphere	% Pyrochlore phase	% Other phases
PLMN-13PT- vacuum	2	3
PLMN-13PT-O_2	3	3
PLMN-13PT-N	10	4
PLMN-13PT-Ar	2	3
PLMN-13PT-Air	3	3

References

[1] A.J. Moulson, J.M. Herbert, Electroceramics, 2nd ed., John Wiley & Sons, Chichester, 2003
[2] K. Uchino, Ferroelectric Devices, Marcel Dekker, New York, 2000
[3] L. Swartz and T. R. Shrout, Mater. Res. Bull., 17 (1982) 1245.
[4] A. Ianculescu et al., J.Thermal Anal. Calorimetry, Vol 80 (2005) 663-670