$LiNbO_3:Eu^{3+}$ / SODA-LIME-SILICA GLASS COMPOSITES

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We have prepared soda-lime-silica glass $LiNbO_3:Eu^{3+}$composites by sintering at 700, 850 and 1000 °C[1]. The monolithic samples so obtained were analyzed by Energy Dispersive X-rays Analysis (EDX), Micro-Raman Scattering and Eu^{3+} Luminescence Spectroscopy. $LiNbO_3:Eu^{3+}$ rich regions were identified in the samples treated at 700 and 850 °C, suggesting that the sintering methodology can be utilized in the preparation of such composites. We observed dissolution of the crystalline phase $LiNbO_3:Eu^{3+}$ into the glassy matrix in the materials treated at 1000 °C.

Glass-ceramics can be obtained either by direct nucleation and growth of a crystalline phase from a mother glass or by incorporation of previously prepared crystalline phases in glass hosts by means of conformation and sintering of powdered precursors[2]. In this work we studied this last method for the preparation of ferroelectric-glass composites involving two technologically important phases: lithium niobate crystal[3] and soda-lime-silica glasses.

Three different sintering temperatures were used and micro analytical techniques, i.e., Scanning Electron Microscopy (SEM), Energy Dispersive X-rays Analysis (EDS), micro-Raman Scattering and Eu^{3+} Luminescence characterized the materials so obtained.

$LiNbO_3:Eu^{3+}$ was prepared by the polymeric precursor method[4] (the so-called "Pechini Method"). Glass spheres of soda-lime-silica composition were utilized and crystal-glass mixtures were sintered at 700, 850 and 1000 °C during 2 hours in an electric furnace. The composite materials obtained were analyzed with a Scanning Electron Microscope LEO S440 equipped with a microanalysis setup (energy dispersive Link ISIS L300 with SiLi detector, ultra fine window ATWII, from 133eV to 5,9KeV). All analyses were performed with 20kV electrons accelerating voltage. The images were produced from backscattered electrons with a BSE detector. The EDS microanalysis resolution is about 133eV, penetrating about 1.5 to 5 μm, depending on the material density in the analyzed point.

Figures 1a and 2a show the micrographs obtained for the composites treated at 700 and 850 °C, respectively. $LiNbO_3:Eu^{3+}$ rich regions (white spots) were identified in both cases. The insets show in more detail the analyzed regions from which EDS spectra were obtained. Relatively intense peaks of Nb and Eu^3 were observed. The micrograph of the composite treated at 1000 °C is shown in Figure 3a. The absence of $LiNbO_3$ rich regions suggests the dissolution of the crystalline phase in the vitreous matrix at that temperature. The EDS spectrum (Figure 3b) shows less intense lines for Nb peaks together with intense peaks of Si and Ca.

The micrographs show $LiNbO_3:Eu^{3+}$ regions in the composite treated at 700 and 850 °C, but not in the material treated at 1000 °C. The EDS spectra of composites treated at 700 and 850 °C show intense peaks of Nb and peaks of Eu. The Nb peaks appear less intense in the spectrum of a composite treated at 1000 °C.

New $LiNbO_3:Eu^{3+}$/soda-lime-silica glass composites were obtained and characterized. The present results agree with previous reports of X-rays Diffraction and Eu^{3+} Luminescence Spectroscopy and also show that for high temperatures (above 1000 °C) the crystal phase dissolves in the glass environment.

ACKNOWLEDGEMENTS

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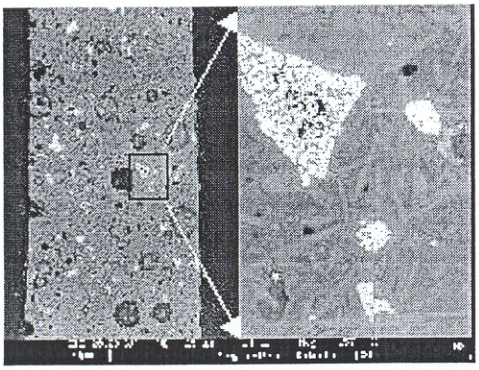
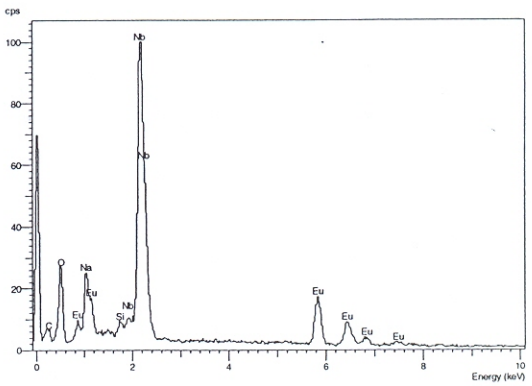
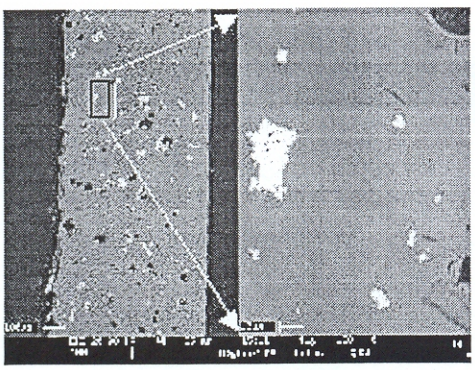
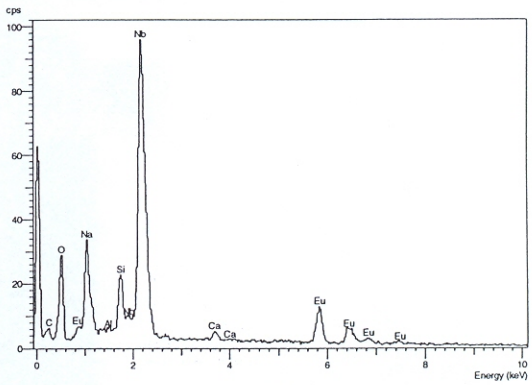




(a) (b)

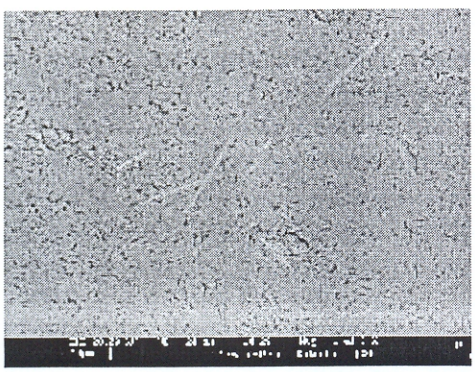
Figure 1. Sample treated at 700°C; (a) micrograph, (b) EDS spectrum.

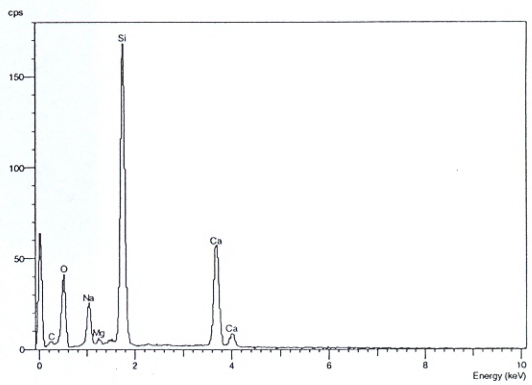




(a) (b)

Figure 2. Sample treated at 850°C; (a) micrograph, (b) EDS spectrum.





(a) (b)

Figure 3. Sample treated at 1000°C; (a) micrograph, (b) EDS spectrum.