

Poster Presentation

MM.P05

Vitreous and Crystalline Phosphates : elaboration and electrical properties

F. Dardar¹, A. El jazouli², A. Lachgar³, M. Gross⁴, C. Day⁴

¹Faculte des Sciences Ben M'sik Universite Hassan II, Department of Chemistry, Casablanca, Morocco, ²Department of Chemistry, Casablanca, Morocco, ³Wake Forest University, Department of Chemistry, Winston Salem, USA, ⁴Department of Chemistry, Winston Salem, USA

Interest in mixed metals titanium phosphates, both in glass and crystalline forms, for their properties as ionic conductors or non-linear optical materials, has led to large number of studies of these materials [1-3]. Our investigation of Na₂O – Li₂O – CaO – TiO₂ – P₂O₅ system led to the discovery of Na_xLi_yCa_zTi(PO₄)₃ solid solutions which exist in both glassy and crystalline forms. Here the preparation of Na_xLi_yCa_zTi(PO₄)₃ will be described. The compounds were structurally characterized by PXRD, and their chemical and physical characterization were completed using DTA, XRD, UV-visible, Raman spectroscopy and ionic conductivity measurements. Na_xLi_yCa_zTi(PO₄)₃ glasses have been prepared by heating stoichiometric amounts of Na₂CO₃, Li₂CO₃, CaCO₃, TiO₂ and NH₄H₂PO₄ in a platinum crucible at 1050 °C. The microcrystalline samples were obtained by crystallization of the corresponding glasses at 640°C or by standard solid state preparation at 700 °C. The values of the characteristic temperatures T_g (glass transition), T_c (crystallization) and T_f (fusion) of the glasses are found in the following ranges: 400 – 500°C for T_g, 440 – 650°C for T_c and 780 – 830 °C for T_f. Rietveld refinements of powder X ray diffraction data collected on crystalline samples of Na_xLi_yCa_zTi(PO₄)₃ shows the existence of a solid solution in the domain $0 \leq X \leq 0.5$ and the presence of a mixture of phases in the domain $0.5 < X \leq 1.5$. The crystalline phases belong to the Nasicon family, space group R32. Their structure, determined from refinement of powder diffraction patterns , consists of a 3D network of AO₆ (A = Ti, Na, Li, and Ca) octahedra and PO₄ tetrahedra linked by corners. Sodium atoms fully occupy the M(1) sites and partially the M(2) sites.

[1] E. M. Vogel, E. W. Chase, J. L. Jackel and B. J. Wilken, *Fabrication of thin film nonlinear optical glasses using pulsed exciter laser deposition, Applied optics*, 28, 649 (1989)., [2] E. Fargin, A. Berthereau, T. Cardinal, J.J. Videau, A. Villesuzanne and G. Le Flem, *Contribution of theoretical chemistry to the investigation of optical non linearities in glasses, Ann. Chim. Sci. Mat.*, 23, 27 (1998)., [3] J. L. Souquet, T. Pagnier and M. Fouletier, *Electrochemical properties of phosphate based semi conductive glasses, Solid State Ionics* 9 – 10, 694 (1983)

Keywords: Nasiglass, Synthesis, Ionic conductivity