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2013-02-26

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Reunião Anual de Usuários do LNLS, XXIII, 2013, Campinas.
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Size-dependent phase transitions in nanostructured zirconia-scandia solid solutions

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Size effects on phase stability and phase transitions in technologically relevant materials have received growing attention. Several works reported that metastable phases can be retained at room temperature in nanomaterials, these phases generally corresponding to the high-temperature polymorph of the same material in bulk state. Additionally, size-dependent shifts in solubility limits and/or in the transition temperatures for on heating or on cooling cycles have been observed.

ZrO_2-Sc_2O_3 (zirconia-scandia) solid solutions are known to exhibit very high oxygen ion conductivity provided their structure is composed of cubic and/or pseudocubic tetragonal phases. Unfortunately, for solid zirconia-scandia polycrystalline samples with typical micrometrical average crystal sizes, the high-conductivity cubic phase is only stable above 600°C. Depending on composition, three low-conductivity rhombohedral phases (β, γ and δ) are stable below 600°C down to room temperature, within the compositional range of interest for SOFCs. In previous investigations, we showed that the rhombohedral phases can be avoided in nanopowders with average crystallite size lower than 35 nm.

In this work, the dependences of phase stability and solid state phase transitions on the crystallite size in ZrO_2-10, 12 and 14 mol% Sc_2O_3 nanopowders were investigated by X-ray powder diffraction (XPD) at the D10B-XPD beamline of the LNLS [1]. The average crystallite sizes were within the range of 35 to 100 nm, approximately. At room temperature these solid solutions exhibit mixtures of a cubic phase and one or two rhombohedral phases, β and γ, with their fractions depending on composition and average crystallite size, while at high temperatures these solid solutions become cubic single-phased. The size-dependent temperatures of the transitions from the rhombohedral phases to the cubic phase at high temperature were determined through the analyses of a number of XPD patterns. These transitions were studied on cooling and on heating, exhibiting hysteresis effects whose relevant features are size and composition dependent.

[1] P.M. Abdala, A.F. Craievich, D.G. Lamas, RSC Advances 2 (2012) 5205

Acknowledgements: This work was supported by LNLS (proposals D10BXPD-7296 and 9195), CNPq (PROSUL program), ANPCyT and CLAF.