Nanocrystalline ZrO_2 powders prepared by sol-gel method: The role of carbon on the phase stabilization

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Abstract

Zirconia nanocrystalline powders were prepared by sol-gel with acetates precursors. The samples were characterized by thermal analysis, X-ray diffractometry and FT-IR spectroscopy to investigate the influence of structural carbon on stabilization of high temperature crystalline phases of Zirconia. Residual carbon remains in Zirconia structure even at heat treatment temperature of 700°C and phase transformation occurs on pure Zirconia only after carbon losses near 850°C. Then, carbon plays important role to phase stabilization of Zirconia tetragonal phase.

Nanocrystalline powders of pure and 7.0 mol % Pr^{3+} doped zirconia were prepared by sol-gel route from zirconium and praseodymium acetate. The gels were dried at 110°C/24h in air and calcined during 4h at 426°C, 520°C, 700°C, and 950°C to study the influence of carbon on phase stabilization. The dried gels and powders obtained from calcinations were characterized by thermal analysis (DTA/TG), X-ray diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FTIR). Both pure ZrO_2 and 7.0 mol % Pr_2O_3 doped ZrO_2 presented tetragonal phase after calcinations at 426°C/4h. The tetragonal phase remains stable for both pure and doped zirconia up to heat treatment at 700°C/4h. For the dried gel calcined at 950°C/4h a significant change occurs: pure zirconia powders presented tetragonal and monoclinic phases, and 7.0 mol % Pr_2O_3 doped Zirconia remains stabilized in tetragonal phase. These phase transformations occurred with mass loss observed in TGA curves attributed to carbon losses. FTIR characterization shows residual carbon at relatively high temperatures remaining mainly as Zr-O-C bonds. The residual carbon content plays an important role on tetragonal phase stabilization of pure Zirconia.

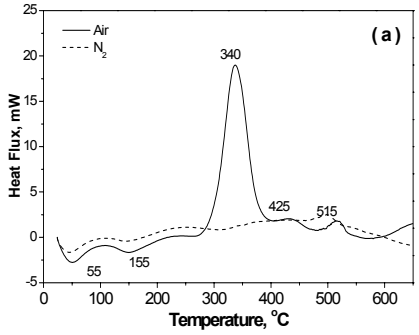


Fig.1: DSC and TG curves of pure Zirconia dried gel (110°C/24h)

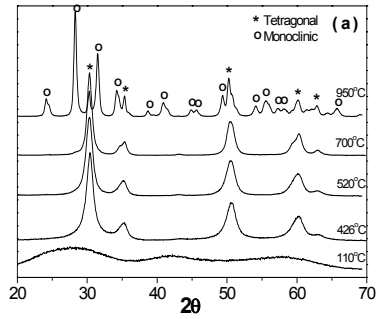


Fig.2: Diffractograms of (a) pure and (b) doped Zirconia

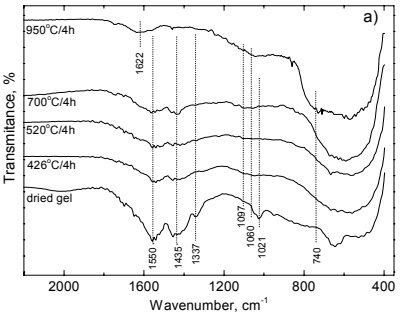
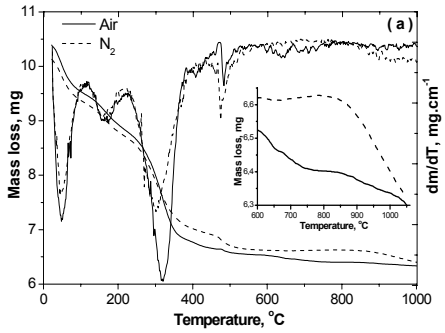
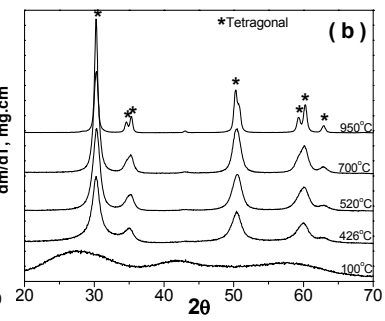
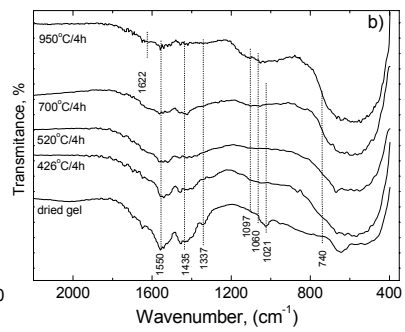


Fig.3: FT-IR of (a) pure and (b) doped Zirconia







Key words: Zirconia, sol-gel, Praseodymium, carbon elimination, phase stabilization