Crystal phase identification of silica-zirconia based materials obtained by sol-gel

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Silica and zirconia based materials have numerous applications, ranging from dielectric materials for reduced size transistors[1] to thermal barrier coatings for stream turbines[2]. The appropriate application of this type of material depends on its crystalline structure. In this work, SiO_2-ZrO_2 composites were obtained by the sol-gel process, using tetraethylorthosilicate (TEOS) as silica precursor and zirconium acetate solution as zirconia precursor. Samples were prepared with varying Si:Zr ratios (from 0.75 to 9), heat treated to 1000 and 1500 °C, and the crystalline structures were evaluated by X ray diffraction (XRD). XRD analysis of the samples heated at 1000 °C showed that those synthesized with smaller amounts of Zr presented amorphous silica as the preferential phase. For samples synthesized with equivalent amounts of zirconium and silicon, the resulting material showed the tetragonal zirconia (t-ZrO_2) as the main phase. This phase is not the stable one at ambient temperature and its identification indicates that silicon stabilizes this metastable phase. For zirconium-rich samples treated at 1000 °C, the main phase obtained was also the t-ZrO_2, but traces of crystalline silica and $SiZrO_4$ phases were also observed. For the samples treated at 1500 °C, the silicon-rich samples showed two polymorphic structures, a cristobalite and a β-cubic SiO_2 phases, which were determined by the Rietiveld method. Besides that, t-$ZrO_2$and traces of $SiZrO_4$ were also identified. For the samples containing equivalent amounts of zirconium and silicon, it was observed the intensification of the crystalline peaks related to $SiZrO_4$ compound. For the zirconium-rich samples, it was observed the intensification of the $SiZrO_4$ diffraction peaks and the presence of monoclinic zirconia, indicating that the amount of silicon was not sufficient to maintain the t-ZrO_2 phase stabilized. Summarizing, heating enhanced the crystallization of silica stable phases as expected; nonetheless the metastable tetragonal zirconia phase was found to be stabilized at room temperature by the presence of silicon. The characterization of the different phases formed allows the appropriate choice of application of these materials.

Keywords: zirconium silicate, sol-gel

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