Effect of Synthesis on the Phases in Scandia-Stabilized Zirconia (ScSZ)

Conghua Wang* and Wayne L. Worrell
Department of Materials Science and Engineering
University of Pennsylvania
3231 Walnut Street, Philadelphia, PA 19104

Introduction

Zirconia-based electrolytes have been extensively researched for their applications in sensors, dense oxygen-ion membranes and solid oxide fuel cells (SOFCs). Scandia-stabilized zirconia (ScSZ) has the one of the highest oxygen-ion conductivities in the cubic-flourite zirconia phase (1). However, it is very difficult to obtain a single-phase material because of the compositional inhomogeneity during the processing (2). In this paper the effect of synthesis methods on the phases in ScSZ has been investigated.

Synthesis Methods

ScSZ powders are synthesized using wet chemistry processes including Pechini, co-precipitation, and sol-gel methods. The pyrolytic decomposition behaviors of the as-dried powders are examined using Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) in air. As shown in Figure 1, most of the weight losses of co-precipitation and sol-gel powders occur below 300°C. The exothermic DTA peaks around 450°C (associated with a small and abrupt weight loss) indicate the beginning of ScSZ crystallization. The Pechini powder has an abrupt weight loss associated with a big and sharp exothermal peak in the DTA curve between 510 and the 535°C, which is due to the carbon combustion and oxides crystallization. After 550°C, the weight loss for all powders is negligible.

All ScSZ powders are calcined at 600°C for 10 hours in air for complete decompsition. Some powders are further heated between 800~1600°C for 10 hours, and cooled down to room temperature for XRD experiments.

Results

The phase structure and the lattice parameters are determined from the XRD spectra. As shown in Figure 2, all these heated Sc8SZ powders have broad diffraction peaks of fluorite phase, indicating small crystalline sizes and high amorphous content. The phase structure varies dramatically after these powders are further annealed at higher temperature.

The new diffraction peaks of the tetragonal phase for the Pechini and co-precipitate Sc8SZ powder appear after annealed at 1000°C and 1100°C respectively, and do not disappear in powders even after heated at 1600°C. The tetragonal-phase diffraction peaks of the sol-gel powder are seen only after heated at 1200°C and then disappear after annealed at higher temperatures.

The differences of the XRD spectra of these powders reflect the ionic-inhomogeneity of the ScSZ powders synthesized by different methods. The sol-gel is the best method for cation-mixing because of the small particle sizes resulting from the slow sol-gel transition. The co-precipitation method is not as good as sol-gel due to the bigger particles formed in the rapid precipitation process. The Pechini ScSZ powder has worst inhomogeneity, probably due to the selectivity of Sc^{+3} and Zr^{+4} on hydroxycaproxylic acid groups. Therefore, only the sol-gel Sc8SZ powder shows a single cubic-fluorite phase structure after heated at 1600°C. The lattice parameter of 6~10 mole% Sc_2O_3 doped ScSZ cubic-fluorite phase by the sol-gel process is between 5.085~5.087 Å.

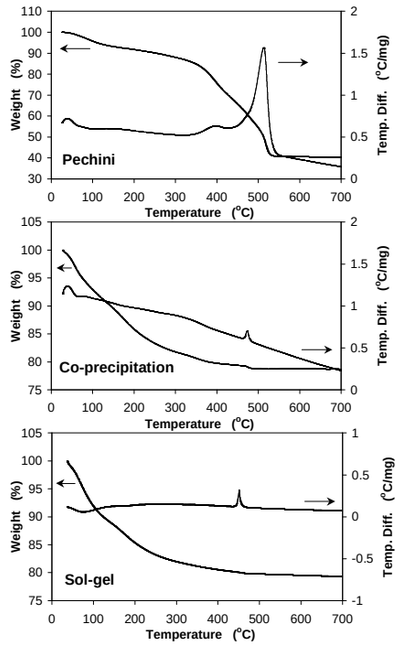

Figure 1. TGA and DTA curves of Sc8SZ powders prepared by different methods

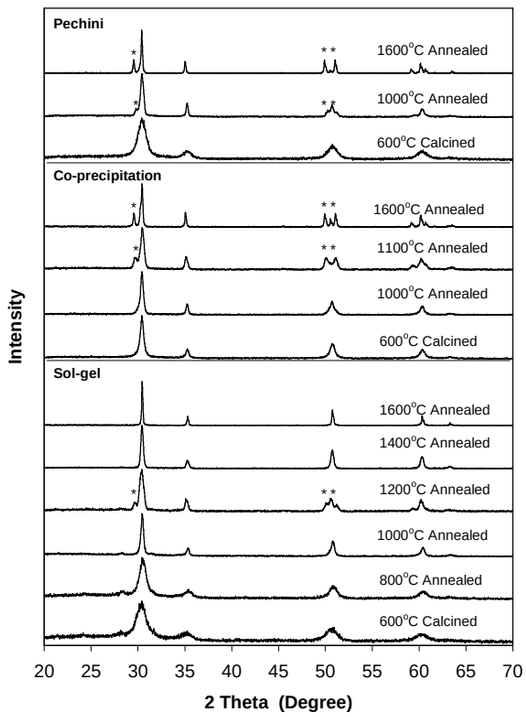

Figure 2. XRD spectra of Sc8SZ powder prepared by different methods
*** marks peaks of the tetragonal phase**

References:

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* Current address: Sarnoff Corporation, 201 Washington Rd., Princeton, NJ 08543