Scaled-Up Manufacturing of Nanostructured Refractory Ceramics for High-Temperature Applications

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The goal of this project was to study the synthesis of carbide-based nanostructured powders via a *unique and extremely promising* solvothermal reduction technique in order to produce materials that have a direct application in aerospace and electronic applications. Initial efforts have involved the synthesis of silicon carbide and tantalum carbide nanopowders through the solvothermal reduction process by using metal powders and carbon tetrachloride (CCl_4) as silicon and carbon sources, respectively, and metallic lithium as the reducing agent.

A typical reaction for the formation of silicon carbide was carried out with the following reactants: silicon powders (98+%, Aldrich), analytical grade CCl_4 (anhydrous, ≥99.5%, Aldrich), and metallic lithium (stabilized powder, 50-150 μm, Aldrich). The reactants were placed in a stainless steel iron tube and sealed under argon atmosphere. The iron tube was heated to 600°C for 8 hours. After synthesis, the contents inside the tube were washed with reagent grade methanol, acetone, and distilled water to remove lithium chloride, carbon, and other impurities. The efforts and success in cleaning the powders will be discussed. After cleaning, the powders were characterized by x-ray diffraction using CuKα radiation on a Philips 3100 diffractometer, dynamic light scattering on a Nanotrac Ultra instrument and scanning electron microscopy on a Hitachi S-4700 instrument. The x-ray diffraction pattern of the product powders was indexed as cubic SiC with a crystallite size between 15-20 nm. The dynamic light scattering analysis of the powders showed that the particle size distribution consisted of powders between 1-4 μm. This was also confirmed by scanning electron microscopy.

For the synthesis of tantalum carbide, tantalum metal or a salt of tantalum was mixed with a stoichiometric amount of carbon. In addition, a fuel and oxidizer was added upon which heating to an intermediate temperature ignited the powders to form tantalum carbide. The fuel to oxidizer ratio was varied to investigate and optimize the effects of reaction temperature and time.

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