Mechanochemical synthesis of HfB_2-HfO_2 ceramic powders from $HfCl_4$-B_2O_3-Mg system

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Transition metal-diborides are referred as ultra high-temperature ceramics and have remarkable properties including very high melting point, high hardness and Young's modulus, high thermal and electrical conductivity and excellent corrosion resistant to molten slags. These superior characteristics make HfB_2-based materials appealing candidates for some high technology applications including aerospace industry. Because of its excellent strength and thermal properties, it has found application areas in nuclear reactors as heat shield or control rod. Also, small amount of HfO_2 containing HfB_2-based materials has attractive properties for microelectronics and for wear resistant coatings.

Hafnium diboride-hafnium oxide (HfB_2-HfO_2) powders were fabricated via mechanochemical synthesis route. Hafnium tetrachloride ($HfCl_4$) powders, boron oxide (B_2O_3) and magnesium (Mg) powders were used as starting materials. The powder blends were prepared in a glove box and charged into the milling vial which was evacuated and filled with Ar several times. The starting powders were milled in a high energy ball mill, Spex 8000D Mixer/Mill, at 1200 rpm for different durations from 30 min to 5 h. Annealing in a tube furnace were applied to the selected milled powders at 1000°C for 3 h. A constant ball-to-powder weight ratio of 10:1 was used during milling experiments. Undesired products of mechanochemical reaction were removed by acid leaching under the effect of ultrasonic stirring and obtained products were washed repeatedly with distilled water. The effects of milling and annealing on the microstructure and properties of HfB_2-HfO_2 ceramic powders and process efficiency were examined. The milled, annealed and leached products were characterized by XRD, SEM, SM, DSC and particle analyzer.

Keywords: HfB_2-HfO_2 ceramic powders, mechanochemical synthesis, leaching