Abstract

Studies on synthesis, densification and oxidation of zirconium diboride based materials

ZrB$_2$ is leading materials in the category of ultra high temperature ceramics (UHTC) due to very high melting point (>3200$^\circ$C), high thermal conductivity, good thermal shock resistance, low coefficient of thermal expansion, retention of strength at elevated temperatures and stability in extreme environments. It is considered a suitable material for leading edge applications in hypersonic re-entry vehicles. Due to high neutron absorption cross section of boron, it is also a suitable material for neutron absorber in nuclear reactors. Despite having excellent properties, the actual uses of these ceramics are limited due to poor sinterability and low fracture toughness. Due to strong covalent bonding and low self-diffusion, simultaneous application of high temperature and external pressure is required to densify monolithic ZrB$_2$.

This thesis presents the results of investigation carried out on synthesis, densification and oxidation of ZrB$_2$. Effect of sinter additives on densification, mechanical properties and oxidation behaviour were also investigated. ZrB$_2$ have been synthesized by solid state reaction between ZrO$_2$, B$_4$C and carbon according to reaction (1) at high temperature in vacuum.

$$\text{ZrO}_2 + \frac{1}{2}\text{B}_4\text{C} + \frac{3}{2}\text{C} \rightarrow \text{ZrB}_2 + 2\text{CO}$$\(\text{(1)}\)

Synthesis experiments were carried out in a vacuum induction furnace. The products were characterized by XRD, SEM and chemical analysis. The effect of temperature and charge composition on product quality was studied. Pure phase ZrB$_2$ was synthesized at 1875 $^\circ$C in vacuum using a charge mixture containing excess B$_4$C. Investigation was carried out on densification of ZrB$_2$ by hot pressing. Monolithic ZrB$_2$ was densified to near theoretical density at 1850$^\circ$C and 35 MPa. Silicide additions lowered the densification temperature to 1650 $^\circ$C. EuB$_6$ addition decreased the hot pressing temperature to 1750 $^\circ$C. Addition of silicides and EuB$_6$ has been found to increase the fracture toughness and oxidation resistance of ZrB$_2$. 