Chapter 12
Fabrication, Microstructure, and Properties of Zirconium Diboride Matrix Ceramic

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ABSTRACT

The crystal structure, synthesis, and densification of zirconium diboride (ZrB₂) are summarized in detail. In this chapter, ZrB₂-ZrC-SiC ceramic was synthesized by reactive hot pressing a mixture of Zr, B₄C, and Si powders. The thermal shock resistance of the ZrB₂-SiC-ZrC ceramic was estimated by the water-quenching method and was significantly greater than that of a ZrB₂-15vol.% SiC ceramic. The isothermal oxidation of the ZrB₂-SiC-ZrC ceramic was carried out in static air at constant temperatures of 1000±15, 1200±15, and 1400±15 ºC for different amounts of time at each temperature. The mechanism of strength increase for the oxidized specimen indicated that the strength increased with the reaction rate, which was related to the rate of change in volume induced by reaction, initial crack geometry, elastic modulus, and surface free energy. The formation of oxide layers resulted in (I) repair of surface flaws, (II) increase in flexural strength, (III) appearance of a compressive stress zone beneath the surface oxide layers, (IV) decrease in thermal

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stress, and (V) consumption of thermal stress. These five aspects were favorable to the improvement of the thermal shock resistance of the ZrB$_2$-SiC-ZrC ceramic. The isothermal oxidation of the ZrB$_2$-SiC-ZrC ceramic was carried out in static air at 1600±15 ºC. In the different oxidation stages, quantitative models were proposed for predicting oxidation kinetics.

INTRODUCTION

Zirconium diboride (ZrB$_2$) is a member of a family of materials known as ultra-high-temperature ceramics (UHTCs). Several diborides, carbides and nitrides of the group IVB and VB transition metals are also considered UHTCs primarily because their melting temperatures exceed 3000 ºC. Very few elements or compounds from any class of ceramic materials have melting temperatures approaching 3000 ºC (Brochu et al., 2008). Applications that take advantage of these properties include refractory linings, electrodes, microelectronics and cutting tools. In addition to their high melting temperatures, zirconium diborides have a unique combination of chemical stability, high electrical and thermal conductivities, and resistance to erosion/corrosion (Talmy et al., 2008) that makes them suitable for the extreme chemical and thermal environments associated with hypersonic flight, atmospheric re-entry, and rocket propulsion. Hardness, bulk modulus, Debye temperature, coefficient of thermal expansion, thermal conductivity, and enthalpy of formation are some properties that are related to bond strength (cohesive energy) (Kuźma and Mikhalenko, 1985). Generally, the combination of bonds (Zr-Zr, B-B, and Zr-B) influences the properties of a material (Lundstroem, 1985). Zirconium borides have a wide range of compositions, with boron: metal (B:Zr) ratios ranging from 1:4 to 12:1. The B:Zr ratio affects both properties and electronic structure. Changing the B:Zr ratio changes the electronic structure of boron, which leads to the formation of different structural complexes containing one-, two-, and three-dimensional (3D) B networks. Increasing the number of B atoms in a structural complex leads to an increase in the B–B bond strength and an increase in the stiffness of the crystal lattice, along with increases in melting temperature ($T_m$), hardness ($H_v$), strength ($\sigma$), and chemical stability. The Zr–B bond strength is dependent on the degree of electron localization around the Zr atoms. The valence electron configuration in isolated B atoms is 2s$^2$2p. In ZrB$_2$, the outer electron configurations are sp$^2$ and sp$^3$, which promote strong covalent bonding. B atoms are electron acceptors, while Zr atoms are electron donors. Each Zr atom donates two electrons (one to each B), which convert Zr atoms to doubly charged cations and B atoms to singly charged anions. As shown in Figure 1, the crystal structure of ZrB$_2$ is primitive hexagonal (P6/mmm space group).
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