Abstract

A study has been carried out on processing and microstructure-property relations of ZrB_2 and HfB_2 based ultra-high temperature composites for applications in leading edges of hypersonic vehicles. The effect of SiC content, additives (B_4C and C), and process parameters on densification and structure–property relations have been studied for pressureless sintered ZrB_2–(10–40 vol. %) SiC particulate composites. The amount of densification is found to increase with sintering duration, and by prior holding at 1250 and 1600 °C for reduction of oxide impurities. Both SiC and WC appear to aid in reduction of oxide impurities. Elastic moduli, hardness and indentation fracture toughness increase with increasing SiC content. Presence of 20 vol. % SiC leads to the most optimum combination of mechanical and thermal properties among the pressureless sintered composites.

Composites having compositions of ZrB_2-20 vol.% SiC (ZS), ZrB_2-20 vol.% SiC-5 vol.% Si_3N_4 (ZSS), ZrB_2-20 vol.% ZrC-20 vol.% SiC-5 vol.% Si_3N_4 (ZZSS) and HrB_2-20 vol.% SiC (HS) have also been processed by hot pressing at 2000 °C. These composites have been found to possess >99% of the theoretical density. The elastic moduli, Poisson's ratio, hardness, flexural strength, and fracture toughness have been measured. Among the hot pressed composites, the ZSS composite has shown the most optimum combination of room temperature mechanical properties. Whereas thermal conductivity of ZrB_2 or HfB_2 is increased on addition of SiC, coefficient of thermal expansion (CTE) is reduced on reinforcing with SiC, Si_3N_4 or ZrC.

The non-isothermal, isothermal and cyclic oxidation behaviors of the hot pressed composites have been studied. The oxide scales have been examined using X-ray diffraction, scanning electron microscopy accompanied by energy dispersive spectroscopy facility as well as electron probe microanalyzer with wavelength dispersive spectroscopy. These studies have shown that the oxide scales have an outer layer of borosilicate (B_2O_3 + SiO_2) and inner layer comprising ZrO_2 or HfO_2 in case of ZS or HS, respectively. It has been observed that oxidation resistance of HS is superior to that of ZS on isothermal exposure at 1200 °C and 1300 °C. However, the trend gets reversed on cyclic oxidation, because of greater coefficient of thermal expansion mismatch between oxide scale and the substrate is greater in HS than that in ZS. Addition of Si_3N_4 is found to increase oxidation resistance by enhancing kinetics of borosilicate scale formation.

Compressive creep tests have been performed for ZS and ZSS at constant load in air at different combination of stress (93−140 MPa), temperature (1300−1425 °C) and time duration (20−40 h). The stress exponents of ZS and ZSS have been found to decrease from 1.7 to 1.1 and from 1.6 to 0.6, respectively with increase in temperature. Furthermore, the activation energies have been found to be ≈95±32 kJ/mol at temperatures ≤ 1350 °C, and as ≈470±20 kJ/mol, at higher temperatures. These results along with study of post–creep microstructures suggest that grain boundary sliding is the operating mechanism and it is accompanied by O^{2-} diffusion through intergranular glassy film at temperatures ≤ 1350 °C, whereas it occurs by viscoplastic flow of at higher temperatures. Addition of Si_3N_4 is found to enhance mechanical properties at ambient temperature besides oxidation resistance, but it lowers the creep resistance at elevated temperatures. This study has shown that addition of 20 vol. % SiC leads to the best combination of desirable mechanical, thermal and oxidation properties. However, damage resistance during ablation at 2200 °C is enhanced by ZrC addition. The mechanisms of thermal transport, creep, oxidation and damage during ablation have been established through this study.

In Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications, Fahrenholtz, W.G., Wuchina, E., Lee, W.E., and Zhou, Y. eds.; Wiley-Blackwell: New York, 2015; pp. 33â€“59.Google Scholar. 55.Â Gallagher, M.K., Rhine, W.E., and Bowen, H.K.: Low-temperature route to high-purity titanium, zirconium and hafnium diboride powders and films. In Ultrastructure Processing of Advanced Ceramics, Mackenzie, J.D. and Ulrich, D.R. eds.; Wiley Interscience: New York, 1988; pp. 901â€“906.Google Scholar. Zr82 based UHTCs with appropriate compositions are attractive materials for high temperature engineering applications because of their combination of high melting point, high strength and hardness, high thermal and electrical conductivity, thermal shock resistance, and chemical stability. 1- 4 Despite these attractive properties and proposed applications, difficulties in densification as well as high processing costs of ZrB2 based UHTC components remain challenges.Â Ofthe transition metal diborides, HfB2 and Zr82 were identified as the most promising candidates for high temperature applications such as nose caps, sharp leading edges, vanes and similar objects for use in high velocity flight or on future generations of reentry vehicles.4. B. Ultrahigh Temperature Ceramic Composites: Studies on zirconium and hafnium diboride based ultrahigh temperature ceramic (UHTC) composites with SiC, ZrC and Si3N4 reinforcements have been carried out with financial support from DRDO aiming at developing materials for extreme environments faced by nose-cones and leading edges of hypersonic vehicles during re-entry into earthâ€™s atmosphere.Â D. Interfaces in Aluminium Matrix Composites: It has been shown that in-situ Al-TiC or Al-TiB2 composites have particle-matrix interfaces exhibiting misfit-strain localization, and excellent bond integrity.The primary contribution in this area is evaluation of the effect of particle-matrix interfaces on mechanical behavior.