Cyclic oxidation behaviour of ZrB2-20 vol.%MoSi2 based ultra-high temperature ceramic matrix composite between 1100°C and 1300°C

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Cyclic oxidation behaviour of ZrB_2 -20 vol.%$MoSi_2$ based ultra-high temperature ceramic matrix composite between 1100°C and 1300°C

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Abstract- While descending through different layers of atmosphere with tremendously high velocities, hypersonic re-entry nosecones fabricated using ultra-high temperature ceramic matrix composites (UHTCMCs) are subjected to repeated thermal shocks. This necessitates extensive investigations on the cyclic oxidation behaviour of UHTCMCs at temperatures ranging from 1100°C to 1300°C (service temperature of the nosecones). To this end, the present work is aimed at investigating the cyclic oxidation behaviour of ZrB2 -20 vol.%MoSi2 (ZM20) UHTCMC (a very widely investigated ZM CMC) by carrying out cycles for 6h, at 1cycle/h and estimating oxidation kinetic law. This has been followed by extensive characterisation using X-Ray Diffraction (XRD) to indicate the phases formed during oxidation and Scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), in order to determine the chemical composition of the oxides formed between 1100°C and 1300°C.

Keywords- Borides; ceramic composites; cyclic oxidation; kinetics; oxide layer

Among UHTCs, ZrB2-based ceramics have been reported to be potential candidates for the manufacture of reusable Thermal Protection Systems (TPS) in Hypersonic re-entry nosecones, due to very high thermal conductivity and relatively low density [1,2]. However, the low fracture toughness and poor thermal shock resistance of these ceramics pose major obstacles to their use in extreme environment [3]. Moreover, the poor oxidation resistance of ZrB2 at temperatures above 1200°C, due to formation of B2O3, and a non-protective porous scale of ZrO2 [4], poses restrictions to its use at elevated temperatures, especially above 1200°C. Thus, it becomes extremely important to find materials, which may highly enhance the oxidation resistance of ZrB2 [5-8]. A significant amount of work has already been done in that direction [8-12]. Besides, a significant amount of research has been done on reinforcing diborides like ZrB2, HfB2 and TiB2 with SiC, MoSi2, or ZrSi2 for enhanced oxidation resistance beyond 800°C [3-20]. However, a limited amount of study has been made on cyclic oxidation of ZM20 at temperatures exceeding 1100°C, which is not at all unlikely, in the context of Hypersonic nosecones, during a high velocity descent through different layers of atmosphere. Thus, the scope of the present study is to investigate the cyclic oxidation behaviour of ZM20 between 1100 and 1300 °C.

The important conclusions drawn from the results and discussions of this study have been elucidated. Cyclic oxidation behaviour of ZrB2-20 vol.% MoSi2 composite have been studied at 1100 °C, 1200 °C, 1250 °C and 1300 °C for 6hrs. Monitoring weight change and examining oxide scales draw following conclusions:

(i) Weight gain for both the composites increased with increasing temperature and time.

(ii) Weight gain occurred due to formation of ZrO2 and SiO2, at elevated temperatures.

(iii) The main oxidation products were ZrO2, MoO3 and SiO2.

(iv) At 1200 °C and above, the presence of SiC particles markedly improves the resistance to oxidation of the composite due to the formation of borosilicate glass.

(v) Due to formation of oxide layer on the surface, the hardness of the samples i.e. its mechanical properties decreased from center to surface.

(vi) The cyclic oxidation of the samples follow linear oxidation kinetics from 1100 to 1250 ºC while at 1300 ºC it follows parabolic oxidation kinetics due to the protective action of SiO2 above 1250 ºC.

The results of the present study and their analyses lead to the following directions for future work:

(i) The oxidation kinetics of the samples beyond 1300 ºC can be studied.
(ii) Residual strain calculations can be carried out.
(iii) Mathematical modelling study of the oxidation kinetics can be carried out.
(iv) TEM study of the samples can be carried out for more precarious measurements.
(v) Carrying out diffusion studies on oxide layer.

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