HIGH TEMPERATURE CREEP DEFORMATION AND OXIDATION OF HOT ISOSTATICALLY PRESSED (HIPed) SILICON NITRIDE

THESIS SUBMITTED BY

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ABSTRACT

The creep and the oxidation behaviour of hot isostatically pressed silicon nitride with 4 wt% yttria have been studied. For comparison, creep behaviour of hot isostatically pressed silicon nitride with 3.5 wt% yttria and 7.5 wt% yttria + 2.5 wt% silica has also been investigated.

Creep tests have been carried out in four-point bending mode and creep mechanisms were interpreted via the stress exponent (n) and the activation energy (Q) in the general creep equation, $\varepsilon = A \sigma^n \exp(-Q/RT)$. To characterize the materials and to correlate the deformation mechanisms with the microstructure, X-Ray Diffractometry (XRD), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) studies were performed on the as-received as well as the deformed samples. Oxidation experiments have been carried out on the 4 wt% yttria material at 1300 and 1400 °C and the oxidized samples have been analyzed using XRD, SEM and TEM.

The typical microstructure of 4 wt% yttria material consists of a major crystalline phase (α- and β-Si_3N_4), secondary crystalline phase (α-$Y_2Si_2O_7$) and a thin intergranular amorphous phase. Microstructural studies on deformed specimens revealed that the creep processes occurring in the 4 wt% material were similar up to 1300 °C in the compressive side and the tensile side of the deformed material under the bending configuration. However, at 1350 °C and above there was a significant difference in microstructure between the compressive side and tensile side of this material. TEM studies showed that the shear stresses acting on the Si_3N_4 grains promote the formation of multi-grain junction cavities whereas the tensile stresses acting perpendicular

to the grain boundaries promote the formation of lenticular cavities between Si_3N_4 - Si_3N_4 grain boundaries. No such cavities were observed between the $Y_2Si_2O_7$ - Si_3N_4 grain boundaries probably due to the softening of $Y_2Si_2O_7$ phase at high temperature. In the 7.5 wt% Y_2O_3 material, the amount of intergranular phase is high and therefore possibility of accommodating plastic deformation by this phase could be the reason for non-cavitating behaviour observed in this material.

Further TEM studies of 4 wt% Y_2O_3 material showed presence of a $Y_2Si_2O_7$ network in the compressive side of the material. However, there was no such network of $Y_2Si_2O_7$ in the tensile side of the material or in the as-received material. Formation of a $Y_2Si_2O_7$ network only in the compressive side is probably due to the redistribution of $Y_2Si_2O_7$ under the compressive stress.

The morphology of the oxide scale of the heat treated samples of the 4 wt% Y_2O_3 material was found to be dependent on the temperature, nature of the stress and magnitude of the stress. The compressive stresses promote the formation of elongated and more developed Y-rich particles on the surface of the oxide scale. This significant difference observed in the surface oxide scale was related to the observed yttrium rich network in this side of the material.