Evolving microstructures during creep cavitation

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Introduction

Creep is one of the principle factors limiting the high-temperature and high-stress application of components in advanced gas turbines. Previous ultrasmall-angle x-ray scattering (USAXS) studies [1] have been able to follow the development of creep cavities and have shown how they develop at multigrain junctions during deformation. While secondary phases have been used to limit cavity formation (and thus to improve the mechanical performance of ceramics), the evolution of the secondary phases has not been studied due to the lack of a suitable technique. Anomalous-USAXS (A-USAXS) has been used in the present study to measure the size evolution of both the cavities and the secondary phases. A recent cavitation model [2] suggests that the cavities grow via redistribution of the crystalline secondary phases between different junctions. Thus, knowledge of the size evolution of the crystalline secondary phase particles and the concurrent development of creep cavities during deformation is crucial for understanding the mechanisms controlling cavitation and the ultimate development of improved ceramics.

Methods and Materials

A commercial grade of gas-pressure-sintered silicon nitride (designated SN88) [3, 4] was used. It consists of β-silicon nitride grains, voids, and secondary phases containing Yb and a small amount of Y. The major crystalline secondary phase after heat treatment is ytterbium disilicate. Minor phases include residual oxynitrides, silica glass, and porosity.

Five tensile creep tests were carried out at 1400ºC under a 150 MPa load. The details of the testing procedure are described elsewhere [1]. After different periods of time and strain, the tests were interrupted and the specimens were cooled under load. Two samples for A-USAXS were cut from each specimen, one from the undeformed grip area and one from the gage area parallel to the stress axis. The samples were ground and hand polished to thicknesses of approximately 180 μm and 100 μm, respectively.

The 33-ID monochromator was calibrated to 0.5 eV by means of an x-ray absorption spectrum about the Yb L-III edge. The UNI-CAT USAXS instrument [5] in 33-ID-D was used to measure USAXS from the grip and the gage of each specimen at four energies (-250 eV, -100 eV, -40 eV, and -10 eV from the edge). A blank, with no sample in place, was measured at each energy as well. The USAXS data were scaled to units of absolute cross section per unit volume per steradian by a primary method [6] and desmeared by an iterative method [7].

A-USAXS is an element-specific contrast-variation method to vary the scattering contribution from one of the populations while holding that from the other populations fixed. Processing of the data was carried out piecewise using a method described elsewhere [8].

Results

The scattering intensity from all of the samples showed a strong dependence on photon energy, consistent with the change in scattering contrast of Yb disilicate. Figure 1 shows the size distributions that were derived for the Yb disilicate and the voids for a sample that was strained for 50 hours. While the size distributions appear to have a log-normal shape, no such assumption was imposed by the analysis. The peak diameters of the two distributions are comparable, but the quantity of disilicate is much greater than the creep cavities.

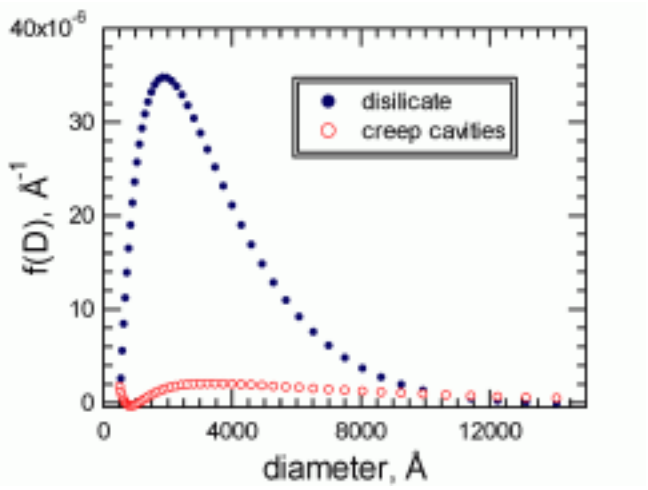

Figure 1: Size distributions of Yb disilicate and voids in a sample tested for 50 hours. While the size distributions are similar, the volume fraction of disilicate particles is much greater than the volume fraction of the voids.

Discussion

The A-USAXS data were analyzed to determine the size distributions of Yb disilicate and creep cavities as a function of tensile creep in SN88 silicon nitride. While the distributions were comparable in size, the volume fraction of the Yb disilicate was five to eight times greater than that of the creep cavities. It was found that the volume fraction of creep cavities increases linearly with tensile strain and the proportionality factor is one. This linear relationship agrees well with earlier data [1] and confirms that cavitation is the main creep mechanism.

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