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To cite this version:

A. Robertson, D. Wilkinson. HIGH TEMPERATURE DAMAGE ACCUMULATION IN HOT PRESSED ALUMINA. Journal de Physique Colloques, 1986, 47 (C1), pp.C1-661-C1-666. <10.1051/jphyscol:19861101>. <jpa-00225496>

HAL Id: jpa-00225496
https://hal.archives-ouvertes.fr/jpa-00225496
Submitted on 1 Jan 1986

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HIGH TEMPERATURE DAMAGE ACCUMULATION IN HOT PRESSED ALUMINA

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Abstract - The development of grain-boundary cavitation in hot-pressed Al₂O₃ both during flexural creep and stress-free annealing in air has been studied. Creep damage accumulation in driven both by the applied stress, and by internal gas pressure in the cavities. The internal pressure results from gas trapped during hot pressing, and from gas-generating chemical reactions active during testing. At machined surfaces, a damage-resistant skin about 15-20 µm thick overlies a heavily cavitated region.

INTRODUCTION

Hot pressed alumina (HPA) is an attractive model material for basic research into creep fracture. The work reported here represents the first stages in applying a damage accumulation approach to this topic. The long term objective is a fundamental understanding of crack nucleation and propagation in the creep fracture regime, applicable to predicting reliability in structural ceramic components in high temperature service. The immediate aims are to evaluate HPA as a model material, and to establish geometries and conditions for future tests. In this work, the levels of internal grain boundary cavitation (area fraction and number density) were determined for HPA both after creep (at 1350 °C) and after stress-free annealing in air (1250, 1350 and 1470 °C). The profiles were generated by image analysis of SEM micrographs taken in step scans across internal sections of test specimens.

EXPERIMENTAL

All specimens were taken from a single billet of hot-pressed Al₂O₃ obtained from AVCO, Systems Division. Bend specimens 5×12.5×65 mm were machined from the billet. The procedure used is outlined elsewhere /1/. Two parallel straight lines, 10 mm apart, were diamond-scribed across the centre of one 12.5 x 60 mm side of each creep bar. From measurements of the separation of these lines,
before and after a creep test, fiber strains were calculated /2/. Bars were crept in air, at 1350 °C, in a four-point bend rig /1/. Curvature at high fiber strains was minimized by creeping bars "on edge". The loading direction was parallel to the original direction of hot-pressing. Control specimens were placed on the lower block of the bend rig. Creep strain was monitored by a 3-probe extensometer. Stresses and strains were calculated after Hollenberg et al. /3/; applied stresses reported here are maximum, steady state values. After creep, samples were sectioned parallel to the loading direction. The cut faces were diamond polished, thermally etched (1600 °C, air, 3½ min.), and coated with gold. Step scans of 12 SEM micrographs each were taken across the polished, etched faces. On each micrograph, the average area fraction and number density of grain boundary cavities was measured.

Fig. 1: Creep damage profiles, with swelling effects removed, for two applied stresses: (a) and (c), area fraction; (b) and (d) number density.

RESULTS

Creep Damage

Damage measurements on crept samples shows that there is a definite creep component to grain boundary cavitation: the damage on any fibre increases with strain. However, the average damage at the neutral axis is also non-zero, and increases with time: the material is swelling. Near the specimen faces, damage
Fig. 2: Swelling cavitation in HPA during air anneals: a) area fraction vs. time, b) number density (voids·cm⁻²) vs time, c) average grain size (μm) vs time, d) number density (voids·gain⁻¹) vs time.

Fig. 3: The swelling rates of average individual pores vs effective pressure, for the p₀fₐ° in Table 2, and n=1.
profiles are perturbed. A damage-resistant 'skin' about 15-20 μm thick overlies a region in which cavitation is heavier than would be expected by extrapolating from the interior of the bar.

The creep component of damage, with swelling effects removed, is shown in Fig. 1. These figures were generated from raw creep damage data by converting position into strain /1/ and by subtracting the average damage level at the neutral axis. The resulting net damage plots for a single stress were superimposed. This approach assumes that the damage level at the neutral axis in a crept bar can be taken to be representative of the swelling component of damage across the bar.

The figures show that near-surface damage consists of a cavitation-resistant 'skin' overlying a heavily-cavitated layer. The average damage level for the combined 'skin and spike' is close to that extrapolated from internal damage. On the area fraction plots (Figure 1a,c) the rate of increase of the 'spike' damage with strain is similar to that for internal damage.

Swelling Damage

Data and typical microstructures for swelling in the absence of applied stress at 1250 °C, 1350 °C and 1470 °C are shown in Fig. 2. The area fraction of cavities (f_A, Fig. 2a) increases with time at all three temperatures. However, this is not the case for the number densities (Fig. 2b). In this figure, N_A initially increases rapidly at all temperatures. This may reflect growth into the detectable size range of pre-existing voids. At 1470 °C however, N_A peaks near 30 hours, after which it decreases rapidly. Fig. 2c shows the variation of grain size, L_g, with time. The dependence of this parameter on time and temperature is similar to that for f_A. At temperatures of 1250 °C and below, grain size is effectively constant. Fig. 2d shows the variation in number density of cavities, expressed as voids per grain. This plot was generated from N_A and L_g data by converting the average 2-dimensional grain size into the area of a regular hexagon. The data suggest that N_A decreases at 1470 °C, at times longer than 30 h, because rapid grain growth causes cavities to coalesce.

DISCUSSION

Creep Damage

The area fraction of grain boundary cavities in the interior of crept bars is strain-controlled at both stress levels. This suggests that the growth of larger cavities is controlled by grain boundary sliding. However, the number density (a parameter dominated by the smaller cavities) is strain-controlled only in the 40 MPa tests. This is not the case in the 12 MPa tests. This indicates that the mechanism controlling the growth of small cavities changes with stress (possibly to diffusion control at low stress). The limited amount of data currently available precludes any firm conclusions.

Near-surface damage profiles suggest that the surface damage gradients result from transient load shedding which occurs early in a creep test, but which is not active subsequently. Work is proceeding to determine whether chemical or mechanical processes are dominant.

Two types of chemical effects may be active. Boundary segregation of solutes, modulated by near-surface concentration gradients over distances of tens of microns, could affect grain boundary cohesion, and so, cavity nucleation. Alternatively, oxygen attack down grain boundaries could generate high-pressure CO or CO_2 gas within cavities /4-6/. Near the bar surface, however, the gas may be able to escape, and the carbon would become depleted relatively rapidly.
Machining effects provide another possible cause for the near-surface damage profiles. In no cases were cracks, suggestive of direct machining damage /7/, seen in regions near tensile faces. However, the machining schedule used would be expected to create a 15-20 μm thick surface layer, in which residual compressive stresses on the order of 100-200 MPa were present /7,8/. Such a layer would overlie a region subject to a compensating tensile stress field. Also, the surface layer would be charged with a high density of potentially mobile dislocations and active sources /9/. Until residual fields and mobile dislocation densities were annealed out during either a heat treatment or a creep test, or were swept away early in a test by creep deformation, the overall fiber stress gradients and matrix 'softness' (that is, its ability to deform without cavitating) would change sharply near machined surfaces.

Swelling

The swelling damage data has been tested against the hypothesis that the driving force for swelling is generated solely by gas trapped within the compact by pore closure /10/, during hot pressing in air. Following pore closure, the mass of gas in pores is assumed to be constant. The hypothesis implies that the residual cavities in the as-received billet are charged with high-pressure gas. The cavities are assumed to be spherical and closed, and the trapped gas to behave ideally. An analysis of the model using the current swelling data /1/ leads to several conclusions. First, we conclude that the amount of gas trapped in the pores is much less than one would expect. It therefore appears that much of the trapped gas is dissolved into the Al2O3 during hot pressing. When this effect is included in the model, the swelling at 1250°C shows a linear dependence of cavity growth rate v and the effective pore pressure, Peff (Fig. 3). However, at 1350 and 1470°C, the swelling rates do not decrease linearly with the calculated Peff. At these temperatures, gas-generating reactions capable of sustaining pore pressures somewhat smaller than those initially present due to trapping, apparently occur within the HPA. As a cavity grows, and the pressure component due to trapped gas falls, and chemically-generated gas pressures progressively become a larger fraction of the total driving force.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the contributions of G. Sutherland, who performed the swelling tests, and the assistance of Norton Research (Canada), Niagara Falls, Ontario, in the use of their Joyce-Loebl image analyzer. This work was supported by funding from the Natural Science and Engineering Research Council, Ottawa, Canada.

CONCLUSIONS

Hot-pressed alumina, containing dissolved carbon and machined with 35 μm diamond, accumulates low levels of cavitation damage internally when crept in air at 1350°C. The damage has 3 superimposed components: one related to creep, one due to swelling, and one occurring near machined faces. Damage accumulation cannot be characterized from surface measurements alone. Up to total damage levels of about F4 = 0.004, the creep component of flexural damage is strain-controlled at applied steady state stresses of 12 and 40 MPa. However, while the number density of cavities is strain-controlled at 40 MPa, this is not the case at 12 MPa. Swelling in air is driven both by the pressure of gas trapped within the compact during hot-pressing, and by internal gas-generating reactions. The reactions are significant at 1350 and 1470°C, but are negligible at 1250°C. Swelling can be used to predamage HPA uniformly before creep testing.
REFERENCES

/10/ A.G. Robertson and D.S. Wilkinson, to be published.