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Dynamic fatigue of a hot glass-ceramic

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The structures of crystalline silicates are characteristically of low symmetry and large lattice parameters, neither of which feature is conducive to easy plastic deformation by dislocation mechanisms. Non-crystalline silicates, on the other hand, are usually Newtonian fluids at high temperatures and it is flow of the non-crystalline material which is believed to be responsible for the plasticity of hot glass-ceramics. The high temperature dynamic fatigue properties of a Li_2O—ZnO—SiO_2 glass-ceramic have been investigated in order to test ideas suggested by tests previously carried out on the same material at constant stresses and at constant strain-rates.

1. Introduction

One characteristic which distinguishes the plasticity of hot glass-ceramics from the plasticity of other materials is that the macroscopic yield strength is considerably greater in compression than in tension [1, 2]. For most materials, metals for example, the yield strength is the same for both signs of applied stress and the observation that this is not so for hot glass-ceramics suggests that it may be possible to observe dimensional instability during cyclic loading between tensile and compressive stresses of equal magnitude.

At high temperature, it is expected that the response to an applied stress will be that of a solid-liquid mixture; the crystalline material will be rigid compared with the non-crystalline material which, since it must have a finite temperature coefficient of viscosity, will be fluid. Cyclic deformation involving flow of the fluid phase through pores in the crystalline material is likely to exhibit hysteresis which, due to the low thermal conductivities of silicate materials, is expected to generate a high stored energy.

2. Experimental

Lithium disilicate glass-ceramic test pieces were manufactured in our own laboratories; the glass making, casting, heat-treatment and shaping operations were similar to those previously reported [1]. The volume fraction of crystalline material was approximately 80% and the average lithium disilicate grain size was 10 μm. Rotating-bending and push-pull tests have been performed in the temperature range 20 to 900°C and at stresses up to about half the maximum stress measured in constant compression rate tests [2]. Above ~400°C there is mechanical hysteresis and it leads to a discontinuous evolution of heat which makes accurate control of temperature impossible.

3. Results and discussion

3.1. Preliminary tests

Fig. 1 demonstrates the general temperature dependence of fatigue life. At high and low temperatures, the number of cycles required for fracture at a given stress amplitude appears to decrease with increasing temperature whereas, at intermediate temperatures, the opposite trend is observed. The behaviour near room temperature is that expected for a ceramic exhibiting static fatigue; the fatigue life is insensitive to frequency, because total time at stress is the important parameter, but is rapidly decreased by raising the temperature since this accelerates the rate of corrosion. Eventually, however, a temperature is reached for which the chemical species responsible for corrosion is driven off and the resistance to failure begins to increase with increasing temperature. The temperature for the peak strength in Fig. 1 corresponds to the onset of plasticity and the subsequent behaviour is strongly sensitive to both frequency and temperature in the manner expected for a viscoelastic material. Since plastic flow occurs at the smallest stresses [2] there is no fatigue limit and the higher the frequency the fewer the cycles to failure. Data for rotating-bending tests at 750°C are shown in Fig. 2. The fatigue cracks

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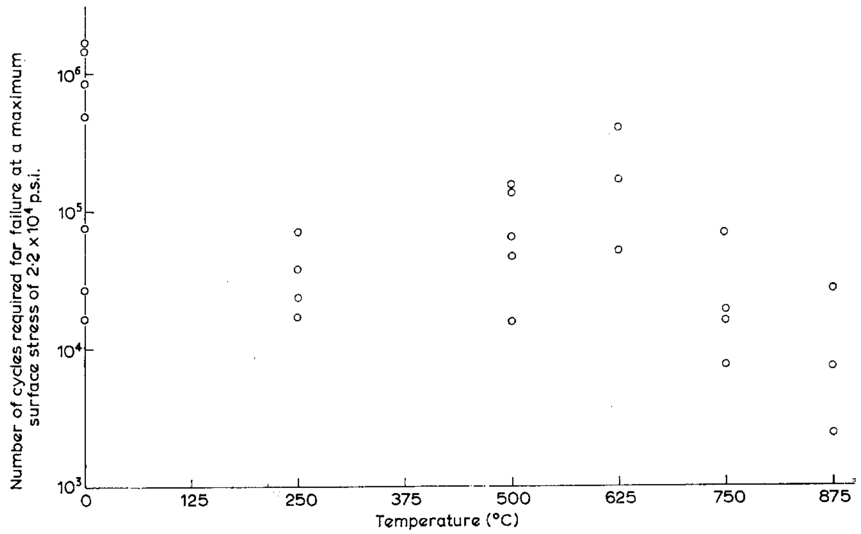

Figure 1 Variation of fatigue life with temperature. Rotating-bending tests at 200 cycles per minute.

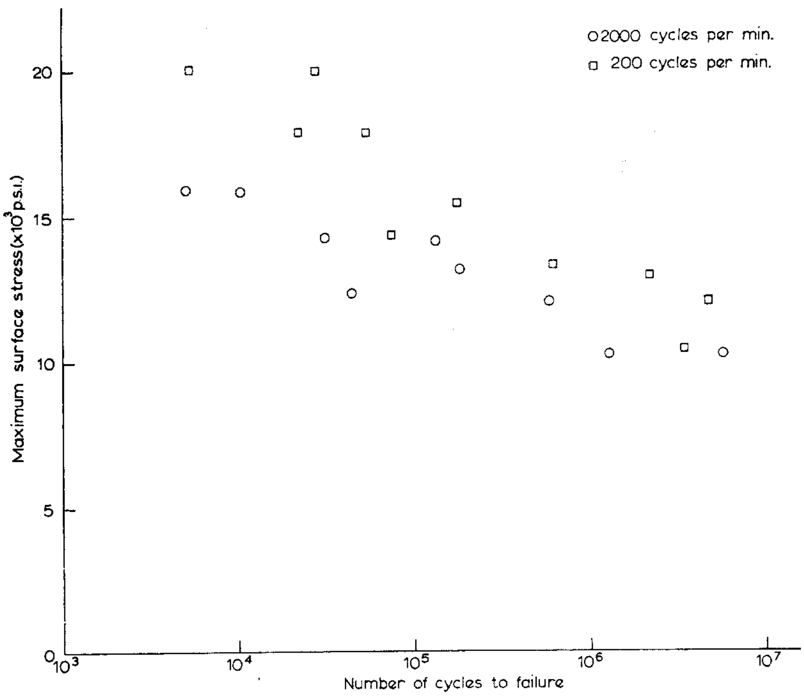

Figure 2 Variation of fatigue strength with frequency. Rotating-bending tests at 750°C.

in these tests tend to propagate on parabaloidal surfaces symmetrical with the specimen axis and have textures very similar to those reported for high temperature tensile tests [2].

The dimensional instability mentioned in the introduction is best illustrated with push-pull tests. In high temperature tests carried out at 2000 cycles per minute using a Haigh machine, necking was observed in lithium disilicate glass-ceramics at low stress amplitudes and barrelling at high stress amplitudes. In extreme cases, the specimen elongation that accompanies necking tripped the cut-out device and it was necessary to re-set the machine in order to continue the test. To a first approximation, the specimen does not contribute to the resonance of a Haigh machine, so if the current supplied to the electromagnets is constant, the force applied to the specimen is constant. Consequently, an increase in true stress amplitude is associated with necking and a decrease with barrelling, from which it is inferred that the fatigued region supports enhanced stress at low stress amplitudes and less stress at high stress amplitudes. Since hardening during plastic deformation is observed for all other mechanical tests on lithium disilicate glass-ceramics [1-4], hardening during cyclic loading might be expected. The unexpected weakening observed at high stress amplitudes is attributed to thixotropy.

3.2. Thixotropy

Owing to shear alignment or to changes in shape or distribution of suspended particles or to uncoiling of molecules, many different kinds of multi-phase materials, from oil-based paints to polymeric materials, become more fluid during mixing. This general phenomenon is known as thixotropy and if it occurs in hot glass-ceramics, it can be expected to manifest itself during dynamic fatigue. Whereas a dilatant solid-liquid system shows Newtonian flow at very low rates of strain, on increasing the shear rate, a departure from this behaviour occurs, the material requiring a disproportionate amount of stress to maintain a certain shear rate. This is attributed to the inability of the fluid to continue to percolate at the rate required by the macroscopic shear, dilatancy hardening being produced [5]. An opposite effect is observed in other solid-liquid systems and is called pseudoplasticity or shear rate thinning. Such thixotropic systems possess an effective yield stress and show properties similar to those of a Bingham plastic solid, that is, little or no flow up to a certain shear stress and relatively easy flow at higher stresses (c.f. non-drip paints). The flow is characterized by a coefficient of dynamic viscosity,

$$\eta' = \frac{\dot{\gamma}}{\tau - \tau_y}$$

where $\dot{\gamma}$ is the imposed rate of shear, τ is the applied shear stress and τ_y is the yield stress.

3.3. Microstructural observations

Fig. 3 shows push-pull data obtained in a stress range which, at 750 and 800° C, overlaps the necking and barrelling regions. Specimens examined before ultimate failure were found to contain surface cracks oriented perpendicular to the tensile axis but no extrusions, and sections

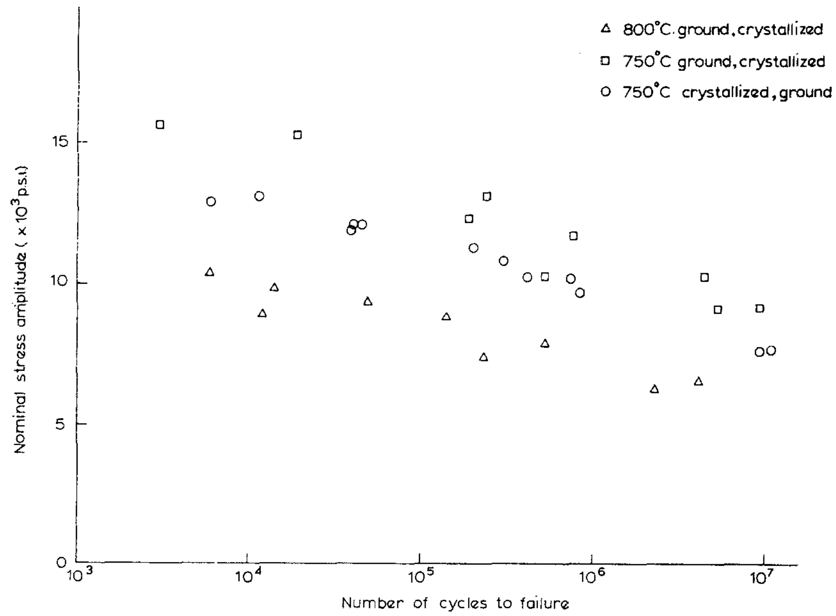

Figure 3 Push-pull fatigue tests at 2000 cycles per minute. The data measured at 750°C demonstrates the enhanced strength produced by grinding the gauge length before devitrification.

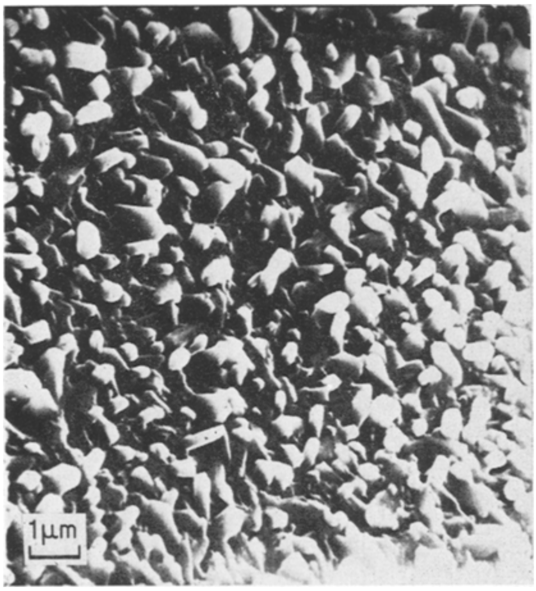

Figure 4 Lithium disilicate crystals standing proud of a fatigue fracture surface. Scanning electron micrograph.

revealed the presence of internal cavitation very similar to that reported earlier for creep tests [1].

It was concluded in [4] that large scale plastic deformation of hot glass-ceramics is the result of stress induced dissolution of crystalline material. The process is not thermodynamically reversible, the dual process of crystal dissolution and crystal growth requires work to be done and under normal circumstances some of this is dissipated as heat and gives rise to hysteresis. During cyclic loading, particularly at high stress levels, the quantity of material migrating may well be so large that the simultaneous movement of large numbers of atoms into and out of the non-crystalline material are locally unbalanced. In physical terms, regions of negative hydrostatic pressure are created within the fluid non-crystalline material and, when the pressure is sufficiently large to overcome the forces of surface tension and viscosity, cavities develop. By linking together, the cavities will eventually constitute cracks. The fatigue fracture surfaces of some specimens are partially covered with small lithium disilicate crystals such as might be either exposed by drainage of fluid or, alternatively, deposited during cavitation. Examples are shown in Fig. 4. Most of the fatigue fracture surfaces, however, are similar in appearance to the tensile fracture surfaces described previously [1, 2].

Finally, it should be noted from Figs. 2 and 3 that, like that for other materials, the fatigue strength of glass-ceramics depends on surface condition and on the type of loading. The strength for axial loading can be as low as 80% of that measured in rotating bending.

4. Conclusions

1. At temperatures high enough for permanent deformation, flow occurs at the smallest stress levels, as a consequence of which no fatigue limit is observed.

2. Owing to the fact that the compressive yield strength is almost an order of magnitude greater than the tensile yield strength, the material is dimensionally unstable during cyclic loading at high temperature.

3. The high temperature thermal conductivity is too small to avoid the build-up of stored energy during high frequency cyclic loading and, as a result, there is a significant rise in temperature.

4. During high temperature fatigue testing, the creation of negative hydrostatic pressure within the fluid non-crystalline material leads to the nucleation and growth of voids which subsequently link together and initiate failure.

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