J-INTEGRAL MEASUREMENTS WITH CERAMIC MATERIALS AT HIGH TEMPERATURES

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ABSTRACT

An alumina containing 3% glassy phase is investigated by three-point bending experiments in a closed loop tensile testing machine at 900°C. A high loading rate dependence of K_{IC} is found. The J-integral formalism is applied. Subcritical crack growth leads to overestimation of J_{IC}-values. When J_R-calculation is performed a "crack-closure" effect is observed and lower J_{IC}-values result.

KEYWORDS

Ceramic, high temperature, loading rate dependence, nonlinear behaviour, subcritical crack extension, J-integral, J-resistance curve, microstructure.

INTRODUCTION

Ceramic materials are more and more used in high temperature technology. At high temperatures, however, ceramic materials behave increasingly "plastic" or nonlinear. Linear elastic fracture mechanics (K-concept) is then no longer valid. The nonlinearity may be caused by grain boundary sliding and/or subcritical crack extension. To account for nonlinearity, especially for nonlinearity caused by slow crack growth of single or multiple cracks, an energy approach like the J-integral formation (J-concept) seems appropriate. The J-integral, as proposed by Rice in 1968, characterizes the crack tip stress and strain field under both elastic and elastic-plastic stress-strain conditions. Originally proposed as an analytical tool for crack tip stress and strain determination, it is increasingly used as a fracture parameter (Begley and Landes, 1972; Landes and Begley, 1974). For the linear-elastic case the critical fracture parameter J_{IC} can be related to the critical parameter K_{IC} (labeled K^*_{IC} in the text later on).

The analytical proof of the path-independence of the J-integral is given for the nonlinear-elastic case and is valid for the elastic-plastic case when no unloading occurs. Therefore, special attention has to be paid to subcritical crack growth when applying the J-concept to insure that no unloading occurs. It is necessary to combine the measured load-displacement curves with direct observation of crack extension and to use formalisms which account for slow crack growth when evaluating J.

EXPERIMENTAL

All experiments were performed with a three point bending device with a 24 mm span which consists of close loop testing machine controlling the displacement (Kromp, 1980a). A Si_3N_4-pushrod and an LVDT were used to determine the displacement at the specimen surface.

High temperatures were achieved by induction heating. The three-point bending specimens were of the dimension 30 x 7 x 2 mm^3. The initial crack length was simulated by a diamond-saw cut of width 100 - 200 μm, the depth of which chosen so that $a_0/w = 0.6$.

MATERIAL

In order to enhance the effects of nonlinearity at relative low temperatures (900°C), an alumina material with 3% glassy phase was used. The specifications of this material are given in Table 1.

TABLE 1 Specifications of Tested Material

Quality	Density	E-Modulus	$\bar{L}_3$ *	K_{IC} **
%	Kg/m^3	GN/m^2	μm	$MN/m^{3/2}$
97+3 SiO_2	$3.65 \cdot 10^3$	350	11	3.86±0.6

* average grain diameter

** fracture toughness at room temperature and loading rate of 0.1 mm/min in air

RESULTS AND DISCUSSION

Loading Rate Influence

At room temperature the K_{IC}-values of the material show a slight dependence on loading rate (increasing about 25% with cross-head speeds of 0.0025-1.0 mm/min. Kromp, 1980a). At 900°C there is a strong loading rate dependence with a maximum in K_{IC} at a cross head speed of 0.025 mm/min (displacement rates of 3 μm/min). The maximum is explained in terms of a continuous glassy grain-boundary phase which blunts microcracks (Fig. 3, centre above). At lower loading rates there is sufficient time for the second phase to flow into voids or triple points (Fig. 1, above left). At higher loading rates the K_{IC}-values decrease below the room temperature values and fracture surfaces are similar to those of specimens fractured at room temperature (Fig. 1, above right).

For temperatures up to 1400°C J-integral values were calculated from load displacement curves obtained from the closed-loop device at the displacement rate where the maximum K_{IC} occurs, 3 μm/min. Four methods were used to calculate the J-values. At 900°C the value calculated using Sumpter's method (Sumpter, 1976), which accounts for subcritical crack growth, fits the experimental data quite well.

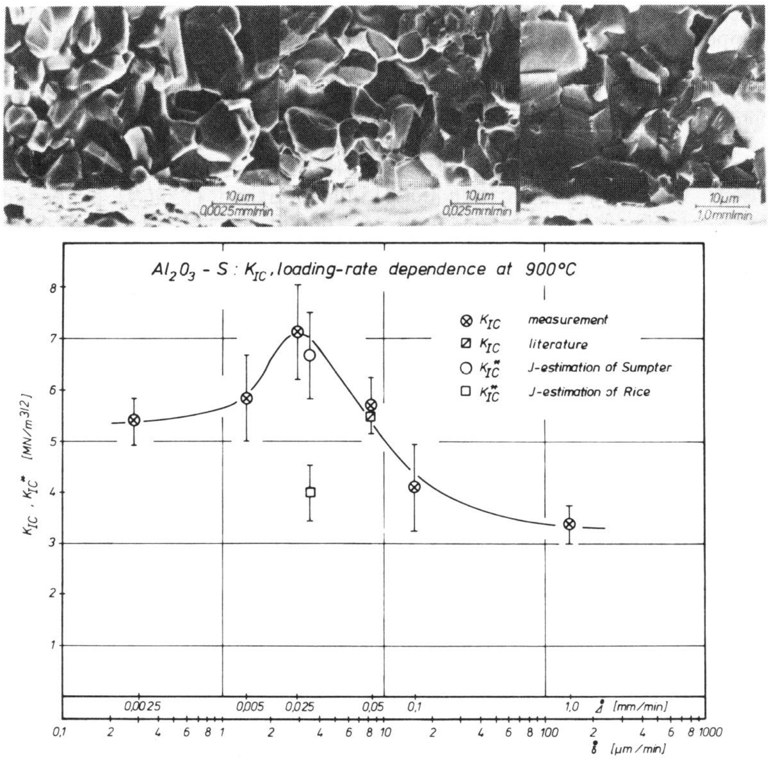

Fig. 1. Dependence K_{IC} and fracture surface appearance from cross-head speed $\dot{\Delta}$ and displacement rate $\dot{\delta}$.

Sumpter's method seems, however, relatively insensitive to slow crack growth. Rice's method (Rice, Paris and Merkle, 1973) seems to be more difficult to apply because of problems in determining the onset of slow crack growth. At 900°C the value calculated by this method lies 40% below the measured K_{IC}-value (Fig. 1). Below 1000°C the loading system was unstable (Kromp, 1980a); introduction of a load-cell of high stiffness cured this problem - for the stiff loading-system a high degree of nonlinearity and subcritical crack growth of single cracks was observed at 900°C.

In order to avoid the difficulties introduced by the evaluation methods of Sumpter and Rice, a J_R-calculation procedure was employed.

J_R-Calculation

The J-resistance curve calculation (J_R-calculation) was initially introduced for mild steels. It offered a method for determining the onset of slow crack growth and thus a possibility of obtaining J_{IC}-values (Landes and Begley, 1974). With the Multiple Specimen Method (MSPM) a set of identical specimens, loaded so that different crack lengths result, is used to calculate a J-Δa resistance curve (Δa = crack extension). J_{IC} is defined by the intersection point of this curve with the "stretch zone line" $J = \sigma_f \Delta a$, where $\sigma_f = \sigma_y + \sigma_u)/2$ and $\Delta a \approx$ COS (σ_f = flow stress, σ_y = yield stress, σ_u = ultimate stress, Δa = crack extension, COS = crack opening stretch). This line is given by the apparent crack extension caused by blunting and subsequent development of a stretched zone. For ceramic materials blunting and stretched zone are not yet known, therefore in the present work COS $\approx \Delta a \equiv$ o is assumed. J_{IC} will be taken as the value at Δa = o.

In the present work, however, another J_R-calculation procedure will be additionally performed: the Single Specimen Compliance Method (SSPCM) proposed by Clark and co-workers, 1974. A single specimen is loaded (still in the linear region) and then periodically unloaded 10% and reloaded. Although theoretically the J-concept is no longer valid, it was shown that the J-values are neglegibly affected, even by 30% unloading (Castro, Radon and Culver, 1979). The increase in compliance is measured and the crack extension can then be calculated by an iterative formula. J-values are then calculated by Rice's formula (Rice, Paris and Merkle, 1973) or the iterative formula of Garwood (Garwood, 1975), referred to later as J_c and J_g.

SSPCM Partial Unloading

In order to apply the Single Specimen Compliance Method (SSPCM), unloading to 50% is necessary to calculate alternation in compliance since there is a strong non-linearity in load-displacement curves; with increasing crack length a "cycling" appears - the loading-unloading cycle gets more and more elliptical. In the computer plot the smoothed curves are extrapolated linearly to the axis. The spacing between the curves is chosen arbitrary (Fig. 2).

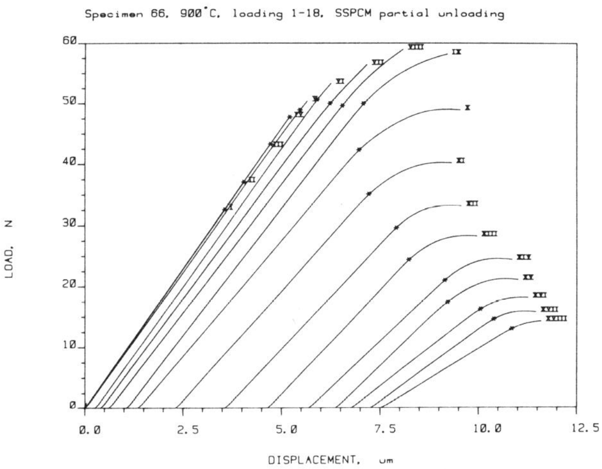

Fig. 2. Computer plot of the load-displacement record for partial unloading.

Figure 3 shows a plot of J-values over the calculated range of crack extensions. There is a large discrepancy between the values calculated by Rice's formula (Jc) and the values calculated by Garwood's formula (Jg) - for applications beyond final load, as realized in this case, this formula surely must be modified (for further discussion and the dotted symbols in Fig. 3 see Kromp, 1980b).

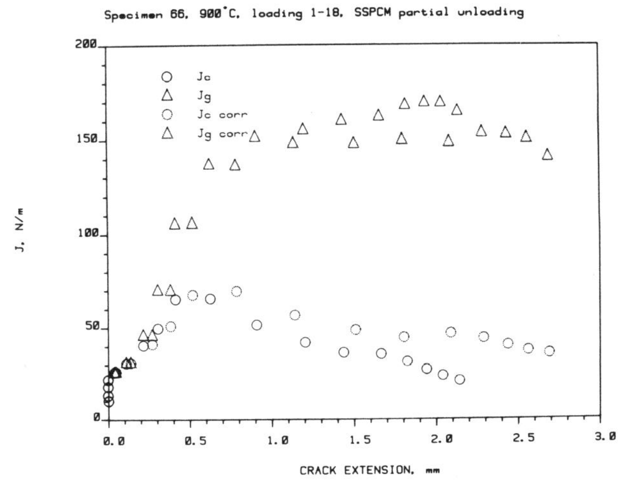

Fig. 3. J_R-curves, calculated from Single Specimen Compliance Method with partial unloading.

A comparison of the total crack extension, as calculated from compliance alternation, and the total crack extension, as measured microscopically after the last cycle, shows that the calculation underestimates crack extension by about 10%.

SSPCM Total Unloading

The compliance corresponding to the actual crack length should be higher than that measured by the partial unloading method. In order to investigate this effect more fully, the Single Specimen Compliance Method was applied with total unloading and measuring of actual crack extension at the end of every cycle (it would be better then to refer to these J values as ΔJ-values).

With increasing crack length a "crack-closure" effect becomes obvious by the appearance of a curvature at the beginning of the loading (see broken curves for cycles VI-X in Fig. 4). For calculating the change in compliance for these loadings the curves are extrapolated linearly by drawing a tangent at the point of minimal curvature. The J-values are not strongly affected by this extrapolation; compare Jo (for extrapolation) and J_{corr}-values (for original curvature) in Fig. 5. (for further discussion and J, Jc and Jg see Kromp, 1980b). From both SSPCM experiments the J_{IC}-value is calculated by extrapolation. Transferred to K-concept the corresponding K^{*}_{IC}-value is 2.4 $MN/m^{3/2}$ ± 15%, below the room temperature value. This is confirmed by application of the Multiple Specimen Method (MSPM) mentioned earlier (Kromp, 1980b).

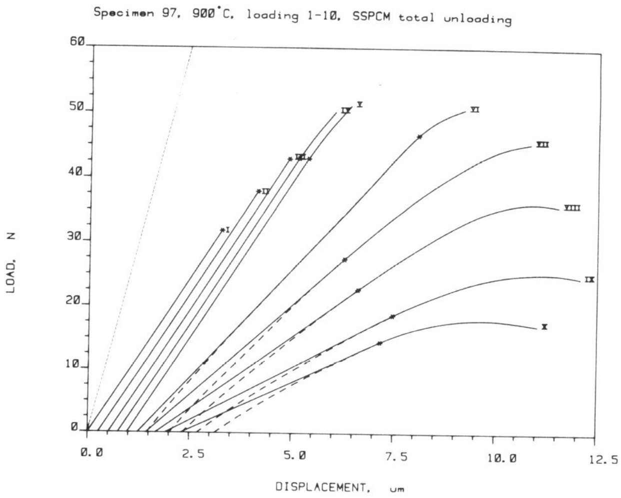

Fig. 4. Computer plot of the load displacement record for total unloading.

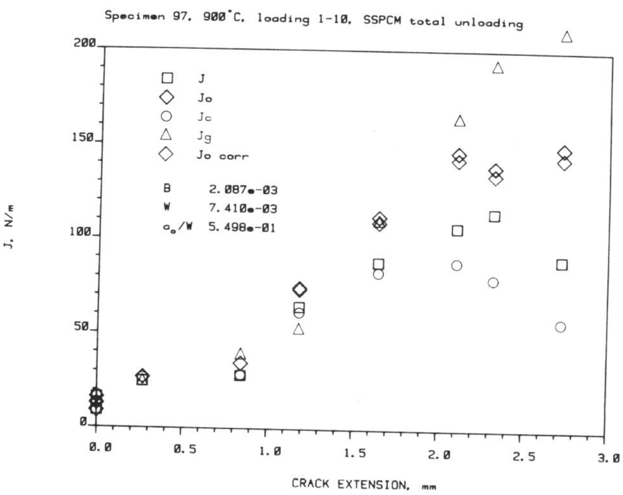

Fig. 5. J_R-curves, calculated from Single Specimen Compliance Method with total unloading.

The "crack-closure" effect is enhanced by the glassy phase. Figure 6 shows a part of the fracture surface, where the influence of the surface tension of the glassy can be shown directly.

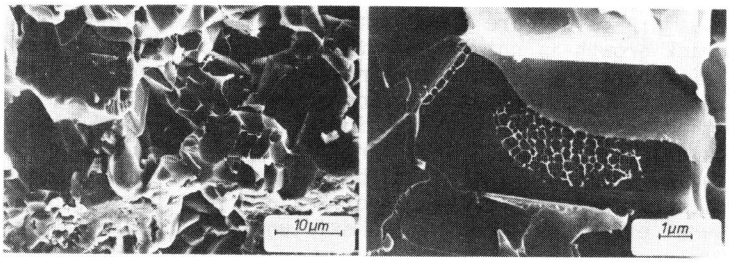

Fig. 6. Fracture surface of specimen 66.

The "crack-closure" effect is increased by total unloading; crack then closes nearly completely, as illustrated in the compliance curves in Fig. 7.

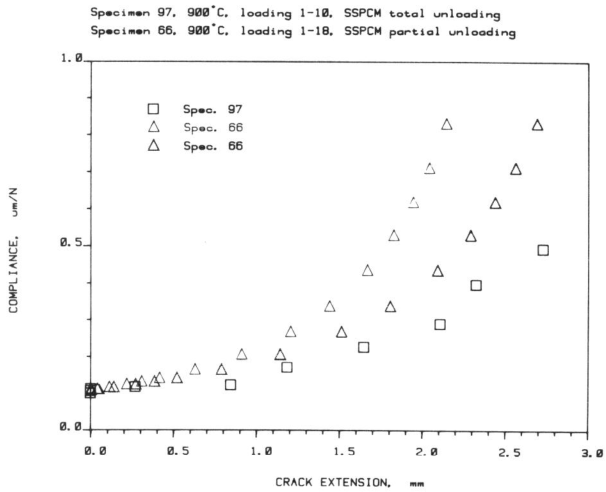

Fig. 7. Compliance curves of partially and totally unloaded specimens.

The compliance of the totally unloaded specimen exhibits a lower value (square symbols in Fig. 7) than the compliance of the partially unloaded (triangular symbols in Fig. 7), even when the scale for the partially unloaded specimen is stretched to the actually measured crack length after the last cycle (dotted symbols in Fig. 7).

SUMMARY AND CONCLUSION

At a temperature of 900^{o}C the strength behaviour of an alumina, containing a glassy phase was investigated. There was found a high loading rate dependence in K_{IC}-measurement; it seems prudent to take high loading rates when applying the K-concept as K_{IC}-values are overestimated at lower loading rates when subcritical crack growth is not taken into consideration. There was found a large amount of subcritical growth of single cracks causing the nonlinearity in load-displacement behaviour at displacement rates of 3 μm/min. The J-concept was applied; the J-calculation with the formulas of Rice and Sumpter over-estimated J-values (K^{*}_{IC}-values). For clarification J_R-calculation was introduced, yielding J_{IC}-values (K^{*}_{IC}-values) below room temperature values. The J_R-curves indicated that the cracks cannot be "Griffith" cracks because of a "crack-closure" effect enhanced by the existence of the glassy phase.

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