DIRECT VISUAL REGISTRATION OF CRACK EXTENSION IN CERAMIC MATERIALS AT ELEVATED TEMPERATURES

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With alumina containing a glassy phase and a commercially pure one for comparison the crack extension is directly measured in a displacement controlled test by microphotographing at 900 °C. It was then possible to calculate J_R-curves (energy approach) and K_I-curves (stress intensity approach) from visually measured crack lengths. These curves are compared with those calculated from a compliance test. The results seem to be better described by the J-integral method than by a K-concept of LEFM.

INTRODUCTION

The mechanical behaviour of ceramic materials at high temperatures is hardly understood, especially if viscous second phase reactions are involved. For instance, reaction bonded SiC with free silicon or alumina with glassy phase show a distinct dependence on loading rate if the fracture toughness is measured (Popp (1), Kromp and Pabst (2), Haug et. al.(3)). Also a rising crack resistance curve (K_I-curve) (Kleinlein et. al.(4)) and a rising J_R-curve is often registrated (Kromp(5)). The behaviour of the resistance curves are obviously due to the test procedure: e.g. for resistance curve calculations the shape of the curve depends whether the load-displacement curve was conducted completely controlled or not. Also, the shape of the curve depends heavily on different methods of crack length calculation. Normally the crack length is measured indirectly by a compliance test (Kromp (2), Kleinlein (4)). But viscous reactions at high temperatures, secondary crack formation and crack closure effects (Kromp (2)) suggest that it would be of great advantage to measure the crack length by either photographing or filming. In addition the kind of secondary cracking and viscous reactions may be studied. In the following the experimental equipment is presented which permits direct observing of the crack tip and continuous in situ measurements of crack propagation. With this equipment load-displacement curves were recorded. The continuously measured controlled crack growth permitted the calculation of K_I-curves, G_I-curves and J_R-curves which were compared to values calcu-

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lated from an indirect method of crack propagation measurement from compliance.

MATERIAL

For the experiments an alumina containing glassy phase was used, a material whose behaviour is expected to be nonlinear at high temperatures (Kromp (5)). For comparison experiments on pure alumina material were performed (Table 1).

Table 1: Materials

Material	Grain size μm	Young's Modulus MPa/m^2	K_{IC} MPa$\sqrt{m}$
Al_2O_3 + 3% SiO_2	~10	270.000 (900°C)	3.0 ± 0.1
Al_2O_3 99.7%	~ 1	290.000 (900°C)	4.2 ± 0.1

EXPERIMENTAL

The measurements were performed in a three point bending device, the displacement rate was controlled and directly measured by a Si_3N_4-pushrod in load line beneath the specimen (Kromp (5)). The displacement rate was chosen to 3 μm/min, corresponding to a loading rate where a maximum in K_{IC} for the glassy material at 900°C could be expected (Kromp (5)). The camera was fixed to a travelling microscope with a high working distance which enabled the crack propagation to be measured in situ by slide series with constant time spacing and by filming (Fig. 1b). From the correlation between constant displacement rate and photo series having constant time spacing, the crack growth and crack growth rate could be measured and calculated. The high temperature was achieved by induction heating of a $MoSi_2$-tube which surrounded the specimen and which can be used up to 1700°C in air. The experiments were performed at 900°C. The specimen dimensions were 35 x 7 x 2.3 mm^3 the span being 30 mm. The initial crack was simulated by a diamond saw cut resulting a crack to height ratio of 0.35 - 0.55 for different specimens.

RESULTS AND DISCUSSION

In the following, two series of results are compared and discussed:

a.) pure material (Fig. 2a-6a),
b.) glassy phase material (Fig. 2b-6b).

The load displacement curves

Figures 2a,b show the measured load-displacement curves, given by the open symbols. The open symbols to the right of the curve peak belong to points where a picture was made. The crossed symbols show the curve, corrected for the system compliance, with which crack lengths from alternation in specimen compliance was calculated.

The pure material exhibits a steep load decrease with a distinct sharp maximum as is expected for a brittle material.

The load-displacement curve of the glassy material is smoother. The peak is rounded and the decreasing part less steep. A more nonlinear behaviour is imposed by this load deflection characteristic.

Direct and indirect crack length measurement.

In Figures 3a,b the optically measured crack lengths (open symbols) are compared with those calculated from compliance. (crossed symbols)

The results are as follows:

1.) In the case of the pure material the direct visually measured crack lengths and those evaluated from compliance calculations are nearly identical (Fig. 3a).

2.) For the glassy phase material the compliance data are lower than the directly measured ones.

So the conclusion may be drawn that viscous reactions within the glassy phase material can cause an underestimation of the crack length.

The K_I-curve

The displacement controlled curve and the crack lengths computations, directly measured or by compliance , enable the calculation of the K_I-dependence as functions of crack lengths using the relation:*

$$K_I = \sigma\sqrt{a} \cdot Y(a/w) \quad \ldots\ldots\ldots\ldots \quad (1)^*$$

In the case of the pure material the K_I-curve is flat.(Fig.4a) Also, the K_I-curve calculated from compliance measurements is almost the same as that evaluated from directly measured crack length. The directly measured curve (open symbols) shows a certain maximum at higher crack lengths. It is assumed that wedging effects at the upper load bearing together with the shape of the correction function Y at high normalized crack lengths a/w (Fig. 5a,b) are responsible for the strange behaviour.

For the glassy phase material an increasing K_I-curve is measured if direct crack lengths are used (open symbols, Fig. 4b). It seems therefore that something like an energy "dissipation zone" has developed at the crack front due to viscous reactions which influences the K_I values with increasing normalized crack length a/w.

Concerning the K_I-curve calculated from compliance, the effect is much less distinct. The K_I-curve is flatter. The reason is that the crack length is underestimated by viscous processes. The underestimation increases with increasing crack length.

The G_I-curve

In the case of linear elastic behaviour and plain strain conditions the energy release rate G_I may be calculated from stress-intensity K_I by:

$$G_I = K_I^2(1-\nu^2)/E \quad \ldots\ldots\ldots\ldots\ldots (2)^*$$

*Relation (1) and (2) should only be used for linear elastic behav.

G_I-curves calculated from K_I data using the relation (2) are flat for the pure alumina (Fig. 5a). This corresponds to the K_I behaviour. The maximum G_I values at high crack lengths calculated with direct crack lengths also correspond to the maximum measured in the K_I evaluation (Fig. 4a) but is now multiplied by Y^2 instead of Y.

The J_R-curve.

From the load displacement curves and the visual measured crack length values for the external forces U were calculated. These were corrected for the elastic deformation energy (see Fig. 2a,b, open symbols). The values were normalized for thickness B and plotted over the normalized crack length a/w (Fig. 6a,b).

A numerical regression of various degrees for the normalized energy values U/B showed no tendencies for the J values calculated by:

$$J = - \frac{\partial}{\partial a} (U/B) \quad \ldots\ldots\ldots\ldots \quad (3)$$

For this first approximation, the J values were constant for all a/w (Fig. 7a, pure material; Fig. 7b, glassy phase material).

For the linear elastic case the value of J (see Fig. 7a) is identical to the value of G (see Fig. 5a) as was expected (pure material).

For the glassy phase material the increasing G_I values are contrary to the constant J values. At lower a/w G and J are nearly identical, with increasing a/w the G values increase rapidly.

The reason for this behaviour may be explained as follows: The glassy phase material is not linear elastic at high temperatures. The question is whether a energy "dissipation zone" is developing at the crack front whose size increases with increasing crack length. This nonlinearity is not considered in the stress intensity relation (1). It is to be assumed that the shape of the stress distribution σ is changed by nonlinear effects. In any case, a modification of crack length in the region of high normalized crack lengths has a strong effect on Y and Y^2 and therefore on K_I and G_I (see Fig. 4a,b; 5a,b).

CONCLUSION

For special ceramic materials like alumina with glassy phase at high temperatures the concept of LEFM cannot be applied. Non linear behaviour, caused by viscous reactions gives inconsistant results if a linear elastic K-concept is performed. An energy concept like J seems to be more practicable.

SYMBOLS USED

a = crack length (mm)
B = thickness of specimen (mm)
E = Young's Modulus (MPa/m^2)
G = Energy release rate (N/M)
K_I= stress intensity ($MNm^{-3/2}$)
J = J-Integral (N/m)
W = width of specimen (mm)
Y = geometrical correction function (/)
σ = applied stress (MPa)
v = Poisson's ratio

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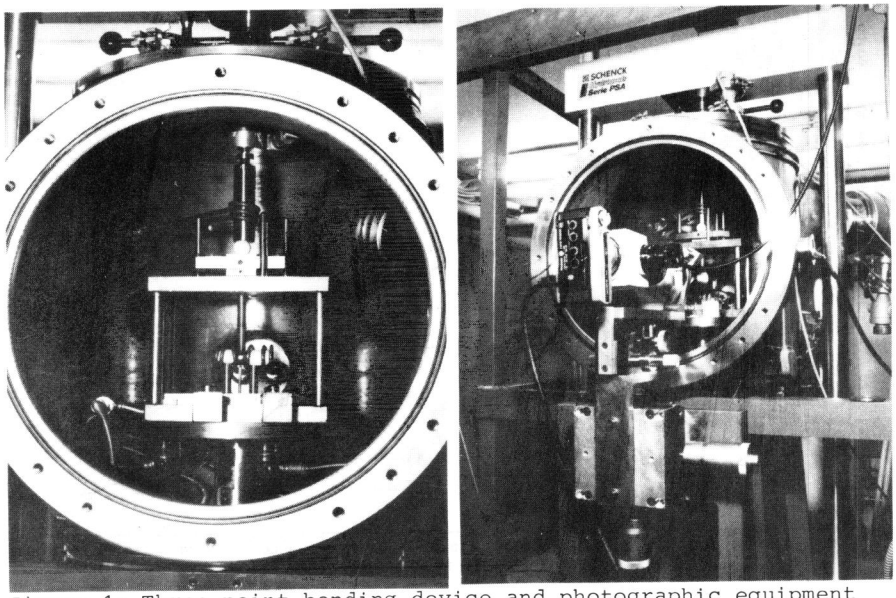

Figure 1: Three point bending device and photographic equipment for the measurement of the controlled crack growth.

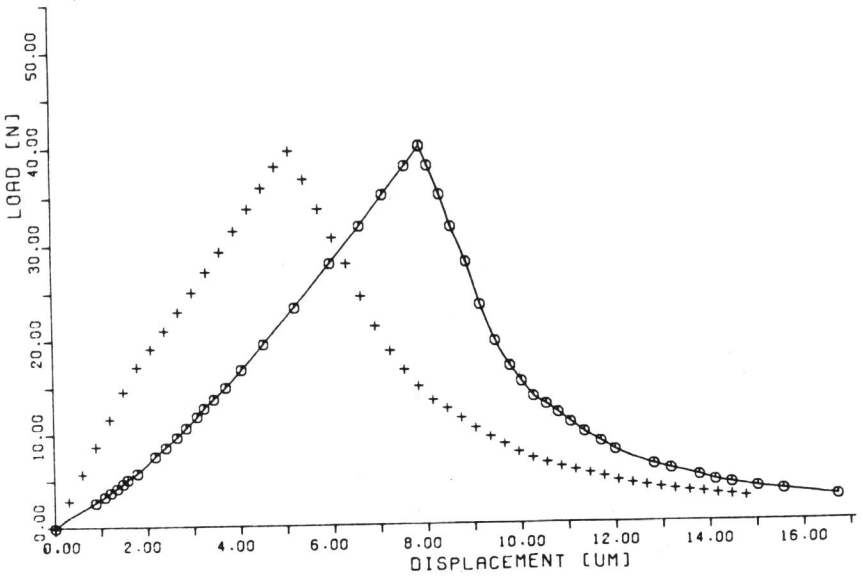

Figure 2a: Load-displacement curve for the pure material.
(Open symbols: original curve, crossed symbols: corrected curve)

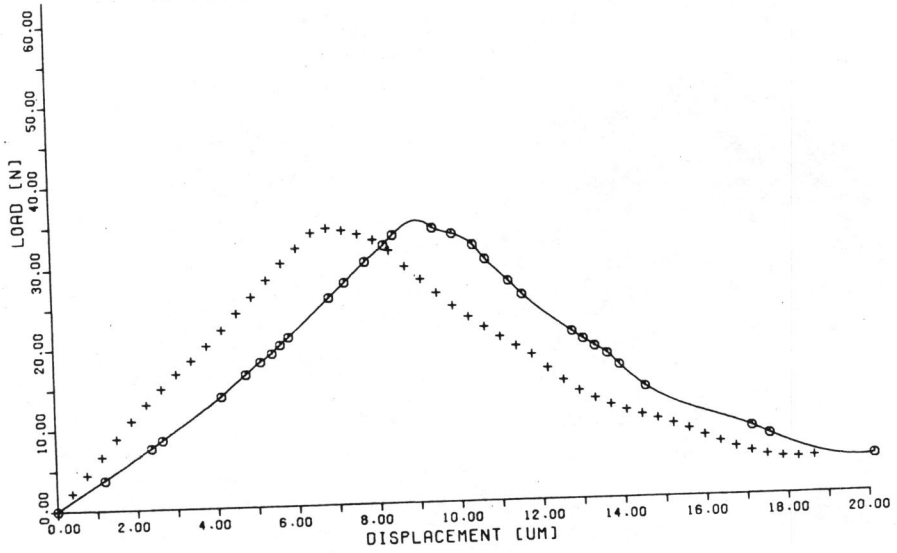

Figure 2b: Load-displacement curve for the glassy material.

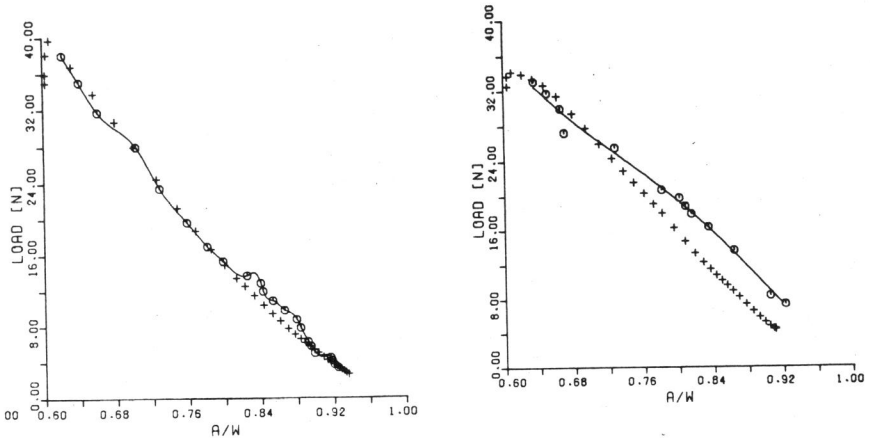

Figure 3a,b: Crack length comparison.
(Open symbol: direct visually measured crack length,
crossed symbols: crack length calculated from compliance)

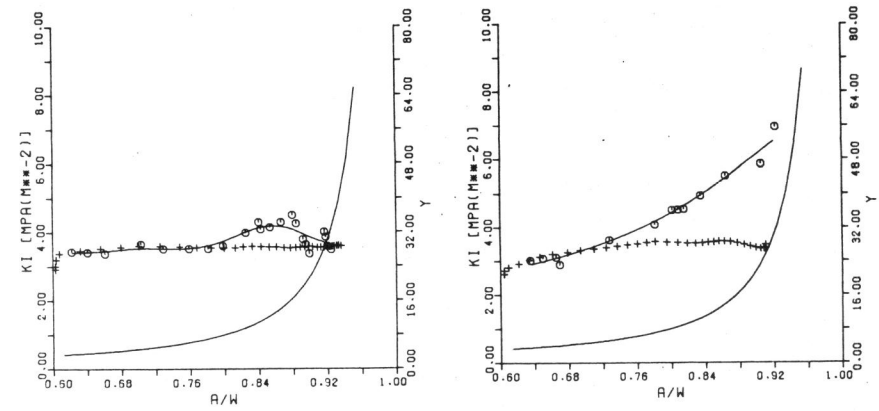

Figure 4a,b: K_I values over a/w.
(Drawn line: Y-correction function over a/w)

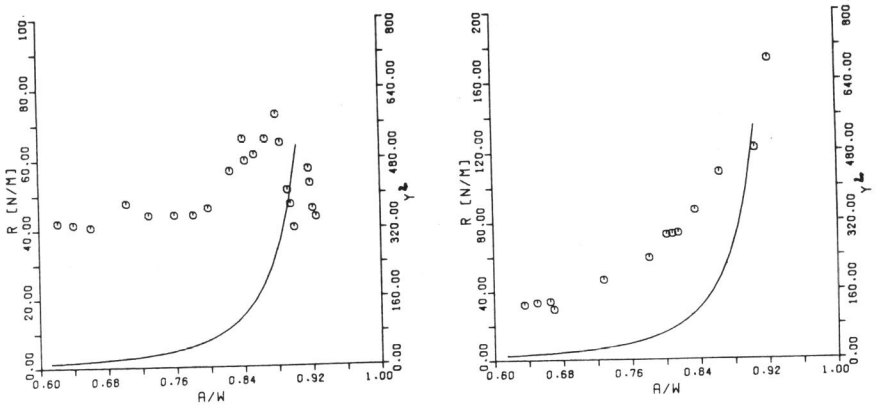

Figure 5 a,b: G_I-curves, calculated from K_I values.
(Drawn line: Y^2 values for corresponding a/w)

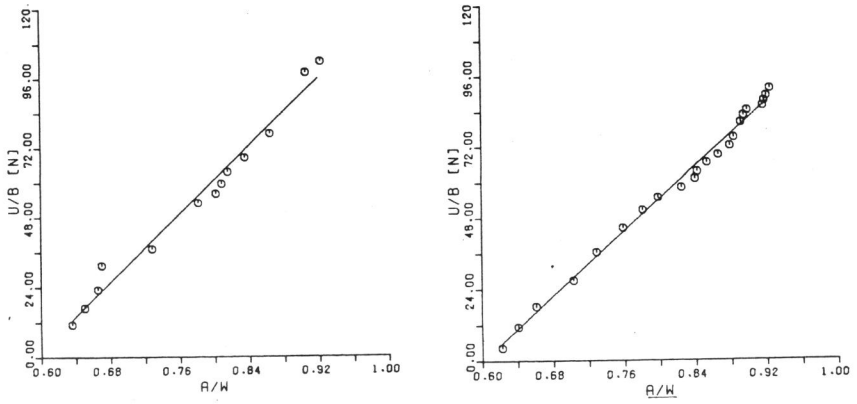

Figure 6a,b: Normalized work of external forces, spent from crack propagation and calculated from load-displacement curves (from Fig. 2a,b crossed symbols)

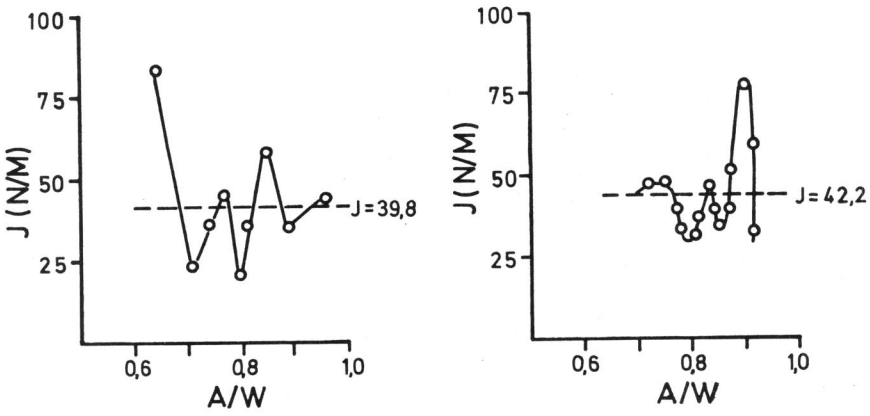

Figure 7a,b: J-Integrals