JOURNAL OF MATERIALS SCIENCE 18 (1983) 265–270

Increase of crack resistance during slow crack growth in Al_2O_3 bend specimens

R. STEINBRECH, R. KHEHANS, W. SCHAARWÄCHTER
Lehrstuhl für Werkstoffwissenschaften, Abt. Chemietechnik, Universität Dortmund, West Germany

Fracture experiments under conditions of slow crack growth were performed with prenotched three-point bend specimens of polycrystalline alumina. The influence of notch depth, specimen geometry, mean grain size and deformation velocity on the crack-resistance force (R) was investigated. Within one specimen R increases with crack propagation up to a factor of 4 (R-curve) accompanied by small changes (slight decrease) in crack velocity. No unique R-curve exists for these ceramics. Both the shape and the position of the R-curve are influenced by deformation velocity and notch depth. The latter effect means that for a certain crack length, R is larger in a specimen with the shorter notch (memory effect). The results are discussed in terms of energy dissipation by microcracks. The significance of both single R value and R-curve for fracture characterization of polycrystalline alumina is questioned.

1. Introduction

The brittle fracture behaviour of alumina at room temperature is usually described by fracture parameters derived from linear elastic fracture mechanics. The critical stress intensity factor, K_{IC}, and the crack resistance force, R, are often used as relevant values. Experiments show, however, that different fracture test techniques yield different results for the same material. K_{IC} measurements on identical Al_2O_3 can scatter by about 40% [1]. The use of a single K_{IC} value seems particularly uncertain, if slow crack growth precedes catastrophic fracture.

Measurements of R during slow crack growth through the complete ligament of notched bend specimens show, in fact, that R generally increases with crack propagation (R-curve) [2, 3]. It has been proposed to use such an R-curve for fracture characterization of alumina [1]. The scatter in $R-(K_{IC})$ values for bend specimens should then refer to different points on the R-curve due to slightly different experimental procedures. But this does imply that the R-curve is unique and characteristic for a certain alumina material.

It was the aim of this work to examine this supposition and therefore to investigate the influence of some appropriate parameters on the R-curve of prenotched three-point bend specimens. The experiments were performed with two polycrystalline alumina batches of different grain size. First it was determined if the shape of the R-curve was caused by crack propagation kinetics. The deformation (cross-head) speed and, therefore, the crack velocity was varied. Then the specimen geometry was altered to investigate the influence of notch depth and specimen height on the shape of the R-curve.

2. Experimental details

2.1. Experiments

Prenotched alumina three-point bend specimens were prepared in the following way: alumina powder (CT 8000, purity > 99.8%, Giulini GmbH, Ludwigshafen) was isostatically pressed and sintered in vacuum. Two batches of different mean grain sizes, D, were adjusted: Al_2O_3(I), $D \simeq 10$ μm; Al_2O_3(II), $D \simeq 2\,\mu$m. The geometry of the specimens (Fig. 1 and Table I) was chosen so that for constant width, $b = 5$ mm, the height, W, varied from 4 to 7 mm. The ratio between lower support length S and W was taken constant ($S/W = 8$). With a diamond saw (width of saw cut $\simeq 150$

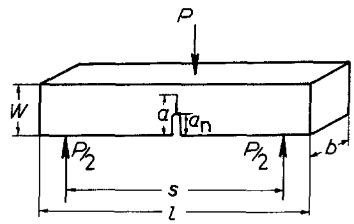

Figure 1 Three-point bend specimen geometry.

μm) the specimens were notched to different depths, a_n.

The specimens were deformed in a testing machine (Instron 1195) with a stiff three-point loading adjustment that allows slow crack growth. Load–deflection curves were measured at two constant cross-head speeds ($V_T = 10$ and 1000 $\mu m\ min^{-1}$).

All experiments were performed at room temperature and in silicone oil to avoid the influence of humidity [4].

2.2. Determination of crack-resistance force by compliance measurement

The crack length, a, was actually determined from the load–deflection $P(d)$-curve by measuring the compliance $C^{exp}(a) = d/P$ and comparing this value with the result of theoretical compliance calculation $C^{calc}(a)$ [2, 5]. The calculation method varies the crack length until $C^{calc} = C^{exp}$.

This method of crack length determination was sustained by microscopic observation of the crack length on the specimen surfaces. Fig. 2 shows the excellent coincidence of both methods. Thus the crack length can be determined for any point on the non-elastic $P(d)$-curve from a compliance measurement. The crack-resistance force is then given by

$$R = \frac{P^2}{2b} \cdot \frac{dC}{da}.$$

If the load–time curve is also plotted the mean crack velocity $\Delta a/\Delta t$ can be determined as well.

3. Results

A typical load–deflection curve, $P(d)$, and the resulting R-curve as a function of normalized crack length (a/W) are shown in Fig. 3. R increases with crack extension. Starting from the normalized notch depth, $a_n/W = 0.57$, to the end of experiment at $a/W \simeq 0.93$, there is an increase of R by a factor of 4. For illustration, the calculated $P(d)$-curve for R = const. is also plotted in Fig. 3a (dotted line). The deviation of the measured $P(d)$-curve from the dotted one is evident and depends on parameters which will be treated in the following sections.

3.1. Crack velocity

It is well known that the resistance force, R, which has to be overcome in order to extend a crack, depends on crack velocity $\dot{a}$ [4]. Measurements of crack extension in double torsion specimens (where R is independent of crack length) yield a weakly increasing R when the crack velocity increases by some orders of magnitude [6]. To estimate the influence of this effect on the R-curve, the mean crack velocity $\Delta a/\Delta t \simeq \dot{a}$ is plotted in Fig. 3b in addition to the $R(a/W)$-curve. For the given constant cross-head speed ($V_T = 10\ \mu m\ min^{-1}$) the crack-velocity is nearly constant ($\Delta a/\Delta t \simeq 3 \times 10^{-4}\ m\ sec^{-1}$) with only slight decrease at higher crack lengths. Thus $\dot{a}$ lies within the range of subcritical (slow) crack growth and is not related to the permanent increase of R.

3.2. Cross-head speed

If the crack extension is controlled by a higher cross-head speed, V_T, the shape of the R-curve changes (Fig. 4). Although no systematic measurements yet exist, some first results indicate: (i) at

TABLE I Experimental parameters

Material	Mean grain size D (μm)	Specimen size* W (mm)	a_n/W 0.2	0.4	0.6	0.8	Cross-head speed V_T ($\mu m\ min^{-1}$)
Al_2O_3 (I)	10	4			X		10
		5	X	X	X	X	
		6			X		
		7			X		
Al_2O_3 (II)	2	5			X		10
					X		1000

*b = 5 mm, $S/W = 8$.

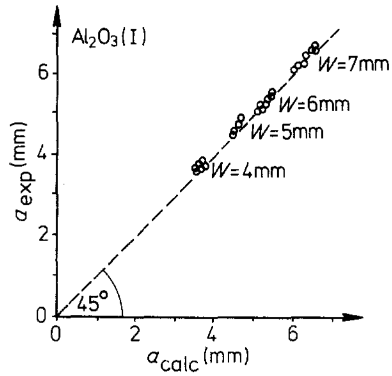

Figure 2 Comparison of calculated (a_{calc}) and microscopically observed (a_{exp}) crack length.

higher V_T the R-curve increases no longer monotonically, a maximum in the curve is observed after some crack extension; (ii) the values of R are over a wide range above those at slow V_T; (iii) both R-curves start at nearly the same notch value, $R_0 = R\ (a_n/W)$.

3.3. Notch depth

The influence of different notch depths on R-curves is shown in Fig. 5. For simplification only one of at least three measured curves is plotted for each notch depth. The R-curves are clearly separated over a large range of crack extension until they meet at $a/W > 0.9$. The notch values (R_0) show no systematic relationship with notch depth (mean value of 47 specimens of Al_2O_3(I): $\bar{R}_0 = 32\,\mathrm{N\,m^{-1}}$, standard deviation $7\,\mathrm{N\,m^{-1}}$). So R_0 is considered to be constant within experimental scatter. Starting from R_0, the crack resistance increases with crack propagation up to a factor of 4. From Fig. 5 it is obvious that different values of R result from the saw cut notch (artifical crack) and a real crack of the same length. Moreover, for the same crack length (a/W) R is highest for specimens with the shortest notch (a_n/W), as can be seen in Fig. 5 for example at $a/W = 0.6$: values of R of about 20, 60 and $90\,\mathrm{N\,m^{-1}}$ result from notch depths of $a_n/W = 0.6$, 0.4 and 0.2, respectively.

It must be emphasized that this effect is not caused by the method of determining R because the calculation programme cannot distinguish between the crack length, a/W, and the same notch depth, a_n/W. Furthermore, measured compliance values of saw cut notch and cracks of the same length are equal within experimental scatter. The different values of R at equal total crack length, a, are caused by the fracture behaviour of alumina, the crack obviously remembers from which notch depth it originally started ("memory effect").

3.4. Specimen height

At different notch depths (Fig. 5) and given a/W, the absolute crack extension length, $a - a_n$, is different. Thus it was investigated if the increase in R depends on $a - a_n$ or on the normalized crack extension $(a - a_n)/W$. For constant a_n/W the specimen height was varied from 4 to 7 mm to obtain different ligament lengths. Fig. 6 illustrates the results for $a_n/W = 0.6$ with R plotted as a function of normalized crack length (a/W). Within a range of scatter the different R-curves match each other. There seems to be no systematic

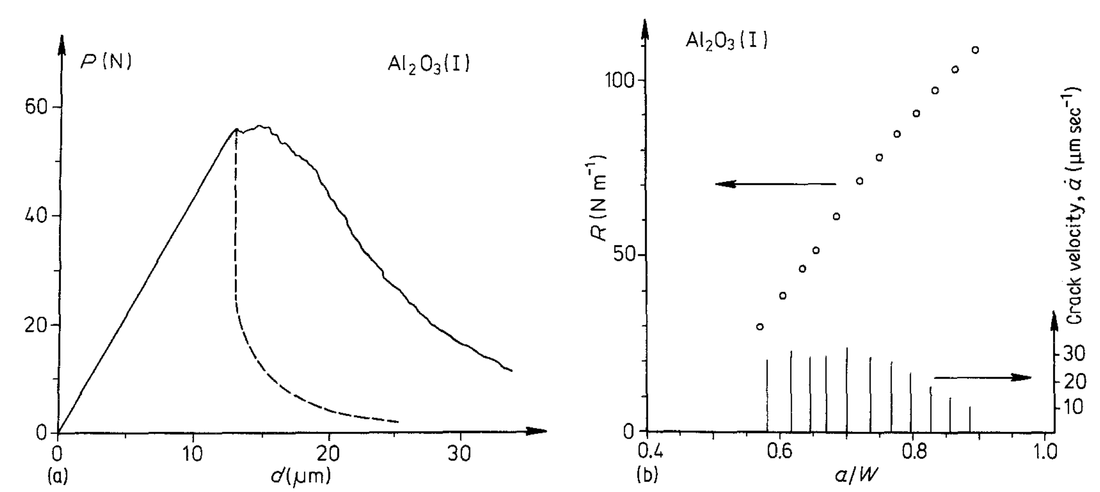

Figure 3 (a) Load–deflection curves for constant crack resistance: solid line, measured values; broken line, calculated values for constant crack resistance. (b) Crack resistance curve determined from (a).

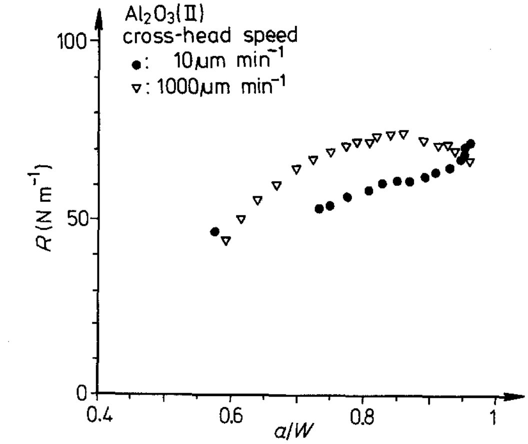

Figure 4 Influence of cross-head speed on the shape of the R-curve.

deviation. Therefore $(a-a_n)/W$ appears to be an appropriate variable for plots of R-curves.

3.5. Grain size

R-curves of the two alumina batches (mean grain size 10 and 2 μm), determined at $a_n/W = 0.6$ and $V_T = 10\,\mu\text{m}\,\text{min}^{-1}$, start at different values, R_0, and also have different slopes (Fig. 7). The finer grained material results in a higher value of R_0 (mean value of 17 specimens of Al_2O_3 (II): $\bar{R}_0 = 42\,\text{N}\,\text{m}^{-1}$, standard deviation $3.5\,\text{N}\,\text{m}^{-1}$) but a more gentle slope. Therefore, the beneficial effect of a smaller grain size for the value of R for the start of the crack is lost during further crack propagation. However, more experiments with batches of different grain size are necessary to quantify this effect.

4. Discussion

Summarizing the results of Section 3, the crack resistance force, R, can be resolved into two additive terms. The first term, R_0 (notch value), is constant for different notch depths, a_n/W but depends on grain size (D). In addition, a weak dependence of R_0 on crack velocity ($\dot{a}$) is expected (Section 3.1) but could not be observed because of experimental scatter. The second term, ΔR, describes the increase of crack resistance during crack propagation (comparable to strain hardening) and is responsible for an additional energy dissipation. It depends on notch depth (a_n/W), normalized crack extension ($(a-a_n)/W$), grain size (D) and deformation velocity (V_T):

$$R = R_0(\dot{a}, D) + \Delta R\left(\frac{a_n}{W}, \frac{a-a_n}{W}, D, V_T\right).$$

Although a quantitative analytical relationship is not yet established, some remarks concerning the mechanism of the increase in the value of R can be made

(i) Crack propagation kinetics are not of primary importance for an explanation of ΔR as the influences of crack velocity on R can be neglected in comparison to ΔR differences within one R-curve. Furthermore, a model based only on thermally activated crack growth would not explain the interdependence between ΔR and $(a-a_n)/W$.

(ii) Recent models try to explain the increase of R by microcracking effects. An energy dissipating (stress releasing) zone of microcracks in front of the crack tip is postulated (process zone [7]). The increase of R with crack extension then

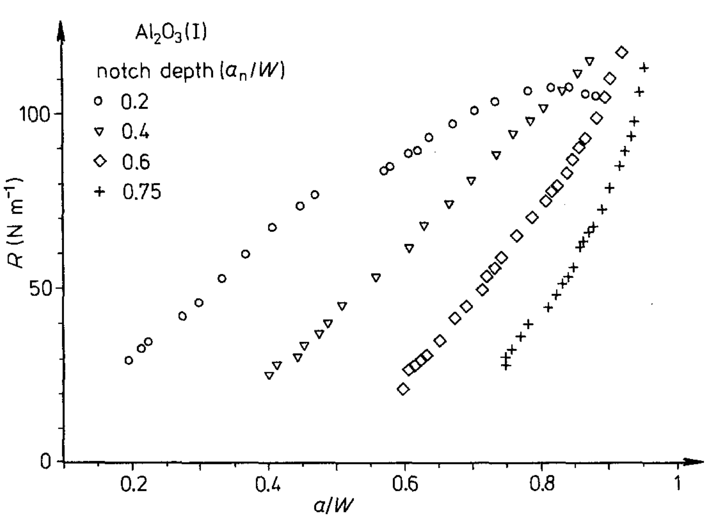

Figure 5 R-curves for constant specimen size ($W = 5$ mm) but different notch depths.

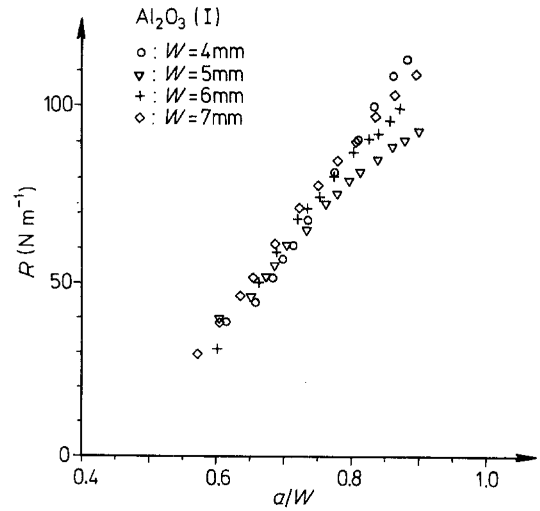

Figure 6 R-curves for constant relative notch depth, $a_n/W \simeq 0.6$, and different specimen heights, W.

could be a consequence of a growth of this zone [8]. Direct experimental observation of such a growing microcrack zone is not known to the authors, yet the results of Section 3 enables one to check this assumption to a certain extent: following Hoagland [9] the density of microcracks within the process zone should be independent of crack extension, whereas the size of the zone (ρ) is proportional to R. An estimate of this size yields $\rho \simeq 200\,\mu$m if the measured values of R_0 are used [10]. As R increases during crack extension up to a factor of 4, the process zone should reach the compression surface of the specimens far before the "main" crack. Then, ρ cannot grow any longer and a significant change in the R-curve for $a/W > 0.8$ ($W = 5$ mm in all figures

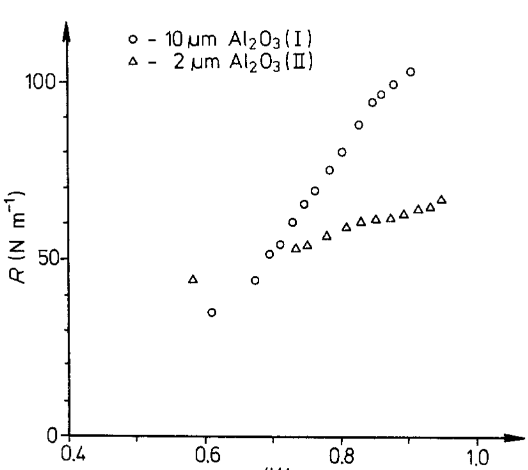

Figure 7 Effect of grain size on the R-curve.

except Fig. 6) should be the consequence. The experimental results hardly confirm this model. Within the process zone concept, it seems to be more reasonable to assume that the density of microcracks rather than ρ increases with crack extension, as has been suggested by Buresch [11].

(iii) However, it should be emphasized that the model of a process zone in front of the crack tip can hardly account for the observed memory effect. If microcracks cause the energy dissipation effect a possibly better explanation may be given by a model [12] which takes into account microcracks at the rear part of the crack. Microcracks formed in the process zone remain as a "debris" layer along the crack walls when the crack grows. These rear microcracks can dissipate additional energy during crack propagation. The size of the "debris" layer and the density and distribution of microcracks in it should depend on crack extension, stress distribution, grain size and crack velocity. The consequences deduced from such a "debris" layer model would qualitatively agree with the observed R-curve behaviour.

It should be mentioned, however, that friction of serrated crack walls has to be considered as an alternative mechanism which could equally as well allow a rationalization of the experimental observations.

Experiments to distinguish between these alternative models are in progress [13].

5. Conclusions

The results have shown that there exists no unique R-curve which enables one to characterize the fracture behaviour of an alumina material. Both the shape of the R-curve and the magnitude of R also depend on external test parameters.

On the basis of actual understanding it must be doubted if it is sufficient or even meaningful to describe the fracture behaviour of polycrystalline alumina by either a single R–(K_{IC}) value or a single R-curve.

Acknowledgements

The authors are grateful to B. Arsenijevic for specimen preparation and to W. Krüger for technical assistance.

References

1. H. BRETFELD, F. W. KLEINLEIN, D. MUNZ, R. F. PABST and H. RICHTER, *Z. Werkstofftechnik* **12** (1981) 167.
2. F. W. KLEINLEIN and H. HÜBNER, in "Advances in

Research on the Strength and Fracture of Materials", edited by D. M. R. Taplin (Pergamon Press, Oxford and New York, 1978) p. 883.
3. F. W. KLEINLEIN, thesis (doctorate), Universität Erlangen/Nürnberg, (1980).
4. S. M. WIEDERHORN, in "Fracture Mechanics of Ceramics", Vol. 2, edited by R. C. Bradt, D. P. H. Hasselman and F. F. Lange (Plenum Press, New York, 1974) p. 613 ff.
5. J. NAKAYAMA, *J. Amer. Ceram. Soc.* 48 (1965) 583.
6. R. STEINBRECH and R. KNEHANS, to be published.
7. F. E. BURESCH, in "Fracture Mechanics of Ceramics", Vol. 4, edited by R. C. Bradt, D. P. H. Hasselman and F. F. Lange (Plenum Press, New York, 1978) p. 835.
8. K. KŘÍŽ, H. HÜBNER and F. W. KLEINLEIN, DVM Bruchvorgänge, 1978, in "Vorträge der 10. Sitzung des Arbeitskreises Bruchvorgänge" (DVM, Berlin, 1979) p. 87.
9. R. G. HOAGLAND, *Scripta Metall.* 9 (1975) 970.
10. F. E. BURESCH, ASTM-STP 678 (1979) 151.
11. *Idem*, private communication (1981).
12. W. TRADINIK and U. KROHN, "Festigkeitsseminar" (colloquium), MPI, Stuttgart, 1981, private communication.
13. R. KNEHANS and R. STEINBRECH, *J. Mater. Sci. Lett.* 1 (1982) 327.

Received 31 March
and accepted 2 July 1982