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Simple method to measure the crack resistance of ceramic materials

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A new and simple method to study the change in crack resistance during the process of crack growth in ceramic materials has been developed. The method is based on using the chevron-notched short-bar test which is generally accepted as a convenient method for measuring fracture toughness. The simple modification described here allows one to measure fracture toughness using the assumption that fracture toughness changes in the process of crack growth (presence of crack resistance curve, or R-curve). This method presents many advantages, especially the long stable crack growth under mode I fracture specimen and plane strain, small specimen size and no need of pre-cracking for measuring R-curve behaviour in ceramic materials.

1. Introduction

Crack stability is one of the most important properties for damage-tolerant structural and engineering ceramics. When the stable crack in such materials is larger than the size of any pre-existing flaw, material failure is less influenced by the nature of the pre-existing flaws.

The crack resistance to growth is often characterized by measuring the crack resistance curve (R-curve). The curve shows, in terms of the critical values of stress intensity factor or the energy release rate, the increase in resistance with increasing crack length. The crack growth resistance curve has been studied by several techniques [1–7] ; however, these techniques are difficult to implement and are time consuming.

The chevron-notched short-bar test introduced by Barker [8] is, at present, generally accepted as a convenient method for measuring the plane-strain critical stress-intensity factor [9]. The growing crack in the chevron-notched specimen is stable in the long range due to the nature of the specimen geometry. Therefore, the measurement of the critical value of the stress intensity factor can be performed on the same specimen for different lengths of a growing crack. Thus, in principle, the method can be used for measuring R-curve behaviour [10]. The chevron-notched specimen has other advantages, such as relatively small size, no need for pre-cracking and the presence of a guiding notch to give a straight crack path.

Recently, Descamps *et al.* [11] presented a new method called "crack line wedge loading (CLWL) " to determine the R-curve behaviour of ceramic materials. Application of their method, however, presents difficulties in making a pre-crack, in indirect measurement of load and ambiguity in determining crack size. This paper presents the results of a study on the use of the chevron-notched specimen to measure the R-curve behaviour for several silicon nitrides and SiC whisker-filled alumina ceramic materials. In addition, this paper discusses the use of the chevron-notched test for fracture toughness measurements (K) when the materials have a rising crack growth resistance.

2. Materials

The study was performed by using three types of sintered silicon nitride (Si_3N_4), which A–C, and 20 vol % SiC whisker-filled alumina matrix ceramic materials. Material A is a commercial silicon nitride with low fracture toughness which does not exhibit R-curve behaviour. This sample is hot-pressed silicon nitride (SN-84H), made by NGK Technical Ceramics. Materials B and C have relatively high fracture toughnesses and R-curve behaviours. These samples, supplied by Dr C. W. Li of the Metals and Ceramics Laboratory, Corporate Research and Technology at Allied-Signal Inc., Morristown, NJ, USA, and designated as AS-700, are monolithic Si_3N_4 prepared by gas pressure sintering green bullets, which were formed by cold isostatic pressing. A SiC(20 vol % whisker)-reinforced alumina (Ceramics Inc., Salt Lake City, UT, USA) is made by hot isostatic pressing. The details of materials composition, structure and sample preparation can be found in Refs [12] and [13]. Table I shows the mechanical properties of the tested materials.

3. Experimental procedure

The short-rod specimens (Fig. 1) were machined to the dimensions: $H = 11.05 \pm 0.04$ mm, $B = 12.7 \pm 0.05$ mm and $W = 19.05 \pm 0.075$ mm. A 0.35 mm thick diamond saw was used to make the chevron-notched specimens with parameters: $a = 6.75 \pm 0.07$ mm and $2\theta = 55.2 \pm 0.5$, as shown in Fig. 1. All tests were performed on a Fractometer II tester (manufactured

TABLE I Mechanical properties of the tested materials and compliance as a function of crack length

Ceramic	Density ($g.cm^{-3}$)	Four-point bend strength (MPa)	Fracture toughness ($MPa\,m^{1/2}$)	Weibull modulus	Young's modulus (GPa)
A	3.24	850	5.7 ± 0.1	13.5	311
	$C = 0.132a^2 + 1.2a + 5.97$				
B	3.30	580	9.0 ± 0.5	15.7	303.5
	$C = 0.213a^2 - 0.52a + 7.99$				
C	3.31	550	10.6 ± 0.8	18.2	300.6
	$C = 0.152a^2 - 1.94a + 9.82$				
Alumina	–	565	6.5 ± 0.2	–	350
	$C = 0.223a^2 - 0.32a + 7.18$				

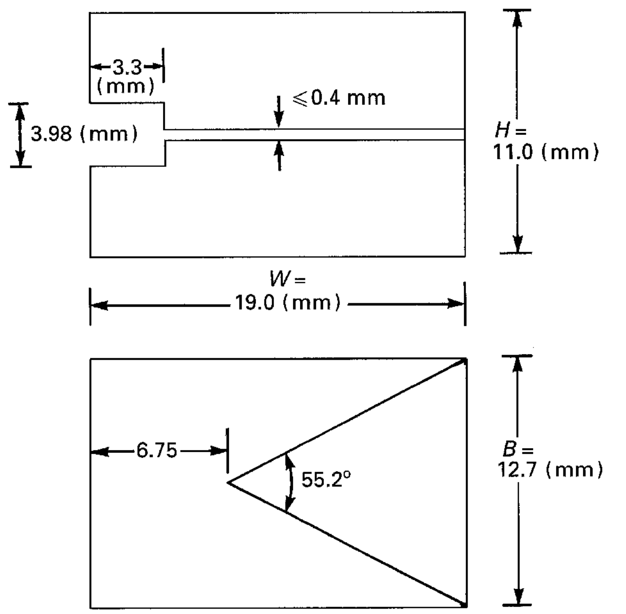

Figure 1 Specimen geometry and associated dimensions.

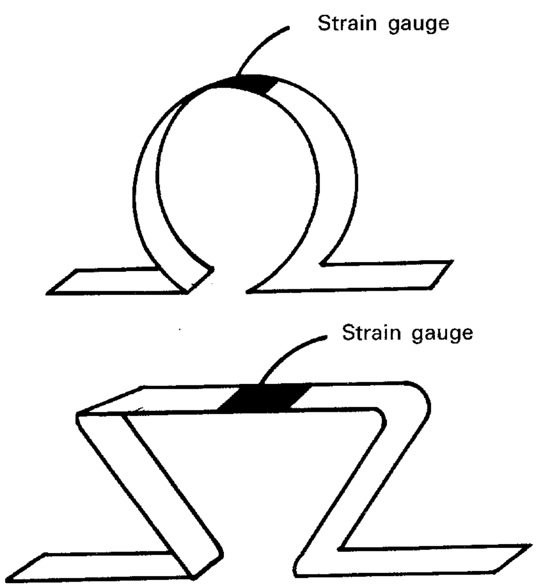

Figure 2 Schematic of device to measure crack opening.

by TerraTek System, Salt Lake City, UT), at room temperature and a constant loading and unloading crack opening speed of $0.05\,mm\,min^{-1}$. The opening of the Fracjack test fixtures was measured by using a special device glued to the fixture. The device (Fig. 2) is a bent section of nichrome tape (cross-section 0.16×3.1 mm) with an attached strain gauge. The idea of the device is to record the relatively large displacement of the tester fixture by measuring deformations in the metallic tape. The tape deforms elastically throughout the entire range of displacements. In order to test the reliability of the device measurements two different sizes and shapes of the device were used. To calibrate the devices, a high magnification extensometer calibrator was used.

The fracture toughness values of all ceramics were obtained by a standard short-bar test procedure and employing the elastic-plastic analysis, developed by Barker [14]. The values were confirmed by using the chevron-notched three-point bending test [13].

It was found that the single specimen method, in which the compliance and the respective crack length are measured on the same specimen at each loading–unloading step, is difficult to use with the chevron-notched specimen because the crack length measurement is obstructed. Two identical specimens of each material were used for R-curve determination by the compliance method. The first sample was used to establish the relationship between the crack length and the elastic compliance. The same diamond saw was used to imitate the propagating crack by a sequence of cuts each of which increased the slot length by ca. 0.5 mm. After each cut, the loading and unloading cycle in the elastic range was performed and plotted without any changes in test conditions. The measured compliances at different crack lengths were plotted as shown in Fig. 3. It is believed that this compliance curve will determine the nature of R-curve [5]. The experimental relationship was approximated as:

$$C = K_1 a^2 + K_2 a + K_3 \quad (1)$$

where C is the compliance, a is the crack length and K_1, K_2 and K_3 are constants. The constants were found by a least-square fits. From Equation 1, the slope (dC/da) is:

$$dC/da = 2K_1 a + K_2 \quad (2)$$

The second specimen and the same testing set-up were used to grow the crack in small increments and to establish the corresponding values of the critical force, P. After each increment, the specimen was partially unloaded (ca. 15% of the maximum load) to determine

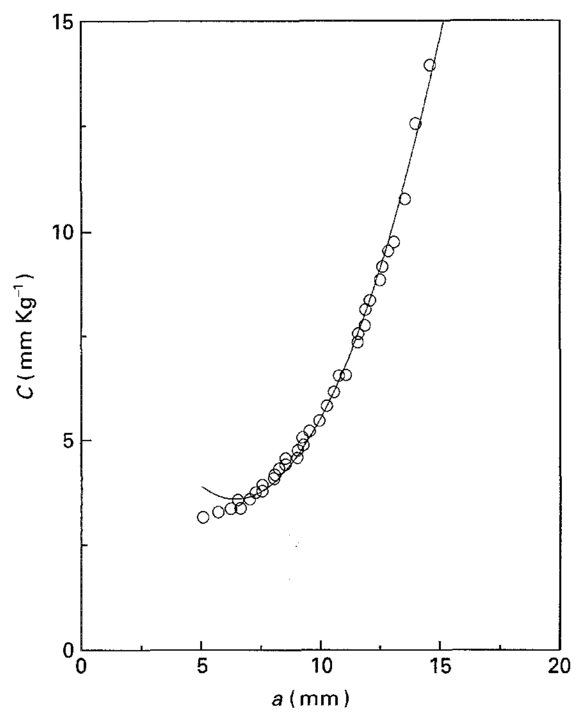

Figure 3 The measured compliance as a function of crack length and its polynomial fit. Si_3N_4, material C; $C = 0.1521\,a^2 - 1.941\,a + 9.816$.

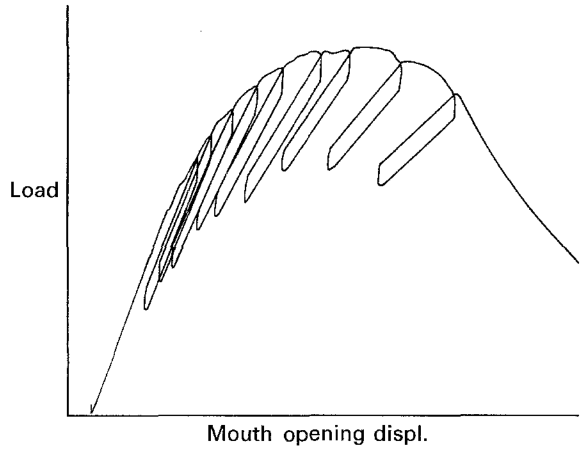

Figure 4 Load–displacement curve for Si_3N_4, material A.

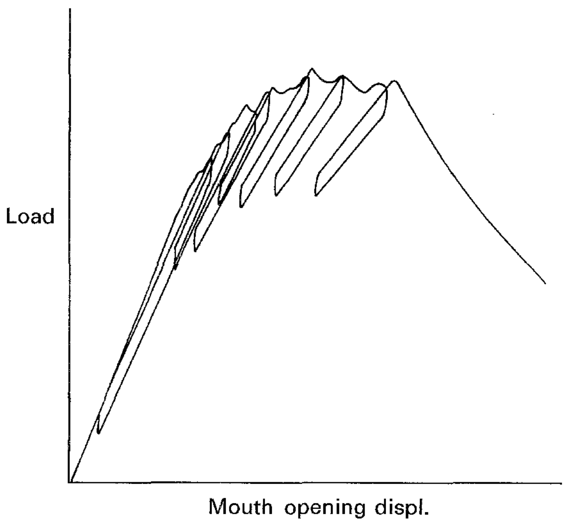

Figure 5 Load–displacement curve for SiC-whisker filled alumina.

the elastic unloading compliance. The critical value of energy release rate, G, and the critical value of the stress intensity factor, K, can then be obtained from the following expressions [15] :

$$G = 1/2\ P^2/b \cdot [\mathrm{d}C/\mathrm{d}a] \tag{3}$$

$$K = [EG/(1 - v^2)]^{0.5} \tag{4}$$

where $b = L(A - a)\ /(B - a)$ is the crack width at a given crack length a, E is the elastic modulus and v is Poisson's ratio.

A typical load–displacement curve is shown in Fig. 4 for hot pressed silicon nitride and in Fig. 5 for the polycrystalline alumina. The corresponding crack lengths are calculated by using the compliance measurements and Equation 1. The derivatives $\mathrm{d}C/\mathrm{d}a$ are calculated from Equation 2.

To compare the measured R-curve, the crack growth resistance of material C was measured using short DCB specimen [12, 13] with dimensions of 30 (length) × 25 (height) × 3 mm (thickness). A 23 mm straight notch was cut with an 800 µm thick diamond saw, and an additional 2 mm notch was cut with a ca. 180 µm diamond saw. One side of the DCB was diamond polished to 1 µm finish in order to facilitate observation of the propagating crack. A universal testing machine and travelling microscope system was used for the measurement. Crack extension, as monitored by the travelling microscope, load and crack mouth opening were recorded simultaneously. The crack growth resistance curve was calculated from the experimentally determined compliance assuming linear elastic material behaviour [15].

4. Results

The measurements of the displacement under the applied force (Fracjack opening) were very consistent. The relationship between the measurements by the devices and the plotter were linear throughout the testing range. All devices gave the same calibration factor for the fractometer plotter. To avoid scatter in the compliance measurement after each cutting a specimen was always loaded in same direction after each cutting, and the experimental set-up of the fractometer was unchanged.

The values of the fracture toughness, K_{IC}, obtained by the standard chevron-notched short-bar method, the flexural strength (MOR) values, the material properties, and a second degree polynomial fit for the compliance as a function of crack length are summarized in Table I. The values of K_{IC} measured at different crack lengths, a, for material A are shown in Fig. 6. It is clear that K_{IC} is generally constant for this material and indeed is a material's property. Figs 7 and 8 show the relationships between K_{IC} and crack length, a, for two other Si_3N_4 materials. In both cases, resistance to crack propagation increases with crack length (over 3 mm of crack extension), indicating typical R-curve behaviour. The response of material C to stress during the double cantilever beam test is shown in Fig. 9. The data in

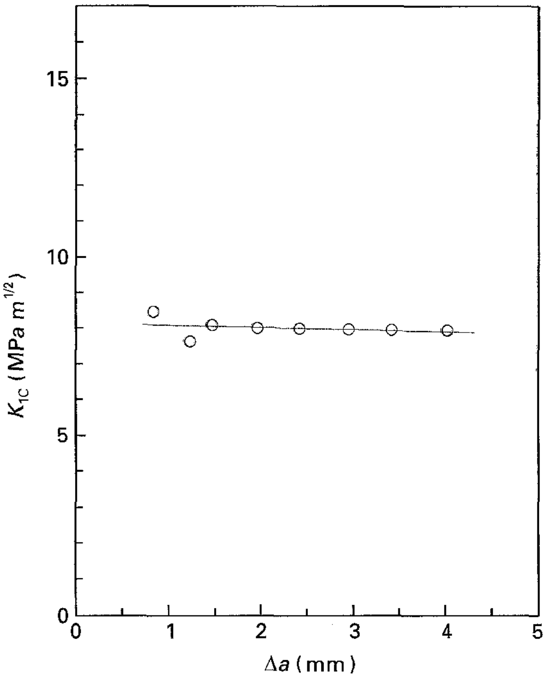

Figure 6 R-curve for Si_3N_4, material A.

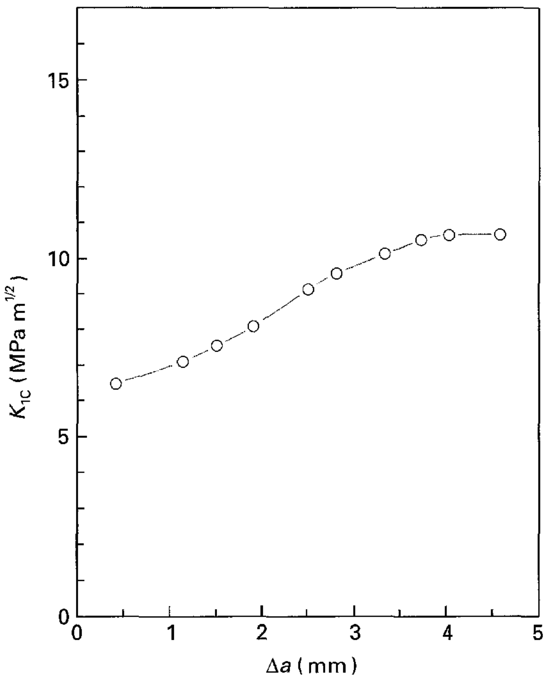

Figure 7 R-curve for Si_3N_4, material B.

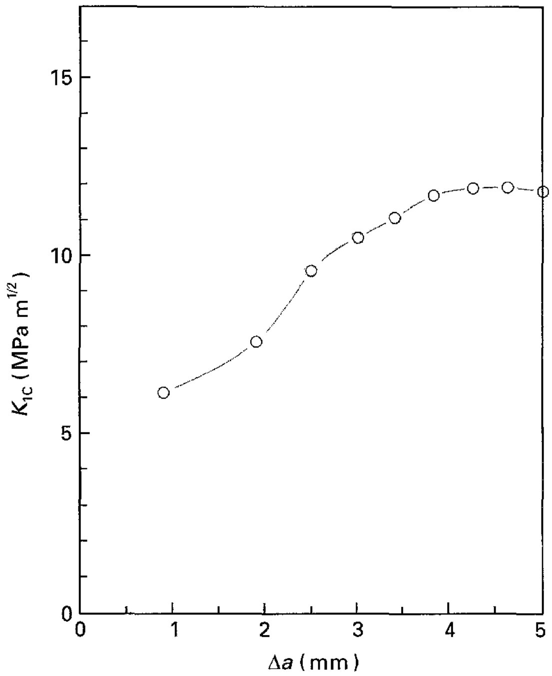

Figure 8 R-curve for Si_3N_4, material C.

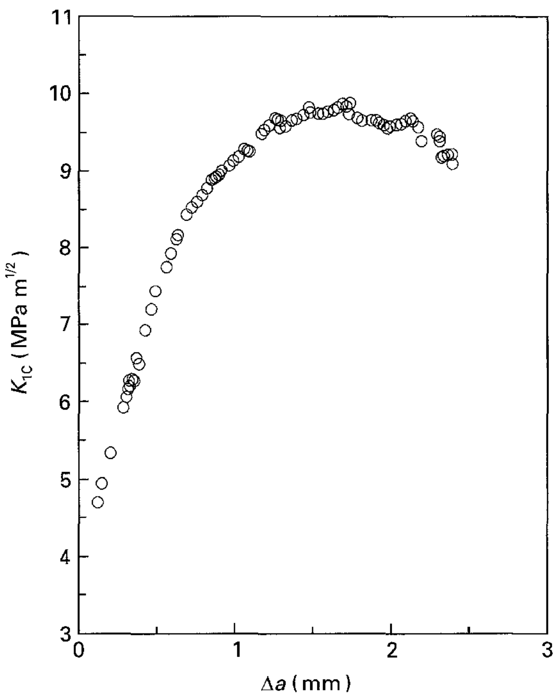

Figure 9 R-curve for material C from DCB measurements.

this figure show a similar curve as Fig. 8, exhibiting pronounced R-curve behaviour. Fig. 10 shows the R-curve of the SiC whisker-filled polycrystalline alumina that was studied extensively for crack resistance. The resistance to crack growth increases with crack length up to 3 mm. The plateau values of the crack resistance curves for all the tested materials are up to 25% higher than the fracture toughness values measured using the standard chevron-notched short-bar procedure or by three-point bending tests.

5. Discussion

For brittle materials, the chevron-notched method for determining plane-strain fracture toughness has many advantages and has been accepted in the technical community as a convenient, economical and simple method. Krause and Fuller [17] studied the fracture toughness for polymer concrete materials using various chevron-notched specimen configurations. They found that the fracture toughness depends on the specimen size and to a lesser extent on the geometry of the chevron notch. Their study indicates the presence of a rising crack growth resistance curve. Recently, Jenkins *et al.* [18] studied the fracture resistance of a SiC whisker-reinforced/polycrystalline Al_2O_3 matrix composite at different temperatures using chevron-notched and straight-notched three-point bending specimens. They found a slow and linearly

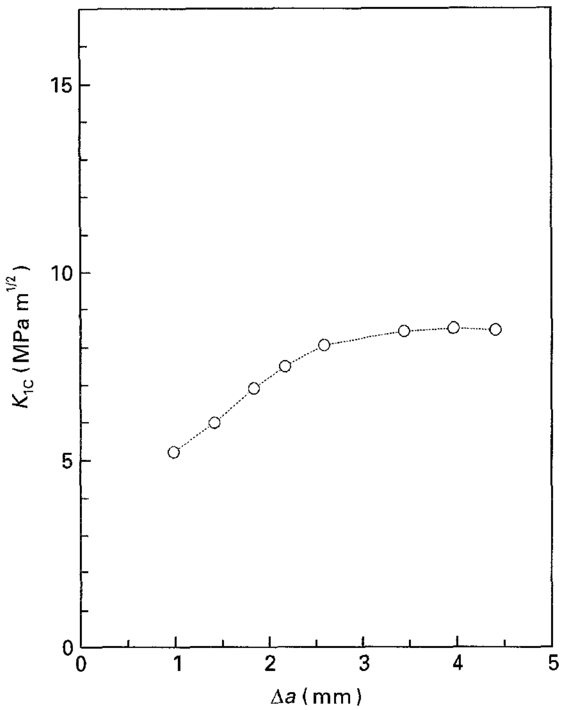

Figure 10 R-curve for SiC-whisker filled alumina.

rising resistance to crack growth at low temperatures and a substantially rising R-curve at higher temperatures.

The values of K_{IC} for the silicon nitride materials studied in the present work are in the range of 6–10.5 MPa m$^{1/2}$. These K_{IC} values are high compared with the published values for silicon nitride. Salem and Shannon [19] measured the fracture toughness of Si_3N_4 using short-bar chevron-notched specimens and found a value of K_{IC} ca. 4.7 MPa m$^{1/2}$. They observed a constant resistance to crack growth. The tested materials that have R-curve behaviour show higher fracture toughness as compared to the material that does not exhibit R-curve behaviour. However, it is clear from Figs 6–8 that the present method shows a difference in Si_3N_4 materials resistance to crack propagation. Similar R-curve behaviours were obtained by other techniques, such as direct measurement of crack growth in double-cantilever specimen (Figs 8 and 9) and studies from flexural strength of specimens with an indentation flaw [1]. The difference between two curves in the beginning of crack extension is believed to be associated with the initial crack shape. The chevron-notched specimen shows ca. 2 mm delay before there is an increase in resistance to crack propagation.

In the specimens with a high fracture toughness and resistance to crack growth, the maximum load seems to be reached after a large crack extension (possibly up to 4 mm), as shown in Fig. 4. Also, fracture toughness for materials with rising R-curves remains the same after a certain amount of crack extension. It makes sense to consider the plateau values of the crack resistance curve, similar to using the maximum load for standard method suggested by Barker [14], as the material's property.

As compared to the fracture toughness measured by the standard method, the upper values of crack resistance curve are ca. 25% higher for the present materials. A similar trend was observed by Beeches and Ingraffea [20]. Hong and Schwarzkopf [21] compared the fracture toughness of hard metals measured by the short-rod chevron-notched method. They also found that K values from the compliance method are consistently higher than those obtained from other methods such as single-edge notched beam four-point bending or microstructural indentation tests. Their explanation was that the measured crack length values in their experiments were smaller than the actual crack lengths, or alternatively the presence of residual stress resulting from loading and unloading. In the case of whisker-filled silicon nitride (non-transforming ceramics), the higher values of compliance, and therefore the lower estimates for the crack lengths, can be induced during unloading because of friction or resistance forces caused by whiskers.

As for the measuring technique used in this study, it is appropriate to mention the work of Bornhauser *et al.* [2] who studied R-curve behaviour of ceramic materials at elevated temperatures. They found that the R-data evaluated by the compliance approach and directly measured crack lengths were identical. In addition, the single specimen compliance method with partial and total unloading, and the multiple-specimen method, both resulted in rising crack resistance curves.

Steinbrech *et al.* [22] investigated the influence of specimen geometry, mean grain size and deformation velocity on the crack resistance in Al_2O_3 using three-point bending tests. They showed an increase in crack resistance with increase crack growth and found the same compliance values for saw-cut and cracks of the same lengths. They concluded, however, that there is no unique R-curve which enables characterization of the fracture behaviour of alumina materials. The SiC whisker-filled alumina used in this study showed similar patterns of behaviour (Fig. 10) with a similar a/w relation. The resistance to crack growth increases with crack extension up to 3 mm. Compared with Si_3N_4 (Figs 7 and 8), the crack extension before catastrophic failure is shorter in the case of Al_2O_3. This also can be observed from Figs 4 and 5. Relative to silicon nitrides (materials B and C), alumina has the earlier and more drastic crack jump after a certain crack extension.

Considering the importance of fracture toughness measurements, it is recommended that there are standard experimental procedures, as is the case of linear fracture mechanics, to study R-curve behaviour of ceramics materials. Since the chevron-notched short-bar test is generally accepted for measuring the critical stress intensity factor, and is easy and simple, the method described in the present paper is recommended for measuring R-curve behaviour in ceramics.

6. Conclusion

A simple method to measure the crack resistance of ceramic materials has been developed. Only two chevron-notched short-bar specimens are needed: one for calibration of crack lengths and elastic compliances, and a second one for the fracture behaviour measurements. Since the chevron-notched short-bar test is generally accepted for measuring the critical stress intensity factor, and is easy, economical and simple to implement, the method described in the present paper is recommended as a standard method of measuring R-curve behaviour in ceramics. In addition, this method can be extended to a single specimen test method to measure the R-curve behaviour using the elastic compliance measurement of crack length and load cycling.

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