Crack resistance curves of alumina and zirconia at room temperature

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Ceramic three-point bend specimens were pre-cracked in a displacement-controlled test in air at room temperature to form sharp cracks of different lengths. Critical stress intensity factors \(K_{IC}\) were then measured as a function of sharp crack length in a fast-fracture, load-controlled test. Crack resistance curves \(K_{IC} \) against crack length were determined for three commercially pure aluminas of different grain size, a debased alumina containing a glassy phase, and a partially stabilized zirconia (PSZ) material. The crack resistance curves proved to be flat for the finer-grained and the debased alumina. A steeply rising crack resistance curve was, however, observed for a pure coarse-grained alumina material which is explained by friction effects of the cracked microstructure behind the measured crack front. The effect is influenced by the test procedure itself. Though crack branching takes place the crack resistance curve of PSZ is completely flat, which is attributed to fast fracture testing where only the most dangerous flaw is activated.

1. Introduction
Ceramic materials are normally completely brittle at room temperature. The brittleness implies that linear elastic fracture mechanics is best suited for fracture toughness measurements and subcritical crack extension characterization. It may be assumed that the fracture toughness value turns out to be a material constant independent of crack length.

In contradiction to these presumptions it was found in an earlier paper [1] that fracture toughness data measured with sharp cracks showed a strong dependence on crack length, in which the fracture toughness or crack resistance increases with increasing crack length.

The load–displacement records of the experiments leading to this dependency were performed in a more or less controlled manner, and showed increasingly non-linear behaviour with increasing sharp, natural crack lengths [2].

In analogy with plasticity reactions with metallic materials the effect was first explained by a “process zone” of microcracking ahead of the actual crack tip, the size of the microcracks being typically of the order of the structural element (i.e. the grain size). The rising crack resistance curve was then explained by an enlargement of this “process zone” with increasing crack length, in complete analogy with the increase in the size of the plastic zone found in metallic materials.

However, as a “process zone” could not be detected [2] it was then alternatively argued [1, 2] that friction effects or adhesive forces at the crack surfaces behind the actual crack tip cause the unusual behaviour.

These arguments were supported by measurements with narrow notches which guarantee traction-free crack surfaces. It could be proved that crack resistance curves measured with narrow notches are completely flat [1] (Fig. 1). Meanwhile similar behaviour was found by other authors [3–6] and a broad discussion has developed about the pre-conditions and the nature of this strange phenomenon.

The existence or non-existence of a rising crack resistance curve has very important practical consequences: the question is whether a single fracture toughness parameter is sufficient to characterize catastrophic failure or if a crack resistance curve is necessary. As mentioned above, the fracture toughness data (as a function of crack length) were evaluated from more or less subcritical, displacement-controlled tests which can cause crack branching and microcrack formation. If fracture toughness should characterize the catastrophic failure in a linear elastic test, then rapid, load-controlled experiments should be performed, as in the present work. Nevertheless, sharp cracks of distinct lengths have to be introduced by a displacement-controlled subcritical test procedure.

For the experiments a variety of materials were chosen with different tendencies towards secondary crack formation and microcracking.

2. Experimental procedure
The \(K_c\) curves were determined as listed below.

2.1. The precracking process
Three-point bend specimens 7 mm \(\times\) 2.5 mm \(\times\)
40 mm in size (span width 30 mm) were notched to a normalized notch depth of 0.2 and then pre-cracked to the desired natural crack length in a displacement-controlled test in air at room temperature. The displacement rate of 1 μm min⁻¹ promotes crack branching and microcracking. After the desired sharp crack length was reached the specimens were unloaded. The displacement-controlled test was made possible by using an extremely stiff bending device with SiC supports together with a stiff testing machine of very low compliance $C_m = 0.0109 \pm 0.0003$ μm N⁻¹. The displacement was directly measured and controlled by a linear voltage differential transformer in contact with the lower specimen surface.

2.2. Crack length measurement
As there is no macroscopic blunting effect with ceramic materials the actual crack tip is hard to detect. The cracked surfaces are close together, which enhances friction effects behind the crack tip. Therefore special attention has to be given to measuring crack lengths and crack elongation during and after the pre-cracking process.

Three methods were used:

(a) Side-light technique (Fig. 2). The crack lengths were measured after pre-cracking and unloading in a microscope on both polished sides of the specimen.

(b) Post-test investigation (Fig. 3). After the fast fracture test the cracked surfaces were investigated. A transition from inter-granular to trans-granular fracture was detected which separates the precracking region from the load-controlled, fast-fracture toughness test region. The pre-crack front was always found to be straight.

(c) Direct, visual observation (Fig. 4). The pre-cracking process was directly observed with a traveling microscope at a magnification of × 250 [7]. With direct observation the crack length could be measured while the specimen was under load. This method facilitates detection of the crack tip. The kinetics of crack growth, secondary crack formation, crack branching and microcracking may also be observed using this technique.

The specimens were polished but not etched, as the width of the grain boundaries is of the same dimension as the distance of the cracked surfaces near the crack tip. Table I shows crack length values for two examples, measured using the three different methods. There is no significant difference between the results of the three evaluation methods. The mean scatter in crack length was found to be about 3%.

2.3. The crack resistance curve
The pre-cracked specimens were rapidly loaded to fracture in a load-controlled test at a loading rate of 100 N sec⁻¹. Thus subcritical crack extension was avoided and a true critical test was performed. The fracture toughness $K_{IC}$ was then calculated using the linear elastic equation

$$K_{IC} = \frac{1}{2} \frac{P_c S}{BW} a_c^{1/2} Y(a/W)$$

where $P_c = $ critical load, $S = $ span, $B = $ specimen thickness, $W = $ specimen width, $a_c = $ critical total crack length (notch length + sharp crack length), $Y(a/W) = $ correction function [8].

The $K_{IC}$ data are plotted against the total normalized crack lengths $a/W$, producing the $K_R$ curve. Equation 1 implies that the cracked surfaces are stress-free or traction-free, where $a_c$ is the crack length measured. Equation 1 does not account for friction effects and adhesive forces behind the measured crack tip. Traction-free surfaces are certainly guaranteed if narrow notches instead of sharp, natural cracks are used.

3. Materials
Four alumina materials of different grain size and purity were used. Three were commercially pure; the fourth, Al₂O₃-S, was of a debased quality with a glassy phase content of about 3 wt % SiO₂ (Table II).

It is well known that the internal stresses of alumina materials increase with increasing grain size. Therefore microcracking and secondary crack formation, and thus the capability for energy dissipation