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Engineering Fracture Mechanics 71 (2004) 2263–2269
Engineering Fracture Mechanics
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A threshold stress intensity factor at the onset of stable crack extension of Knoop indentation cracks

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Received 30 July 2001; received in revised form 17 February 2004; accepted 19 February 2004

Abstract

Observation of the stable growth of indentation cracks in controlled bending tests is an attractive tool to study the *R*-curve behaviour of ceramic materials. In some crack growth experiments deviations from the ideal behaviour were observed—i.e. the cracks did not grow immediately upon application of the external load, but only after a certain applied stress is exceeded. The existence of such a threshold stress intensity for the onset of stable crack growth can be described by a disbalance of the residual stress intensity at the indentation crack tip and the fracture toughness after completion of the indentation cycle. A fracture mechanical description of this phenomenon is presented. The consequences which arise for the evaluation of crack growth data are explained by means of measured crack extension curves of Knoop indentation cracks in silicon nitride.

Keywords: Knoop indentation cracks; Stable crack growth; Fracture toughness; *R*-curve

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Abstract

Observation of the stable growth of indentation cracks in controlled bending tests is an attractive tool to study the R-curve behaviour of ceramic materials. In some crack growth experiments deviations from the ideal behaviour were observed – i.e. the cracks did not grow immediately upon application of the external load, but only after a certain applied stress is exceeded. The existence of such a threshold stress intensity for the onset of stable crack growth can be described by a disbalance of the residual stress intensity at the indentation crack tip and the fracture toughness after completion of the indentation cycle. A fracture mechanical description of this phenomenon is presented. The consequences which arise for the evaluation of crack growth data are explained by means of measured crack extension curves of Knoop indentation cracks in silicon nitride.

1 INTRODUCTION

Indentation cracks that form during the impression of a sharp indenter into a brittle material are an useful tool for fracture mechanic studies. They are simple to generate and because they are sharp and rather short, they are believed to behave like natural cracks. Therefore they are widely used for the determination of the indented material's fracture toughness [1].

Furthermore they are of special interest for the measurement of the R-curve: introduced into the tensile surface of flexural specimens they may act as starter cracks for crack growth experiments. The residual stress field which is pertinent after the indentation as a consequence of an irreversible plastic deformation of the indented material stabilises the crack as it is driven by a superimposed bending stress [2, 3]. Such crack growth experiments on Vickers indentation cracks were performed by several authors [4 - 8]. Measurements of the crack length as a function of the applied bending load can be exploited in different ways to experimentally determine the residual stress parameter.

Crack growth experiments on Knoop indentation cracks were performed in [8]. In these experiments it was observed, that indentation cracks do not grow at low applied stresses, the

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crack extension curves show an offset. The expected crack extension occurs only if a threshold stress intensity is exceeded. In this paper a similar behaviour for Knoop as well a Vickers cracks in silicon nitride together with a possible explanation is discussed.

2 THEORETICAL CONSIDERATIONS

It is usually assumed that indentation cracks are generated and grow as contained subsurface cracks below the indenter tip during the loading cycle. Upon unloading they close up below the surface but simultaneously open at the surface as the elastic constraint is removed. As the residual stress field becomes dominant towards the end of the indentation cycle they grow further to form nearly half-penny cracks [1].

We assume here according to a common interpretation [9] that indentation cracks form at the surface and grow during the loading half-cycle of the indentation process.

Usually indentation cracks are described as half-penny surface cracks point-loaded at the centre [1]. The corresponding stress intensity factor K_{ind} is given by:

$$K_{ind} = \kappa \frac{P}{c^{3/2}} \quad , \tag{1}$$

where P is the indentation load and c the actual crack size. The coefficient κ depends on the indenter shape (Knoop, Vickers) and the elastic/plastic behaviour of the indented material.

During an indentation test the load increases up to a maximum value. The crack grows if the stress intensity factor K_{ind} reaches the fracture toughness K_{Ic}. In this instance the crack size is defined by the condition

$$K_{ind} = K_{Ic} \quad . \tag{2a}$$

In the presence of a R-curve behaviour the crack growth resistance K_R depends on c and condition (2a) reads

$$K_{ind} = K_R(c) \quad . \tag{2b}$$

Under fully applied indentation load we find the related crack size c_0 from

$$K_R(c_0) = \kappa \frac{P}{c_0^{3/2}} \quad . \tag{3}$$

After removal of the indentation load, some residual stresses remain and the crack driving stress field is only partially reduced. Due to the strongly deformed and damaged zone directly beneath the indenter tip the crack is wedged open and consequently the crack size remains c_0. The residual stress intensity factor for this situation, K_{res} is

$$K_{res} = \chi \frac{P}{c_0^{3/2}} \quad . \tag{4}$$

In analogy to the coefficient κ, the factor χ depends on the indenter geometry and the elastic/plastic properties of the indented material. In this partially unloaded state the stress intensity factor is reduced from the maximum value under load $K_R(c_0)$, eq. (3), to

$$K_{res} = \alpha K_R(c_0), \qquad \alpha = \chi/\kappa < 1 \quad . \tag{5}$$

If a suitable indentation crack-damaged specimen is additionally loaded by an external stress (e.g. a bending stress) an additional stress intensity factor K_{appl}, is caused. In general, the total stress intensity factor K_{total} is given as the sum of the residual and the applied stress intensity factors,

$$K_{total} = K_{res} + K_{appl} = \chi \frac{P}{c^{3/2}} + \sigma Y \sqrt{c} \quad , \tag{6}$$

(Y = geometric function). A possible interaction between the residual and the applied stress intensity factors (as outlined in [10, 11]) is neglected here.
Under an increasing applied stress intensity factor K_{appl}, it holds:

$$K_R(c) = \chi \frac{P}{c^{3/2}} + K_{appl}(c) \quad . \tag{7}$$

The condition for stable crack extension can be written in terms of eqs. (4) and (5) as

$$K_R(c) = \alpha \left(\frac{c_0}{c} \right)^{3/2} K_R(c_0) + K_{appl}(c) \quad . \tag{8}$$

Stable crack growth is thus only possible if

$$K_{appl} \geq K_R(c) - K_R(c_0) = (1-\alpha) K_R(c_0) = K_{appl,0} \quad . \tag{9}$$

To provide enough energy for the existing crack c_0 to grow, the externally applied stress intensity K_{appl} has to exceed the threshold stress intensity $K_{\mathrm{appl},\,0}$ (identical to $(1-\alpha)\, K_R(c_0)$) relieved in the unloading part of the indentation cycle.

3 EXPERIMENTAL

Crack growth experiments were performed on a commercial MgO-doped, gas pressure sintered silicon nitride manufactured by ESK, Kempten.
Knoop cracks were introduced into mirror polished tensile surfaces of standard bend bars (3 × 4 × 45 mm^3) with different loads (49N, 98N, 196N and 294N). The long axis of the indentation was aligned perpendicular to the prospective tensile stress. The specimens were then stepwise loaded to increasing stress levels in 4-point bending mode (40/20 mm fixture). After each stress level the crack lengths were measured using an optical microscope. The procedure was repeated until failure occurred at the indentation crack. To calculate the geometry factor Y of the cracks it was necessary to observe the change of the shape of the indentation cracks during stable growth. Some cracks were decorated with lead acetate after a certain stress level was reached. These specimens were then broken to reveal the shape of the crack at that particular stress level. A function $Y(c)$ was determined for each indentation load. Details on this procedure can be found elsewhere [12].
To gain information on the R-curve, Fig. 1., the fracture toughness was determined at different critical crack lengths with SEVN-B-specimens [13], SCF- [14] and SEP-B [15] tests.
A rough description of the data in Fig. 1 is given by

$$K_R \cong K_{I0} + A \left\{ 1 - \exp\left[-\frac{c - c_0}{B} \right] \right\} \tag{10}$$

with K_{I0} = 5.75 MPa√m, A = 1.55 MPa√m, and B = 0.2 mm.

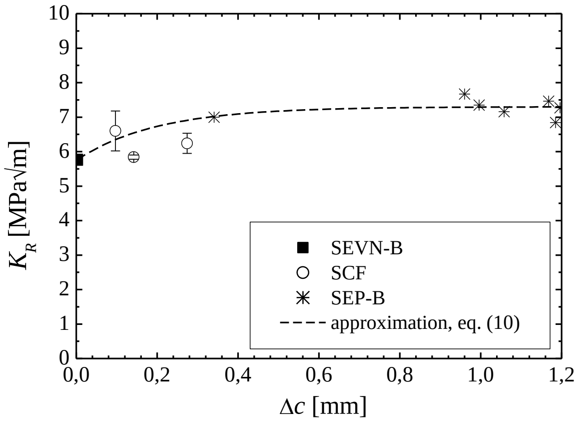

Fig. 1: R-curve of the investigated silicon nitride.

4 RESULTS AND DISCUSSION

4.1 Estimation of the residual stress intensity factor K_{res}

The crack extension curves for Knoop cracks are shown in Fig. 2a where $\sigma\sqrt{c}$ is plotted versus $c^{-3/2}$. By replotting the data as $K_{appl} = f(P/c^{3/2})$ according to an idea of BLEISE & STEINBRECH [6] the plot of Fig. 2b is obtained. To calculate K_{appl}, the geometry factor Y of the actual crack shape at the applied stress and the formula proposed by NEWMAN & RAJU [16] were used.

a) b)

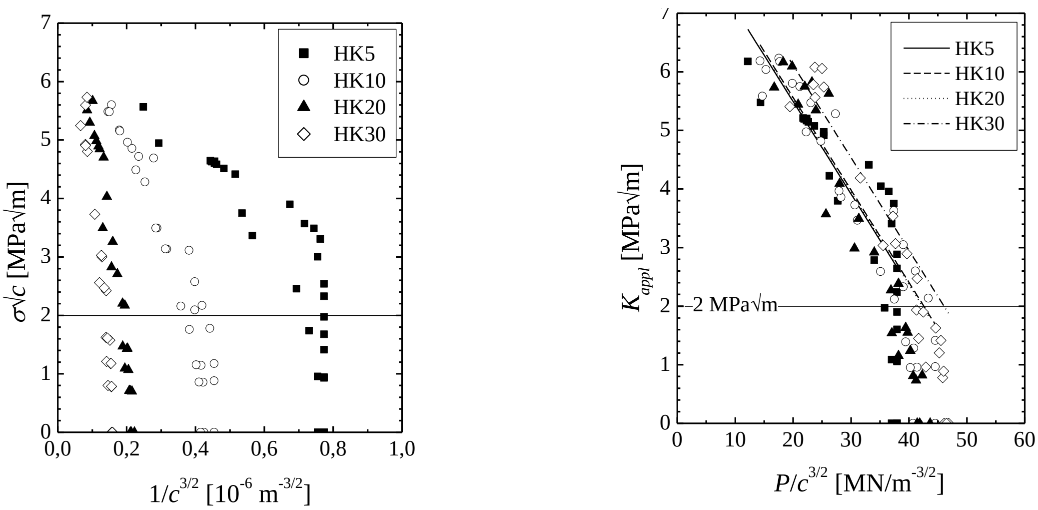

Fig. 2: Crack extension curves of Knoop indentation cracks; a) P = 294 N (open diamond squares), P = 196 N (solid triangles), P = 98 N (open circles), P = 49 N (squares); b) replotted data.

For K_{appl} < 2 MPa√m no crack extension is found, what yields in the vertical part of the curve. Only a slight influence of the initial crack size c_0 on the initial value of $P/c^{3/2}$ can be made out for the data with K_{appl} < 2 MPa√m (Fig. 3). This value is almost independent of the indentation load P and consequently on the initial crack size c_0. Above K_{appl} = 2 MPa√m stable crack propagation occurs.

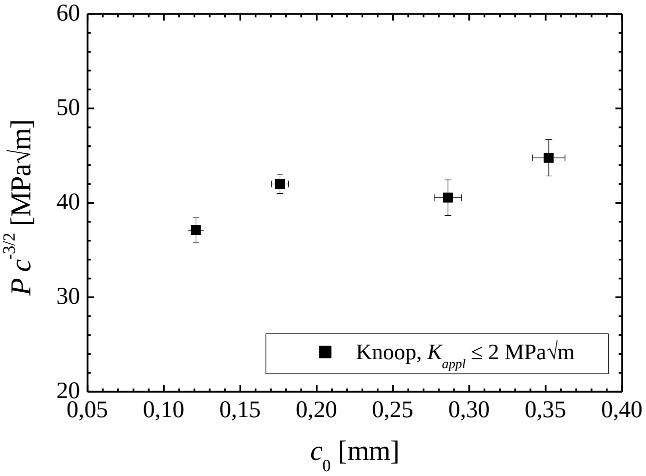

Fig. 3: Initial value of the quantity $P/c^{3/2}$ versus the initial indentation crack size c_0 for Knoop cracks.

The residual stress term can determined from a plot K_{appl} versus $P/c^{3/2}$ [6],

$$K_{appl} = K_R - \chi \frac{P}{c^{3/2}} \quad . \tag{11}$$

In [6] the authors determined χ from the negative slope of the obtained straight line. This evaluation is of course only a rough estimation and would only be correct for materials without an R-curve effect. Following this evaluation procedure, a least square fit for all data with $K_{appl} > 2$ MPa√m of Fig. 2b yields $\chi = 0.16$. Introducing χ into eq. (7) leads to $K_R(c_0) \cong 8.8$ MPa√m for the stress intensity factor at the onset of stable crack propagation. Compared to the R-curve shown in Fig. 1 this is a rather high value. This fact indicates that this procedure is not suitable to evaluate the coefficient χ. This disagreement can easily be understood. By introducing eq. (10) into (11) we obtain

$$K_{appl} = K_{I0} + A\left\{1 - \exp\left[-\frac{x^{-2/3} - c_0}{B}\right]\right\} - \chi P x\,, \qquad x = 1/c^{3/2} \tag{12}$$

This is a relative complicated dependence between K_{appl} and $1/c^{3/2}$ and no longer a straight line relation. Consequently, a linear fit of K_{appl} versus $1/c^{3/2}$ makes no sense.
Following the theoretical consideration of section 2, a different idea to determine the residual stress term is based on the kink in the crack extension curve, Fig. 2b and on eq. (9). Using $K_{appl,\,0}$ = 2 MPa√m and $K_R(c_0) \cong 7$ MPa√m from the R-curve in Fig. 1, we get

$$\alpha \cong \frac{5}{7} \quad , \tag{13}$$

and consequently,

$$K_{res}(c_0) \cong 5\,\mathrm{MPa}\sqrt{\mathrm{m}} \quad . \tag{14}$$

With eq. (4) the value for the residual stress parameter becomes $\chi = 0.11$.

4.2 Discussion

The effect, that indentation cracks do not start to grow immediately upon applying a bending stress has also been observed for Vickers indentation cracks in different ceramics, for example in stabilised zirconias [5], in alumina [6] and in silicon nitride [7]. It also occurred in soda-lime glass [17] in biaxial bending. In the cases, where the residual stress parameter χ was evaluated from the slope of these crack extension curves [6, 7] and subsequently used to calculate the fracture toughness, these values turned out to be close to plateau values of R-curves measured with methods that use "long cracks", i.e. higher than expected.

The offset in the crack growth curves has been explained by a relaxation of the residual stresses in the period between the indentation and the bend testing [5]. KURTH & STEINBRECH [18] annealed indented specimens before the bend test to partially release the residual stresses. They showed that such a relaxation leads indeed to a threshold for the applied stress intensity before stable crack extension occurs. It is however not clear which post-indentation processes may lead to such a pronounced stress relaxation at room temperature. The explanation of the offset by a post-indentation stress relaxation is formally identical with the idea presented in this work. It can be expressed in a form similar to eq. (5).

The example shown in the previous section 4.1 clarifies the consequences of the existence of threshold stress intensity (offset) for stable growth of indentation cracks for the use of such data for the evaluation of R-curves. Evaluation of the residual stress parameter χ solely from the slope of the data results in erroneous results for the R-Kurve $K_R(c)$. A correct value for χ can be determined if additional information on the toughness at the as-indented crack length, $K_R(c_0)$, is provided via an independent measurement. On one hand, this limits the use of stable indentation crack growth data for R-curve measurements, but on the other hand it also provides a means to calibrate the unknown parameter χ, one of the major uncertainties pertinent to indentation crack methods.

5 CONCLUSION

The threshold stress intensity often observed for stable crack growth of Knoop indentation cracks can be explained by a disbalance of the residual stresses and the crack size remanent after completion of the indentation cycle. The residual stress intensity at the crack tip is smaller than the fracture toughness. If such an indentation crack is subjected to an external loading, the difference between residual stress intensity and the fracture toughness has to be equalised by the applied stress intensity before stable crack growth may take place. For a given material this necessary threshold stress intensity does strongly depend on the shape of the indenter.

As a consequence of this threshold stress intensity it is not possible to determine the residual stress parameter of indentation cracks correctly solely from the slope of crack extension curves. The true value of χ can only be found if additional information on the fracture toughness is provided. This fact complicates the use of crack growth data of indentation cracks for the measurement of R-curves.

6 REFERENCES

[1] Lawn, B.: *Fracture of Brittle Materials*, Cambridge University Press, Cambridge (1993)

[2] Lawn, B.R., Evans, A.G., Marshall, D.B.: "Elastic/Plastic Indentation Damage in Ceramics: The Median/Radial Crack System", *J. Am. Ceram. Soc*. **63** [9-10] (1980) 574-581

[3] Marshall, D.B., Lawn, B.R.: "Residual Stress Effects in Sharp Contact Cracking: I, Indentation Fracture Mechanics", *J. Mat. Sci.* **14** (1979) 2001-2012

[4] Smith, S.M., Scattergood, R.O.: "Crack-Shape Effects for Indentation Fracture Toughness Measurements", *J. Am. Ceram. Soc.* **75** [2] (1992) 305-315

[5] Dransmann, G.W., Steinbrech, R.W., Pajares, A. et al.: "Indentation Studies on Y_2O_3-stabilized ZrO_2: I, Toughness Determination from Stable Growth of Indentation-Induced Cracks", *J. Am. Ceram. Soc.* **77** [5] (1994) 1194-1201

[6] Bleise, D., Steinbrech, R.W.: "Flat R-Curve from Stable Propagation of Indentation Cracks in Coarse-Grained Alumina", *J. Am. Ceram. Soc.* **77** [2] (1994) 315-322

[7] Kurth, R., Lawlor, B.F., Stockmann, Y.J., Steinbrech, R.W.: "Toughness Determination from Stable Crack Growth of Long and Short Cracks in Si_3N_4 Ceramics", *Fracture Mechanics of Ceramics* **11** (1996) 151-162

[8] Lube, T.: A Knoop Indentation Method for R-Curve Determination, to be published in: *Fracture Mechanics of Ceramics*, Proc. of 7th FMC, 20 – 22 July 1999, Moscow

[9] Ostojic, P., McPherson, R.: A Review of Indentation Fracture Theory: Its Development, Principles and Limitations, *Int. J. Fract.* **33** (1987) 297-312

[10] Fett, T., Munz, D.: "Knoop-Indentations As Surface Flaws For Subcritical Crack Growth Measurements", *in:* H. Matzke (Ed.) European Applied Research Report (1986) 1183-1196

[11] Fett, T.: "An Analysis of the Residual Stress Intensity Factor of Vickers Indentation Cracks", *Eng. Fract. Mech.* **52** [4] (1995) 773-776

[12] Lube, T.: "Investigations on the Stable Crack Growth of Indentation Cracks": *in* J.G. Heinrich & F. Aldinger (Eds.): Ceramic Materials and Components for Engines, Wiley-VCH, Weinheim 2000, ISBN 3-527-30416-9, 121-126

[13] Damani, R.J., Schuster, C., Danzer R.: "Polished Notch Modification of SENB-S Fracture Toughness Testing" *J. Eur. Ceram. Soc.* **17** [14] (1997) 1685-1689

[14] ASTM C1421-99: "Standard Test Method for the Detemination of Fracture Toughness of Advanced Ceramic Materials at Ambient Temperature" (1999)

[15] Pancheri, P., Bosetti, P., Dal Maschio, R. Sglavo, V.M.: "Production of Sharp Cracks in Ceramic Materials by Three-Point Bending of Sandwiched Specimens", *Eng. Fract. Mech.* **59** [4] (1998) 447-456

[16] Newman, J.C., Raju, I.S.: "An Empirical Stress-Intensity Factor Equation for the Surface Crack", *Eng. Fract. Mech.* 15 [1-2] (1981) 185-192

[17] Marshall, D.B., Lawn, B.R., Chantikul, P.: "Residual Stress Effects in Sharp Contact Cracking: II, Strength Degradation", *J. Mat. Sci.* **14** (1979) 2225-2235

[18] Kurth, R., Steinbrech, R.W.: Influence of Annealing on Residual Stress Intensity of Indentation Cracks in Si_3N_4, *in:* Meriani, S., Sergo, V. (Eds.), 4th Euro Ceramics, Vol. 3, Gruppo Editoriale Faenza Editrice, Faenza (1995)