# On subcritical crack growth in ceramics as influenced by grain size and energy-dissipative mechanisms

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A model is presented concerning the possibility that an unstable crack becomes stabilized by the initiation of some energy-dissipative mechanism if the stress intensity reaches a threshold value  $K_{I}^{D}$ . Depending on the toughness and the grain size distribution of the material, different crack propagation modes may occur. For sintered alumina, measurements of the fracture induced photon emission can be explained in terms of this model, substantiating in this way the assumption that microcracking is the dominating form of energy dissipation in this single phase ceramic material.

# 1. Introduction

Fractographic determination of the flaw size C can be used to calculate the fracture surface energy  $\gamma$ from the usual Griffith equation  $\sigma_f = Z(2E\gamma/C)^{1/2}$ , where  $\sigma_{f}$  is the fracture strength, E is Young's modulus, and Z is a geometry factor. Measuring  $\sigma_{\rm f}$  and the initial flaw size  $C_0$  for several polycrystalline ceramic materials Rice et al. [1, 2] found that the apparent fracture energy  $\gamma$ increases with increasing flaw size  $C_0$  and decreasing grain size D. The effect was attributed to a transition of  $\gamma$  from the (lower) single crystal value  $\gamma_{sc}$  to  $\gamma_{pc}$ , the latter being characteristic for the polycrystalline material. For non-cubic materials it was shown that residual stresses can also be responsible for the variation of  $\gamma$  with the flaw and grain sizes [2]. In some cases the C/D-dependence of  $\gamma$  was also obtained by using the double cantilever beam (DCB) measuring technique.

However, Dalgleish *et al.* [3] have shown that at least for alumina such an effect might be due to the large extent of subcritical crack growth in the DCB specimens. Therefore it must be expected that the fractographic procedure too may lead to unreal variations of  $\gamma$  with C or D if subcritical crack growth is not taken into account, i.e. if the fracture strength is combined with the initial flaw size  $C_0$  instead of  $C_f$ , the crack length at macroscopic instability. This point of view is supported by results of Singh *et al.* [4] who demonstrated that, having two levels of fracture energy  $\gamma_0$  and  $\gamma_1$  with  $\gamma_1/\gamma_0 \gtrsim 5$  we generally must expect  $\gamma = \gamma_1$  if subcritical crack growth occurs. Similar results have been obtained by Kirchner and Ragosta [5]. Taking into account a relation between the stress intensity  $K_I$  and the velocity of quick subcritical crack growth, they have shown that in most cases  $\gamma$  is determined by the polycrystalline value and that  $\gamma = \gamma_{sc}$  is possible only for grain sizes exceeding 50 to  $100 \,\mu\text{m}$  and stressing rates as high as  $10^2$  to  $10^4 \,\text{MPa sec}^{-1}$ .

The above considerations demonstrate that subcritical crack growth has to be taken into account in order to understand the macroscopic fracture behaviour. Nevertheless, locally  $\gamma = \gamma_{sc}$ will be a reasonable assumption for the onset of crack propagation at  $C = C_0$  with a stress intensity  $K_{Ic}^0$  which is considerably below the (macroscopic) critical value  $K_{Ic}$ . From a macroscopic point of view this first stage of crack propagation usually belongs to the subcritical region; on the microscale, however, a preliminary instability may occur. So the question arises whether or not the running crack can be stabilized again. Singh *et al.* [4], Virkar *et al.* [6] and Pompe *et al.* [7, 8] analysed this problem analytically assuming a local fracture surface energy dependent on the crack length.

In the present paper we also investigate cracks which macroscopically are propagating in the subcritical range and which may be stabilized after a first microscopic instability. In particular, we assume that stabilization may be effected by certain toughening mechanisms (especially energy dissipation by microcracking) which become active when the stress intensity  $K_{\rm I}$  exceeds some threshold value  $K_{\rm I}^{\rm D}$ . The validity of this theoretical concept has been confirmed by certain experiments. Especially we have considered alumina ceramics where microcracking may lead to an increasing fracture toughness. In order to analyse the different stages of crack extension the fracture induced photon emission has been utilized.

# 2. Theory

As it is now widely accepted, a crack or flaw in a ceramic material under load may initiate the formation of an energy-dissipative zone at its tip, which under certain circumstances causes an increase in the toughness of the material. For example stress induced phase transformation in partially stabilized zirconia [9, 10] or microcracking in alumina containing  $ZrO_2$ -particles [11] may cause energy dissipation. In other polycrystalline structures without such possibilities "usual" processes as twinning, formation of river patterns, deflection of cracks at grain boundaries, and branching may also result in a critical stress intensity  $K_{Ie}$  which is higher than that of the single crystal.

Now let  $r_{\rm D}$  denote the size of the zone where energy dissipation takes place ( $r_{\rm D}$  is measured perpendicular to the crack plane, i.e. the distance between the outer boundaries of the zone is  $2r_{\rm D}$ ). Different theoretical considerations [12–14] have resulted in the following expression for  $r_{\rm D}$ :

$$r_{\rm D} = \frac{1}{3} \left( \frac{K_{\rm I}}{\sigma_{\rm c}} \right)^2 \tag{1}$$

where  $\sigma_{\rm c}$  is the critical stress at which energy dissipation starts and  $K_{\rm I}$  is the applied stress intensity. The prefactor, of course, may slightly differ from 1/3 depending on the particular energy-dissipative process. Obviously, the idea of a toughness-increasing dissipation zone makes sense only if its size is larger than a certain structural length describing the distance between the dissipating elements (microcracks, transforming particles). In our case of a single phase ceramic this structural length is given by the grain size D. Therefore, an increase in toughness due to energy dissipation requires  $r_D$  to be larger than a critical value, say nD, where n depends on the particular microstructure ( $n \approx 1$  to 4). Using Equation 1 we immediately arrive at a corresponding value for  $K_I$  which we denote by  $K_I^D$ :

$$K_{\rm I}^{\rm D} = \sigma_{\rm c} (3nD)^{1/2}.$$
 (2)

The characteristic  $K_{\rm I}^{\rm D}$  acts as a threshold value for the onset of energy dissipation. Let us denote the two levels of specific fracture surface work by  $\gamma_0$  and  $\gamma_1$ , where  $\gamma_0$  is the reference value without energy dissipation and  $\gamma_1$  is the governing quantity when energy dissipation takes place. Furthermore we assume a step-like change at  $K_{\rm I} = K_{\rm I}^{\rm D}$ :

$$\gamma = \begin{cases} \gamma_0 & \text{for } K_{\mathrm{I}} < K_{\mathrm{I}}^{\mathrm{D}} \\ \gamma_1 & \text{for } K_{\mathrm{I}} \ge K_{\mathrm{I}}^{\mathrm{D}} \end{cases}$$
(3)

This approximation seems to be appropriate in order to discuss the physical effects caused by such a transition of  $\gamma$  (the more complicated case of a smooth transition has been dealt with by Singh *et al.* [4] and Pompe *et al.* [7]). It is useful to define critical stress intensities  $K_{1c}^0$  and  $K_{1c}^1$  as derived from  $\gamma_0$  and  $\gamma_1$  by the usual relations

$$K_{\rm Ic}^0 = (2E\gamma_0)^{1/2}, \quad K_{\rm Ic}^1 = (2E\gamma_1)^{1/2},$$
 (4)

(in this paper we shall not differentiate between plane stress and plane strain conditions, hence we neglect the difference between E and  $E/(1-\nu^2)$ . where  $\nu$  is Poisson's ratio).

In the following we suppose  $\gamma_1 > \gamma_0$ , i.e. energy dissipation increases the toughness. Actually  $\gamma_0$ and  $\gamma_1$  depend on numerous microstructural parameters (e.g. [13]) but these dependencies will not be a subject of this paper. Rather we shall consider  $\gamma_0$  and  $\gamma_1$  as material properties given by theory or experiment. Starting from this position we shall investigate which of the two levels of  $\gamma$ determines the macroscopic fracture toughness  $K_{\rm Ic}$ . In particular we shall consider two questions: 1. Crack propagation starts at an applied stress  $K_{\rm I} = K_{\rm Ic}^0 < K_{\rm I}^{\rm D}$ . Subsequently intensity  $K_{I}$ increases due to the elongation of the crack. Can the crack be stabilized again by the onset of the energy-dissipative process? 2. If we compare two ceramic microstructures having different grain size distributions, how is the threshold stress intensity  $K_{\rm I}^{\rm D}$  influenced by the grain sizes? Let us start with the first problem. As in [4] we make use of the

energy balance

$$-\Delta P = \Delta W_{\gamma} \tag{5}$$

i.e. the crack can only be arrested if the elastic energy  $-\Delta P$  released during crack extension equals the fracture surface work  $\Delta W_{\gamma}$  consumed in this process. Since Equation 5 neglects the possible dissipation of kinetic energy, actually it will provide an upper limit for the crack arrest length. The energy release  $-\Delta P$  may be derived from the energy release rate

$$G = K_{\rm I}^2 / E. \tag{6}$$

The stress intensity  $K_{I}$  depends on the crack size C via

$$K_{\rm I} = \frac{1}{Z} \sigma(C)^{1/2}$$
 (7)

where  $\sigma$  is the applied stress and Z a geometry factor. Due to the exponential increase of crack velocity with  $K_{\rm I}$ , even during subcritical stages we are concerned with a relatively rapid crack propagation. Therefore, we can assume that the applied stress is approximately constant during crack growth and  $K_{\rm I}$  increases mainly due to the growing value of C. Then  $\sigma$  can be calculated from the condition  $K_{\rm I} = K_{\rm Ic}^0$  at the starting length  $C = C_0$ . Equation 7 yields

$$\sigma = Z \cdot K_{\mathrm{Ic}}^0 / C_0^{1/2}. \tag{8}$$

(i)

Substituting for  $K_{I}$  in Equation 6 by the help of Equations 7, 8, and 4 gives the energy release rate at constant load dependent on the crack length C:

$$G = G(C) = 2\gamma_0 C/C_0. \tag{9}$$

The fracture surface work  $\Delta W_{\gamma}$  can be immediately derived from the specific work  $\gamma$  of Equation 3. Since  $K_{\rm I}$  depends on the crack size, the  $\gamma_0 \rightarrow \gamma_1$ transition proceeds after a certain amount of crack elongation. This particular crack size, say  $C_{\rm D}$ , can be calculated from Equations 7 and 8 when  $K_{\rm I}$  is replaced by  $K_{\rm D}^{\rm D}$ :

$$C_{\rm D} = C_0 (K_{\rm I}^{\rm D} / K_{\rm Ic}^0)^2.$$
 (10)

Thus we have the result that for  $C < C_D$  the fracture surface work is given by  $\gamma_0$  whereas for  $C \ge C_D$  it is determined by  $\gamma_1$ . Note that  $C_D$  is proportional to the starting length  $C_0$ , i.e. it is not a material constant.

In order to simplify the analytical procedure, let us assume that the propagation of the macrocrack is a one-dimensional process. Of course this is only an approximation, but it describes well the principal modes of crack extension which occur in reality. Therefore, the energy balance (Equation 5) may be expressed by G and  $\gamma$  in the following form:

$$\int_{C_0}^{C_s} G(C) \mathrm{d}C = 2 \int_{C_0}^{C_s} \gamma(C) \mathrm{d}C \qquad (11)$$

where  $C_s$  denotes the crack arrest length. Inserting Equation 3 and 9 into 11 yields

$$\gamma_0 (C_s^2 - C_0^2) / C_0 = 2\gamma_0 (C_D - C_0) + 2\gamma_1 (C_s - C_D)$$
(12)

from which we obtain for  $C_s$ :

$$\frac{C_{\rm s}}{C_0} = \frac{1}{(K_{\rm Ic}^0)^2} \left[ \left[ (K_{\rm Ic}^1)^2 - \left\{ \left[ (K_{\rm Ic}^1)^2 - (K_{\rm Ic}^0)^2 \right] \right. \right. \right. \\ \left. \times \left[ (K_{\rm Ic}^1)^2 + (K_{\rm Ic}^0)^2 - 2(K_{\rm I}^D)^2 \right] \right\}^{1/2} \right]$$
(13)

Here we have replaced  $\gamma_0$  and  $\gamma_1$  by  $K_{Ic}^0$  and  $K_{Ic}^1$  using Equation 4. From Equation 13 we observe that crack arrest is possible only if the following condition is satisfied:

$$K_{\rm I}^{\rm D} \leq \{ [(K_{\rm Ic}^0)^2 + (K_{\rm Ic}^1)^2]/2 \}^{1/2}.$$
 (14)

Otherwise no real solution for  $C_{\rm s}$  exists, i.e. the increase in toughness at  $K_{\rm I}^{\rm D}$  is insufficient to stabilize the running crack. Now we can summerize the different modes of crack propagation which are schematically represented in Fig. 1.

$$K_{\rm I}^{\rm D} \leq K_{\rm Ic}^0 < K_{\rm Ic}^1 \tag{15}$$



Figure 1 Relations between the crack length C and the applied stress intensity  $K_{\rm I}$ . For the resting crack (solid lines) the increase of  $K_{\rm I}$  is provided by the growing load whereas during unstable stages (dotted lines)  $K_{\rm I}$  increases due to the crack elongation at almost constant macroscopic stress (Equation 7). See text for propagation modes (i), (ii), (iii).

The energy-dissipative zone is already active before the onset of crack propagation. Therefore,  $K_{\rm I}$ must be increased beyond  $K_{\rm Ic}^0$  up to  $K_{\rm Ic}^1$  in order to initiate crack propagation. Then the crack starts from its original length  $C_0$  and no subsequent stabilization takes place.

(ii)

$$K_{\rm Ic}^0 < K_{\rm I}^{\rm D} \leq \{ [(K_{\rm Ic}^0)^2 + (K_{\rm Ic}^1)^2]/2 \}^{\nu_2} < K_{\rm Ic}^1$$
(16)

The crack becomes unstable (at least on the microscale) at  $K_{Ie}^0$ . Due to the increasing  $K_I$ , energy dissipation commences at  $K_I^D$  and the crack becomes stabilized at the length  $C_s$  given by Equation 13. Then the load must be further increased to cause final instability at  $K_{Ie}^1$ . (iii)

$$\{[(K_{\rm Ic}^0)^2 + (K_{\rm Ic}^1)^2]/2\}^{1/2} \le K_{\rm I}^{\rm D}$$
(17)

Here again crack propagation starts at  $K_{Ic}^0$ . Energy dissipation is initiated after a certain amount of crack elongation but, in contrast to mode (ii), it is insufficient to prevail against the energy release of the running crack and to stabilize it. Therefore,  $K_{Ic}^1$  has no influence on this mode of crack propagation.

Now we turn to our second problem concerning the influence of the grain size D on the threshold stress intensity  $K_{\rm I}^{\rm D}$  (Equation 2). Here we shall concentrate on microcracking at grain boundaries since this mechanism appears to be the dominating energy-dissipative process in single phase alumina. In order to calculate  $\sigma_{\rm c}$ , the critical local stress at which microcracking starts, we must take into account the residual stresses being inherent to most ceramics. In polycrystalline Al<sub>2</sub>O<sub>3</sub> they are due to thermal expansion anisotropy. Especially the tensile residual stress  $\sigma_{\rm i}$  is important since it reduces  $\sigma_{\rm c}$  below that critical stress  $\sigma_{\rm mc}$  which would be necessary to initiate microcracking in the absence of residual stresses:

$$\sigma_{\rm c} = \sigma_{\rm mc} - \sigma_{\rm i}. \tag{18}$$

The residual stress  $\sigma_i$  can be assumed to be approximately independent of the grain size D whereas  $\sigma_{mc}$  depends on D. This dependence can be derived assuming that microcracking starts from some defects (e.g. small pores at the triple points of the polycrystalline structure), the size, say 2a, of them being proportional to D. Then the Griffith criterion for a penny shaped flaw yields

$$\sigma_{\rm mc} = \left(\frac{\pi E \gamma_{\rm gb}}{2a}\right)^{1/2} = \frac{A}{D^{1/2}}, \qquad (19)$$

where  $\gamma_{\rm gb}$  is the specific fracture energy of a grain boundary. The new constant A comprises all parameters except the grain size D. An identical relation between  $\sigma_{\rm me}$  and D has been derived by Lange [15] using a more profound model for microcrack formation. Finally we must choose an appropriate value for n representing that dissipation zone size which must be exceeded in order that the energy dissipative process does increase the toughness. In this paper we assume that the diameter  $2r_{\rm D}$  of the zone at the transition point is about 2D, this means n = 1. Using this result and Equations 18 and 19 we obtain from Equation 2

$$K_{\rm I}^{\rm D} = 3^{1/2} (A - \sigma_{\rm i} D^{1/2}).$$
 (20)

Thus we have derived  $K_{I}^{D}$  dependent on the grain size, the residual stress  $\sigma_{i}$ , and the grain boundary fracture energy included in A. The theoretical results, i.e. Equations 13, 15, 16, 17, and 20 will form the basis of our discussion in Section 4.

#### 3. Experimental details

Dense polycrystalline single phase alumina specimens with about  $5 \text{ mm} \times 5 \text{ mm}$  cross-section were fabricated by cold pressing and sintering in  $H_2$ . At a constant average grain size of about  $3 \mu m$  it turned out that the mechanical properties strongly depend on the actual kind of the grain size distribution [16]: Samples having two separate maxima of the distribution  $(\overline{D}_1 = 2.1 \,\mu\text{m}, \ \overline{D}_2 = 10$  to  $15\,\mu m$ ) in the ideal case showed an improved strength of 500 to 600 MPa and  $K_{Ic} = 7$  to  $8 \text{ MPa m}^{1/2}$ . Presumably this effect is due to microcracking preferentially starting around the larger grains and optimized with respect to microcrack length and density by the fine-grained matrix which hinders microcrack coalescence. For the present investigations underfired specimens with a lower fraction of larger grains and lower values of  $K_{Ic}$  were produced. They have been compared with samples made in an analogous manner and having the same average grain size (cf. Table I) but with a narrow monodisperse distribution instead of a bidisperse one.

To examine subcritical crack propagation the fracture induced photon emission was measured during 3-point-bending fracture in a high-vacuum chamber. The emission is generated at the crack tip by stress activated luminescence from a priori present point defects of the lattice. Details have been reported in a previous paper [17]. Due to the limited counting capacity of the electronics there

TABLE I Structure and properties of homogeneous (A) and bidisperse (B) aluminas.  $\rho$  denotes the microcrack density,  $\sigma_f$  the average fracture strength as measured directly in the course of the luminescence tests.  $K_I^*$  and  $K_{Ic}$  are the stress intensities for the macroscopically observable onset of subcritical crack growth and for instability, respectively.  $I_c(B)/I_c(A)$  gives the ratio of cumulative photon emissions registered during bending fracture of specimens (A) and (B). VH is Vickers hardness measured at 98.1 N.

Grain structure	ρ	σ <sub>f</sub> (MPa)	$K_{\rm I}^{*}$ (MPa m <sup>1/2</sup> )	К <sub>Iс</sub> (MPa m <sup>1/2</sup> )	$I_{\mathbf{c}}(\mathbf{B})/I_{\mathbf{c}}(\mathbf{A})$
$(A) \overline{D} = 3.0 \mu\text{m}$	1.4	305 ± 80	2.9 ± 0.3	5.0 ± 0.2	0.05-0.30 at
(B) $\bar{D}_1 = 2.1 \mu \text{m} (75 \text{vol}\%)$	1.0	$480 \pm 60$	$4.6 \pm 0.3$	5.4 ± 0.2 ∫	VH = 16.4 - 17.8 GPa
$\bar{D}_2 = 10 - 15 \mu \text{m} (25 \text{vol}\%)$					
$\bar{D} \approx 3 \mu \mathrm{m}$ (all grains)					

was only negligible contribution from the highspeed crack propagation at  $K_{\rm I} > K_{\rm Ic}$  to the cumulative emission registered by a photomultiplier. Thus the emission  $I_{\rm c}$  is a measure of the total amount of all subcritical crack growth processes. Comparing the two structures we found a systematic variation of  $I_{\rm c}({\rm B})/I_{\rm c}({\rm A})$  with increasing hardness (cf. [17]); in Table I this is reflected by the stated range of data for  $I_{\rm c}({\rm B})/I_{\rm c}({\rm A})$ .

The critical stress intensity  $K_{Ic}$  has been determined by the usual notched beam test, by fractographic means from the Griffith equation using the fracture strength  $\sigma_f$  and the crack length  $C_f$  at final instability, and by indentation analysis. Besides  $K_{Ic}$  we have determined that value  $K_I^*$ where the subcritical crack growth becomes very rapid (10 to 100  $\mu$ m sec<sup>-1</sup>) and measurable crack extension can be observed. Testing the specimens with a usual loading rate (cross-head speed 0.5 mm min<sup>-1</sup> at a span length of 30 mm), further increase of  $K_I$  up to  $K_{Ic}$  occurs primarily by quick



Figure 2 Microcrack system observed on a fracture surface of alumina. TEM micrograph; C-Pt-replica technique marks microcracks by dark contours following cracked grain boundaries.

subcritical crack growth at almost constant load. Therefore, the stress intensity  $K_{\rm I}^*$  requested for the onset of quick subcritical crack propagation can be estimated combining  $\sigma_{\rm f}$  with the flaw size  $C_0$ . This original flaw size  $C_0$  and the subcritical crack growth boundary  $C_{\rm f}$  have been determined on fractographs using a procedure very similar to that described by Kirchner and Gruver [18, 19]. Typically we have found  $C_0 \approx 50 \,\mu{\rm m}$  and  $C_{\rm f} \approx 100$  to  $200 \,\mu{\rm m}$ .

In order to characterize the microprocess of energy dissipation the microcrack density  $\rho$  has been estimated from the lengths of microcrack traces measured on the fracture surface (replica micrographs) [16]. Fig. 2 presents an example of microcracking starting at a larger grain in a somewhat overfired bidisperse structure. The sum of the squares of the trace lengths related to the analysed surface area gives relative values for  $\rho$  as stated in Table I. Due to the special definition used such relative values can exceed unity.  $\rho = 1.0$ for the underfired bidisperse samples was estimated from  $\rho = 1.16$  as measured for the fully developed structure with  $K_{Ie} = 7$  to  $8 \text{ MPa m}^{1/2}$ [16]. Relating the microcracked area to the grain boundary area we obtained absolute crack densities of about 0.40.

#### 4. Discussion

Table I shows markedly different amounts of luminescence emission  $I_c$  for the two ceramic structures. This fact may be explained by the occurrence of different modes of crack propagation. In order to verify this we must evaluate the conditions 15 to 17. To this end we first have to estimate the basic parameters appearing in the derived relations. Fracture starts within a structure which is thought as having no microcracks. First intensive spalling of grains at the tip of the macrocrack (the fracture initiating flaw) will occur at  $K_{1c}^{0}$ , i.e. if  $K_{1}$  equals  $K_{1c}$  of the most easily spalling

TABLE II Estimation of parameters used in the theoretical model for single phase polycrystalline alumina

Basic parameters	Parameters derived from Equ	ation No.	
$\gamma \{ \overline{1} \ 0 \ 1 \ 2; \ \overline{1} \ 0 \ 1 \ 0 \} = 6 - 7 \ J \ m^{-2} $ $[20]$ $\gamma_{gb} \approx \frac{1}{10} \gamma \{ \overline{1} \ 0 \ 1 \ 2 \} \approx 0.6 \ J \ m^{-2}$ $E = 400 \ GPa$ $2a \approx (0.1 \ to \ 0 \ 2) D$	$K_{1c}^{0} = 2.7 \text{ MPa m}^{1/2}$ $A = 2.34 \text{ MPa m}^{1/2}$	(4) (19)	
$\sigma_{i} \approx 150 \text{ MPa [21]}$ $D = \begin{cases} 4 \mu\text{m (A)} \\ 20 \mu\text{m (B)} \end{cases}$	$K_{\rm I}^{\rm D} = \begin{cases} 3.5 \text{ MPa m}^{1/2} \text{ (A)} \\ 2.9 \text{ MPa m}^{1/2} \text{ (B)} \end{cases}$	(20) (20)	

lattice planes of the Al<sub>2</sub>O<sub>3</sub>-crystal. Following Wiederhorn [20] this is about 2.1 to 2.5 MPa m<sup>1/2</sup> for { $\overline{1}$ 012} and { $\overline{1}$ 010}. Since the average orientation of such planes in a fracturing polycrystalline specimen will deviate from that of the macrocrack plane, a slightly higher value may be requested for  $K_{1c}^{0}$  (Table II). Once started, the crack accelerates and can proceed on grain boundaries providing an increased percentage of intercrystalline fracture as it has been observed by Kirchner and Gruver on fracture surfaces of sintered [18] and hot pressed [19] aluminas at  $K_{I} \approx 3$  MPa m<sup>1/2</sup>, which is only slightly beyond our estimation of  $K_{1c}^{0}$ .

Furthermore, Table II presents the estimated values for the residual stress  $\sigma_i$ , for the microflaw size 2a, for the grain boundary fracture energy  $\gamma_{gb}$  and the resulting constant A, which determines the critical stress for microcracking. Finally we must choose a characteristic value for D describing the size of those grains at which microcracking first occurs. Of course, the probability to find a grain of this size at the crack front must not be too low in order that a microcracked zone is really developed. Therefore  $K_I^D$  will be governed by a certain D between the average and the maximum diameter occurring in the grain size distribution.

Now we are in a position to apply the conditions 15 to 17 to our special alumina structures. The parameters  $K_{Ic}^0$  and  $K_{I}^D$  may be obtained from Table II and  $K_{Ic}^1$  is given by the macroscopic  $K_{Ic}$  (Table I).

(a) Homogeneous structure:  $K_{Ic}^0 = 2.7 \text{ MPa m}^{1/2}$ ,  $K_I^* = 2.9 \text{ MPa m}^{1/2}$ ,  $K_I^D = 3.5 \text{ MPa m}^{1/2}$ ,  $K_{Ic}^1 = 5.0 \text{ MPa m}^{1/2}$ . For this structure the experimentally observed  $K_I^*$  practically equals  $K_{Ic}^0$  whereas the threshold value  $K_I^D$  is well above  $K_{Ic}^0$  and  $K_I^*$ . Inspection of condition 16 shows that crack propagation proceeds according to mode (ii). The initially moving crack is stopped at  $C_s = 1.59 C_0$  (Equation 13) after having reached  $C_D$ . Then

again slow subcritial crack growth takes place and the applied load may be raised until  $K_{Ie}$  is reached.

(b) Bidisperse structure:  $K_{Ie}^{0} = 2.7 \text{ MPa m}^{1/2}$ ,  $K_{I}^{D} = 2.9 \text{ MPa m}^{1/2}$ ,  $K_{I}^{T} = 4.6 \text{ MPa m}^{1/2}$ ,  $K_{Ie}^{T} = 5.4 \text{ MPa m}^{1/2}$ . Since the threshold value  $K_{I}^{D}$  is approximately equal to  $K_{Ie}^{0}$  and well below  $K_{I}^{*}$  (describing the onset of quick subcritical crack growth), we conclude that energy dissipation has commenced already before the beginning of crack extension. This means that condition 15 applies and crack propagation proceeds according to mode (i).

Thus we now understand the considerably higher value of  $K_{I}^{*}$  for the bidisperse structure. Due to this  $K_{\rm I}^*$  the outer boundary of the microcracked zone increases up to 50 to 70  $\mu$ m already in the subcritical range (Equations 1, 18, and 19 using the maximum  $D \approx 20$  to  $25 \,\mu\text{m}$ ). This  $r_{\rm D}$ agrees well with our *in situ* observations made by means of a scanning electron microscope. Obviously in this case we must expect that microcracking occurs also around smaller grains situated in the central part of the dissipation zone. Therefore the microcrack density is thought to decrease with increasing distance from the microcrack tip. Although this may cause an additional influence onto the crack propagation mode in the region  $K_{\mathbf{I}}^* \leq K_{\mathbf{I}} \leq K_{\mathbf{Ie}}$  we do not expect substantial changes.

To illustrate our discussion Fig. 3 schematically shows the different stages of crack propagation starting from a flaw size assumed as  $50 \,\mu\text{m}$  for both structures. The intermediate crack lengths were calculated from Equations 10 and 13. Dissipation zone sizes were obtained using Equations 1, 18, and 19 with the average grain size  $\overline{D} = 3 \,\mu\text{m}$ for both structures (cf. Table I); thus the depicted zones represent a certain effective size between a minimum value belonging to the smaller grains and the outer boundary determined by the larger grains. The same effective zone size will be used for the estimation of luminescence.

Although the homogeneous structure exhibits



(b) bidisperse



a higher microcrack density  $\rho$  than the bidisperse one, the critical stress intensity factor  $K_{Ic}$  is lower. This can be understood since beyond a certain limit of the density  $\rho$  the microcracks tend to coalesce thereby reducing the toughness. However, a detailed discussion of  $K_{Ic}$  as dependent on  $\rho$  is not the aim of the present paper. Rather we would like to find out, whether our measurements of luminescence support the concept of different modes of crack extension as shown in Fig. 3. To this end we shall theoretically estimate the luminescence  $I_{e}$  to be expected for the two structures. According to the experimental technique, the cumulative emission  $I_c$  is proportional to the density of microcracks  $\rho$  and the area over which the microcracked zone moves during the subcritical stages of crack propagation:

$$I_{\rm c} \sim \rho \int_{C_1}^{C_2} r_{\rm D}(C) \mathrm{d}C, \qquad (21)$$

where  $C_1$  and  $C_2$  bound the region of subcritical crack growth. The density  $\rho$  in Equation 21

Figure 3 Modes of subcritical crack propagation in alumina structures with homogeneous and bidisperse grain size distribution, respectively.

characterizes the average amount of microcracking exhibited by the different structures. In this sense we assume that  $\rho$  is almost constant in the subcritical stage of crack propagation, In addition we presuppose that only one macrocrack is initiated by the worst flow. For the size  $r_{\rm D}$  of the microcracked zone we again have to use a certain average value since luminescence is a measure of the total amount of microcracking around the grains of all sizes concerned. Therefore we make use of Equation 1 assuming an effective critical stress  $\sigma_{c}$ determined by the average grain size  $\overline{D}$  (cf. the above discussion to Fig. 3). Since  $\overline{D}$  is identical for the two structures (Table I) we obtain from Equation 1  $r_{\rm D} \sim K_{\rm I}^2$ , and Equation 21 may be rewritten as

$$I_{\mathbf{c}} \sim \rho \int \frac{c_2}{c_1} K_{\mathbf{I}}^2(C) \mathrm{d}C.$$
 (22)

Because  $K_{I}^{2}$  is a linear function of C (Equation 7), it can easily be shown that this integral is given by

$$I_{\rm c} \simeq \frac{1}{2} \rho (K_{\rm I}^2(C_1) + K_{\rm I}^2(C_2))(C_2 - C_1). \quad (23)$$

The lengths  $C_1$  and  $C_2$  and the corresponding values for  $K_1$  can be taken from Fig. 3. For the homogeneous structure we have  $C_1 = 73 \,\mu\text{m}$  and  $C_2 = 150 \,\mu\text{m}$ , whereas the behaviour of the bidisperse structure is described by  $C_1 = 50 \,\mu\text{m}$ and  $C_2 = 70 \,\mu\text{m}$ . Using these data we obtain for the ratio of emission intensities  $I_c(B)/I_c(A) = 0.25$ . This result agrees fairly well with the experimentally observed emissions and seems to confirm our notion about different crack propagation modes. In addition, the correspondence between theory and experiment substantiates the assumption about the active role of microcracking in the subcritical stage of fracture. This is also in accordance with the results of acoustic emission experiments, which indicated the formation of a microcracked zone before the onset of subcritical crack growth [22] and a dependence of such crack extension on the actual grain size distribution in sintered alumina [3, 23].

Nevertheless there are still some open problems. These concern especially the determination of the microcrack density, since only a two-dimensional section of the real three-dimensional network of cracks was examined. Moreover, only a certain average density independent of the actual propagation stage was measured. Also the determination of luminescence involved some difficulties since only microcracking on the sample surface contributes to the observed intensity. But one may hope that errors due to these problems are partially ruled out since we have considered only relative values of luminescence. Another problem is the following. The formation of the dissipation zone, especially in the bidisperse structure, is not such a step-like process as assumed by the theory. Hence a more detailed investigation taking into account the inhomogeneous structure of the microcracked zone seems to be useful in order to further understand the processes on the microscale.

## 6. Conclusions

A model has been presented for the initiation of energy-dissipative mechanisms in brittle materials. Energy is dissipated if the stress intensity at the tip of the macrocrack reaches a threshold value  $K_{\rm I}^{\rm D}$ . This can result in a new stabilization of the running crack if some conditions are met concerning the ratio between  $K_{\rm I}^{\rm D}$  and the two toughness levels  $K_{\rm Ic}^{\rm 0}$ (without energy dissipation) and  $K_{\rm Ic}^{\rm 1}$  (generated by the dissipative process). This model holds independently of the actual process of dissipation.

Experimental results obtained by measuring the fracture induced photon emission are readily explained if the theoretical model is combined with the idae of microcracking as the governing energy-dissipative mechanism in single phase sintered alumina. It turned out that the grain size distribution of the material plays an important role since the threshold stress intensity and the fracture toughness depend on it. Finally one may conclude that microcracking appears to be the dominating mechanism being responsible for the observed pecularities of crack propagation and for the mechanical properties of alumina structures.

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