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Threshold microfracture during elastic-plastic indentation of ceramics

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It is shown that for a variety of ceramics the threshold for microfracture during elastic–plastic indentation corresponds to radial, rather than subsurface median, crack formation. This is contrary to the fundamental assumption of current models for threshold crack nucleation by sharp indenters. The significance of this observation in terms of strength reduction is discussed.

1. Background

Models have recently been proposed [1, 2] to predict the critical load and flaw size conditions which should prevail at the threshold for crack formation* (initiation) in ceramics subject to elastic–plastic indentation. Both of the models explicitly deal with the initiation of subsurface median cracks†, the justification for which would seem to be two-fold. First, median cracks have been demonstrated to exist, for example in soda–lime glass, as shown by Lawn and Evans [1]. Second, median cracks lend themselves to analysis, in that they are presumed to form beneath the indenter apex at subsurface origins, where indentation stress field analyses are relatively unperturbed by the constraint-free surface. Unfortunately, however, median cracks have not actually been shown to exist at the threshold.

In fact, there have been relatively few studies of indentation microfracture in which tests were performed at sufficiently low loads to allow the determination of the threshold load and crack size; even fewer tests have been performed in which the appropriate microscopic inspection was actually performed. Recently, the author carried out experiments to determine indentation threshold crack sizes and loads for a variety of crystalline ceramics, including fine-polished Al_2O_3, SiC, Si, as-grown Ge, and cleaved NaCl. Several factors related to the question of radial or median threshold microfracture were established [3, 4], as briefly summarized here. First, for all five materials, the lengths of the individual surface traces (C' in Fig. 1) of the cracks formed by the threshold load, determined by SEM observation and acoustic emission, were in excellent agreement with threshold critical flaw sizes predicted by the Lawn and Evans model. Thus, if the surface traces had represented the intersection of median cracks with the surface, the "parent" median threshold cracks would have necessarily been extremely large, much larger than those predicted by the model. Moreover, no acoustic emission was detected below the apparent threshold for radial cracking, so that, should the threshold surface traces and the associated, initial acoustic emission have been caused by median cracking, the threshold median cracks for each material would have intersected the surface over a distance which happened to agree with the predicted size of the threshold flaw. This is a highly improbable situation and it thus appears most likely that the

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*Crack formation is that which can be experimentally measured. It consists of the critical flaw size, plus any crack extension which may have occurred during the indentation process; the latter is minimized in practice by carrying out tests as near the crack nucleation threshold as is possible.

† The Hagan model [2] is an effort to explain the seemingly too-large "fortuitous" flaws required by the Lawn–Evans model for alkali halides. This it does successfully, indicating that crack-nucleating flaws can indeed be deformation induced, rather than intrinsic. However, similar critical loads for median crack formation are derived. The idea of indenter deformation-nucleated flaws serving to initiate cracks should be applicable to radial cracks as well. Subsequent lateral crack formation is a separate issue not treated by either model.

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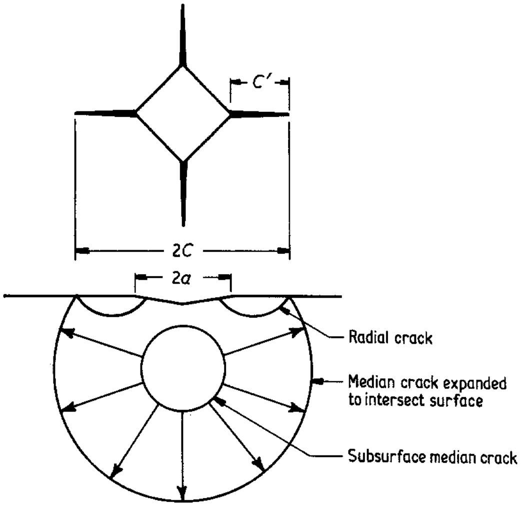

Figure 1 Radial versus median indentation microfracture patterns which produce a common surface trace, *C'*.

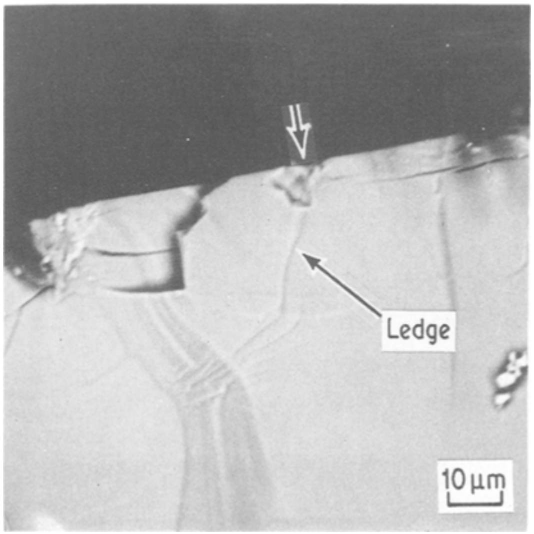

Figure 2 Subsurface damage attending indentation (arrow) produced in SiC by a 200 g load, edge formed by intersecting radial crack segments. No median crack formation.

observed surface traces represented radial crack formation.

These indirect inferences as to the prevalence of radial rather than median threshold cracking were supported by direct observation in two dissimilar ceramic systems. Radial cracking, to the absolute exclusion of median cracking, was observed optically [3] in transparent NaCl, and verified by acoustic emission, i.e., no acoustic emission was detected for loads below the radial crack threshold. In addition, specimens of SiC were cleaved through indentations formed by loads just above the ~ 10 g threshold load for cracking [4]. SEM inspection showed (Fig. 2) that the subsurface indentation regime was typically devoid of median cracks, while, prior to cleavage, radial cracks were present at the corners of each indentation.

2. New experiments

In order to assess the generality of these inferences and observations suggesting radial rather than median threshold crack formation, experiments have now been extended to other materials. For example, loads, P, ranging from 50 to 500 g were applied to polished (using 0.05 μm diamond paste) Lucalox* polycrystalline Al_2O_3. The microcracked indentations were then serial polished down from the original surface in approximately 1 μm increments, using large indentations as references. It was found that at relative low loads (50 to 200 g), but above the microfracture threshold load of about 25 g, only radial cracks were present (Fig. 3), with no discernible traces of median cracks beneath the indents. (Although the indentations were gradually removed during polishing, their positions were indexed, for observational purposes, on the larger reference indentations which remained; thus, the positions which the medians would have occupied were precisely fixed.) At higher loads ($200\,\text{g} < P < 500\,\text{g}$), the radial cracks apparently began to link up below the indentations, without necessarily cracking them, forming an annular configuration. Finally, for $P \gtrsim 500$ g, the cracks began to crack across the indentations, assuming the form of half-penny surface cracks.

The observation of median cracks in glass apparently has been the principle basis for considering median crack formation to constitute the threshold. However, the median crack in soda–lime glass shown by Lawn and Evans (Fig. 1 of [1]) was 2 mm in diameter, produced by a load of 250 N; this certainly was not a threshold crack. Recent observations reported by Hagan and Swain [5] have indicated that for sufficiently low loads, i.e., $\lesssim 300$ g, radial cracking may occur in soda–lime glass without the occurence of median cracking; therefore, the following experiment was performed. An annealed soda–lime glass slide was subjected to diamond pyramid indentation at

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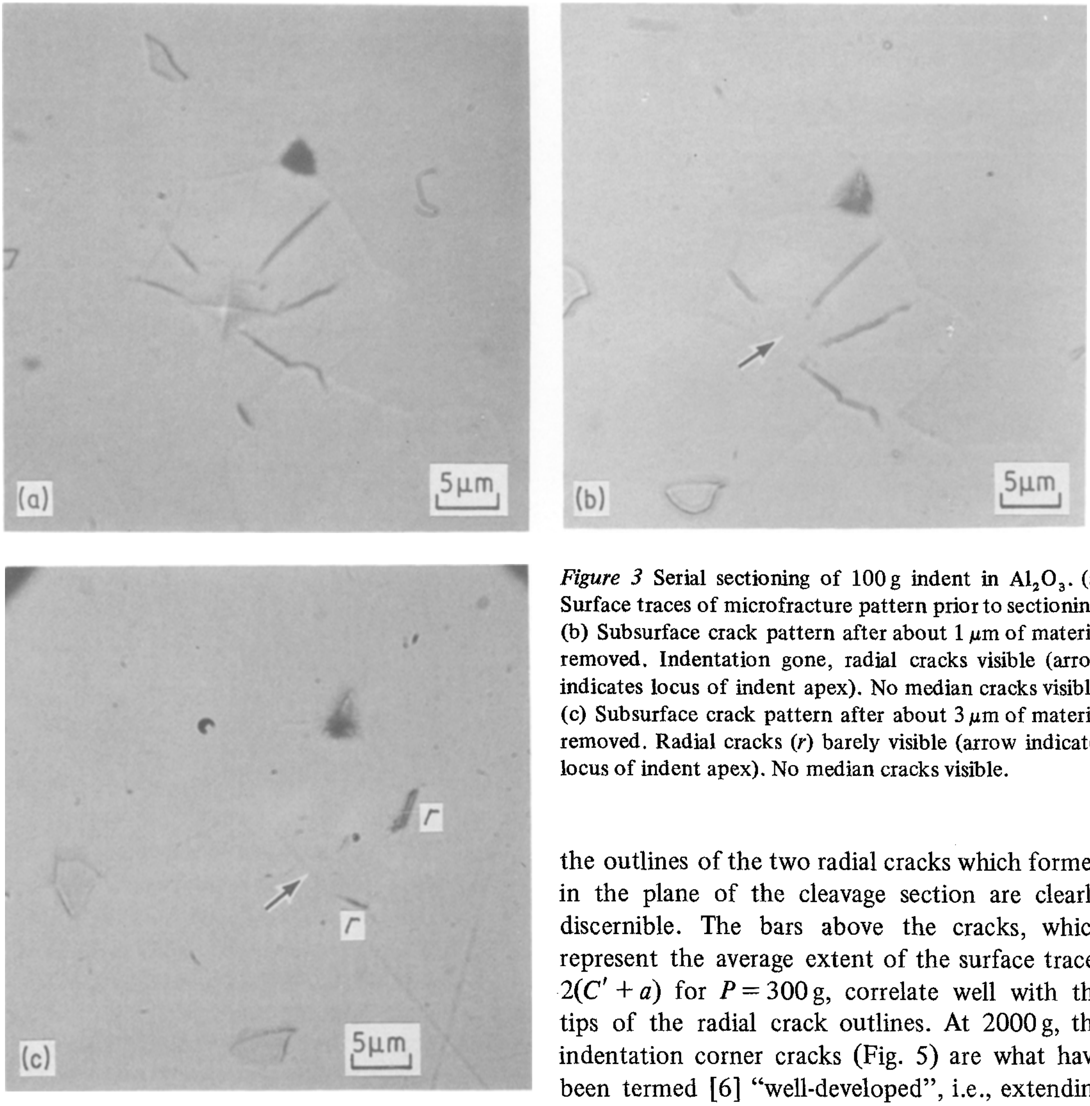

Figure 3 Serial sectioning of 100 g indent in Al_2O_3. (a) Surface traces of microfracture pattern prior to sectioning. (b) Subsurface crack pattern after about 1 μm of material removed. Indentation gone, radial cracks visible (arrow indicates locus of indent apex). No median cracks visible. (c) Subsurface crack pattern after about 3 μm of material removed. Radial cracks (*r*) barely visible (arrow indicates locus of indent apex). No median cracks visible.

various loads, from 10 to 2000 g. Scanning electron microscopy was used to determine the threshold load for the appearance of indentation corner cracks. The indentations, which were lined up in a row, were then cleaved by a single crack passing through the array; indentation subsurface crack morphology was characterized using the SEM.

It was found that the threshold load for indentation corner crack nucleation was approximately 25 g. Fig. 4 shows typical subsurface damage associated with indentations formed under these conditions. In many cases, lateral (see L in Fig. 4) as well as radial cracks form, but median cracks are always absent. This is particularly apparent in Fig. 4a; not only is no median crack visible but the outlines of the two radial cracks which formed in the plane of the cleavage section are clearly discernible. The bars above the cracks, which represent the average extent of the surface traces $2(C' + a)$ for $P = 300$ g, correlate well with the tips of the radial crack outlines. At 2000 g, the indentation corner cracks (Fig. 5) are what have been termed [6] "well-developed", i.e., extending well beyond the central deformation zone. However, even at this stage they clearly are not yet disc-shaped, since they have no subsurface median component (Fig. 4b), but remain unconnected radial cracks attached to a central indentation. The ledge in Fig. 4b indicates where the two radials in the plane of cleavage linked up during cleavage.

Identical experiments were carried out for Si crystals. Again the results were the same: for loads ranging from the threshold (~ 5 g) up to 200 g, no median cracks were present in sections cleaved through indents. Instead, the section revealed subsurface ledges at which radial crack segments had linked up during cleavage.

For all five materials which have been discussed, as well as Ge, which seems to behave like Si, the relationship between $(C' + a)$ and P was established (Fig. 6) for loads including, and in excess

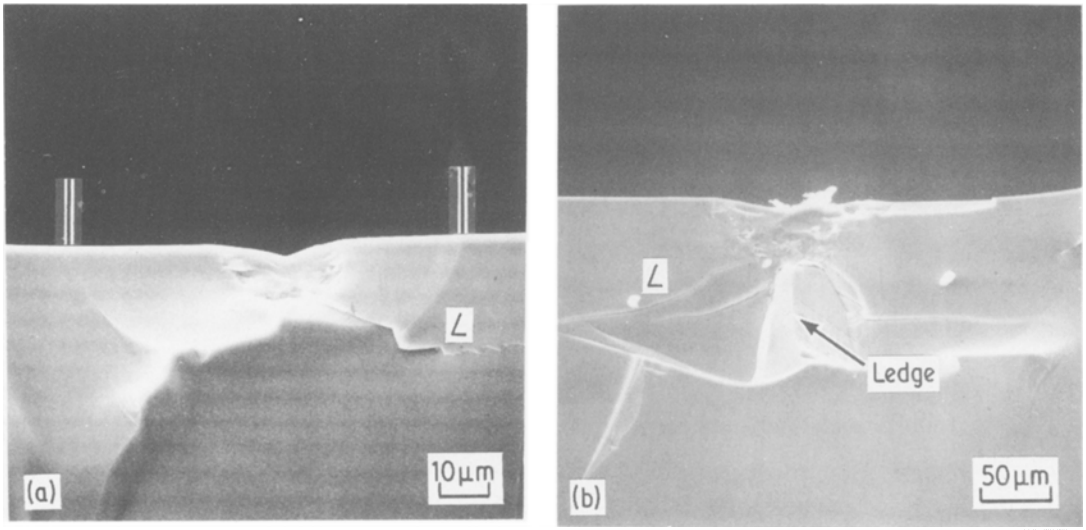

Figure 4 Subsurface damage attending indentation produced in soda–lime glass. (a) $P = 300$ g. Images of two radial cracks lying in the cleavage plane can be seen; tips correlate with average extent of $2(C' + a)$ for this load. Lateral cracking (L) occurs but no median cracks have formed. (b) $P = 2000$ g. Lateral cracks (L)form, but no median cracks have formed. Ledge produced by intersecting radial segments.

of, the threshold, and found to be, approximately, given by

$$(C' + a) \propto P^{2/3}, \qquad (1)$$

with the proportionality constant depending upon both material fracture toughness and indenter geometry [6]. This relationship is a crack growth law; it includes nucleation, but correlates the crack growth which takes place for loads in excess of the threshold. It is the same relationship which prevails at very high indentation loads, where the cracks formed are definitely half-penny shaped. Thus, there is a single functional representation which describes the growth of indentation corner cracks from an initial radial-plus-indentation configuration to a final half-penny shaped geometry. There is apparently no subsurface median crack stage, but simply the joining of individual radial segments; this joining is clearly shown by the connective ledges which are often observed beneath the indentations (see Figs 2 and 4).

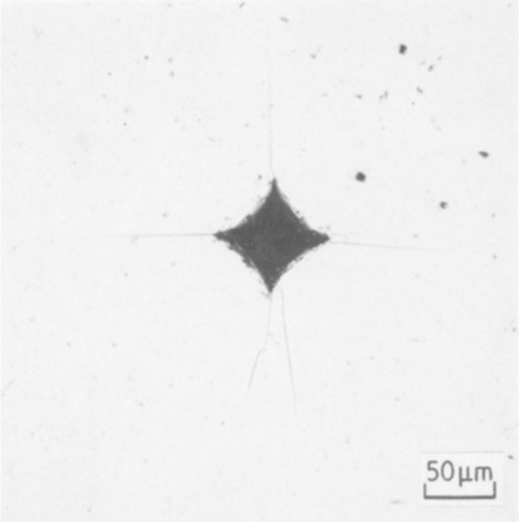

Figure 5 Indentation corner cracks produced by 2000 g load in soda–lime glass.

3. Discussion and implications

The experiments described above provide reasonable evidence that radial cracks are the earliest strength-degrading cracks to form during the elastic–plastic indentation of many ceramics. The materials studied covered a wide range of atomic bonding type, hardness, strength, toughness, and atomic order (crystalline versus amorphous). Acoustic emission did not occur for loads below the threshold loads required for indentation corner cracking. Sectioning, cleavage, and optical observation of transparent materials proved that the threshold surface traces were caused by radial cracks. The fact that no median cracks were observed for loads up to an order of magnitude higher than the radial crack threshold load indicates that median crack formation does not accompany the nucleation of radial cracks.

The data plotted in Fig. 6 provide indirect

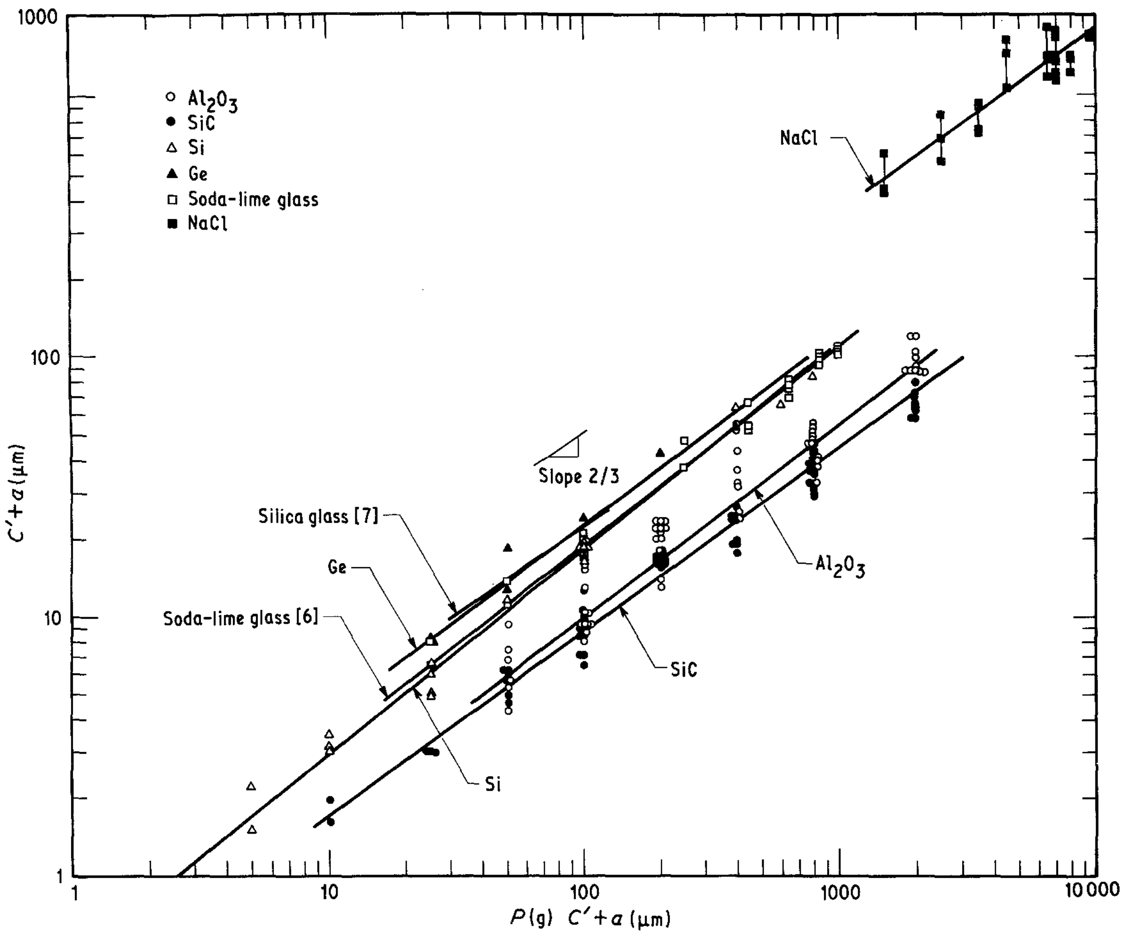

Figure 6 $(C' + a)$ plotted against P for various ceramics; data begin at the (approximate) threshold load in each case.

additional evidence in support of these conclusions. If the observed threshold surface traces represented the intersection of a large median crack with the surface, there should follow at higher loads a transient overshoot in the $(C' + a)$ against P plot, as the crack configuration changes to that of a half-penny by extending rapidly along the surface. This was not observed. Similarly, if the initial configuration (in a given plane) consisted of two radial cracks plus a subsurface median crack, a shift in the $(C' + a)$ against P plot should be observed, corresponding to the load range in which the radial and median cracks grew together to form a half-penny. Again, this is not observed.

Since absolutely no shift or overshoot in the crack growth law was observed, the linear relationship between $(C' + a)$ and $P^{2/3}$ suggests that the radial-plus-indent flaw can be treated as a composite crack of total dimension $2(C' + a)$ in predicting above-threshold crack growth. Inspection of stress intensity solutions for holes with corner cracks, and for elliptical surface cracks of equivalent length [8], suggests, by way of analogy with the present problem, that for $C'/a \gtrsim 1$ (true for all loads in the present case), the stress intensity at the surface should not be significantly different for radial (length C') and half-penny (length $2C$) cracks at indentations where $(C' + a) = C$. This would support $C \propto P^{2/3}$ for both crack configurations, as observed. Moreover, recent experiments by Dabbs *et al.* [7] have shown that the strength degradation of glass specimens indented at loads just above the crack formation threshold* could be predicted using the fracture mechanics relationship for an assumed disc-shaped crack of length $2C$. This effectively treats the radial-plus-indent combination as a single flaw of total dimension $2(C' + a)$.

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*The threshold in these experiments was based on observation of surface traces of corner cracks; considering the present work, this would correspond to radial cracking similar to that shown in Fig. 4.

The indentation load required to produce a threshold crack is critically dependent upon the crack initiation process. The Lawn and Evans model, although attractive in general, errs in its quantitative prediction of the threshold load [3], principally because of its assumption that the threshold relates to median cracking. This assumption leads to calculation of threshold loads based on the application of Hill's solution for an internally pressurized, spherical cavity situated (conceptually) at the subsurface tip of the indenter. If, instead, the assumption is made that the threshold involves radial cracking, a different, more difficult stress analysis, dealing with tensile stresses near the indenter but at the surface, must be carried out. The author has applied the only current analysis which treats this problem, that of Perrott [9], to the model of Lawn and Evans, by simply substituting for the stress field. The loads predicted by this modified model are shown [3] to be much lower, in fact, falling into agreement with experimental results [3]. A recent theoretical stress analysis of the elastic–plastic indentation problem by Evans *et al.* [10], agrees with the present findings in predicting surface radial, rather than subsurface median, initial crack formation.

Finally, in terms of strength reduction, it is much easier to nucleate flaws in engineering ceramics than would be predicted on the basis of the current models which assume a median crack threshold. While there may be a threshold load to form median cracks, it would seem to be of such a high magnitude as to be rather uninteresting compared to the much lower load regime for radial crack nucleation. Fortunately, the present results indicate that, even in their earliest stages, the strength reduction of the initial radial-plus-indent flaws can be treated analytically as if the composite flaws were tiny, half-penny cracks.

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