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Fracture and fracture toughness of stoichiometric $MgAl_2O_4$ crystals at room temperature

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The fracture toughness and path of stoichiometric spinel ($MgAl_2O_4$) crystals were determined at 22 °C for key low-index planes by double cantilever beam, as well as fractography of flexure specimens failing from either machining or indentation flaws. These results are compared with other single and polycrystalline $MgAl_2O_4$ fracture toughness values measured by various techniques, as well as single crystal versus polycrystal results for other materials. Evaluation of experimental and theoretical results shows (1) the fracture toughness of the spinel {1 1 0} plane is only a limited amount (e.g. 6%) higher than for the {1 0 0} plane ($\sim 1.2\ MPa\,m^{1/2}$), (2) fractography of machining flaw fracture origins was the most effective source of K_{IC} results, and (3) caution must be used in applying fracture toughness techniques to single crystals. Cautions include accounting for possible effects of elastic anisotropy (especially for double cantilever beam and probably double torsion tests), the nature of failure-initiating flaws (especially for notch-beam tests), and the frequent lack of symmetric plastic deformation and fracture (especially for indentation techniques).

1. Introduction

Fracture information on spinel ($MgAl_2O_4$) crystals is of practical interest, because they are used in various (e.g. optical and electronic) applications. It is also of broad scientific interest in order to give ultimately greater insight into single-crystal fracture and its relation to polycrystalline fracture. For example, it is important to understand how much higher polycrystalline versus single crystal fracture energies are due to combinations of fracture on different single-crystal planes, and the resultant mixed-mode failure on a microscopic scale, i.e. within or between individual grains [1, 2]. Single-crystal fracture data are also important to understand crack-branching behaviour because different fracture toughnesses of different crystal planes affect the branching behaviour in a different way to materials with uniform fracture toughness, such as most glasses and polycrystalline bodies [1, 2]. Further single-crystal studies should provide an important opportunity to understand possible effects of elastic anisotropy on fracture behaviour, e.g. $MgAl_2O_4$ has elastic anisotropy comparable to, or greater than, a variety of other ceramic materials, e.g. ThO_2, UO_2, MgO, $SrTiO_3$, and $BaTiO_3$ [3]. This paper reports fracture and fracture toughness studies of stoichiometric $MgAl_2O_4$ crystals at 22 °C, complementing other work on such crystals [4–6].

2. Experimental procedure

Specimens were diamond sawn from stoichiometric Czochralski-grown crystal boules (Union Carbide Corporation, Linde Division) ranging in diameter from over 3 to ~5 cm, depending upon orientation. Crystals were oriented for differing tensile axes and surfaces by the Laue method with orientations estimated to be accurate within ~3°. Double cantilever beam (DCB) specimens, nominally 1.25 cm × 0.25 cm × ~5 cm, were diamond machined with a groove ~1.5 mm wide down the centre of one side approximately half-way through the thickness. DCB tests for K_{IC} were carried out using the applied moment technique [7], and the resultant fracture mode observed.

Bars for three-point flexure testing (1.2 cm span) having cross-sections of ~2 mm × 3.5 mm were diamond ground either separately or from the remaining pieces from DCB tests. Such bars were ground either perpendicular or more commonly parallel with their tensile axis [8] and had their edges rounded (parallel with the tensile axis) prior to mechanical testing. A few discs (~2 mm thick) were biaxially tested using a three-ball support with a small centre loading piston. Fracture toughness was obtained from flexure bars used in this and other studies [1, 2, 8]. This was done by utilizing fractography to determine the fracture origins and the size, C, and shape of the fracture initiating flaws via scanning electron microscopy. Fracture toughness, K_{IC}, was then calculated from these data and the strength, σ, using the equation

$$K_{IC} = Y\sigma C^{1/2} \quad (1)$$

where Y is a factor reflecting flaw type and shape (2.0 for slit cracks and 1.2 for half-penny cracks where $C \ll$ specimen thickness). Both naturally occurring

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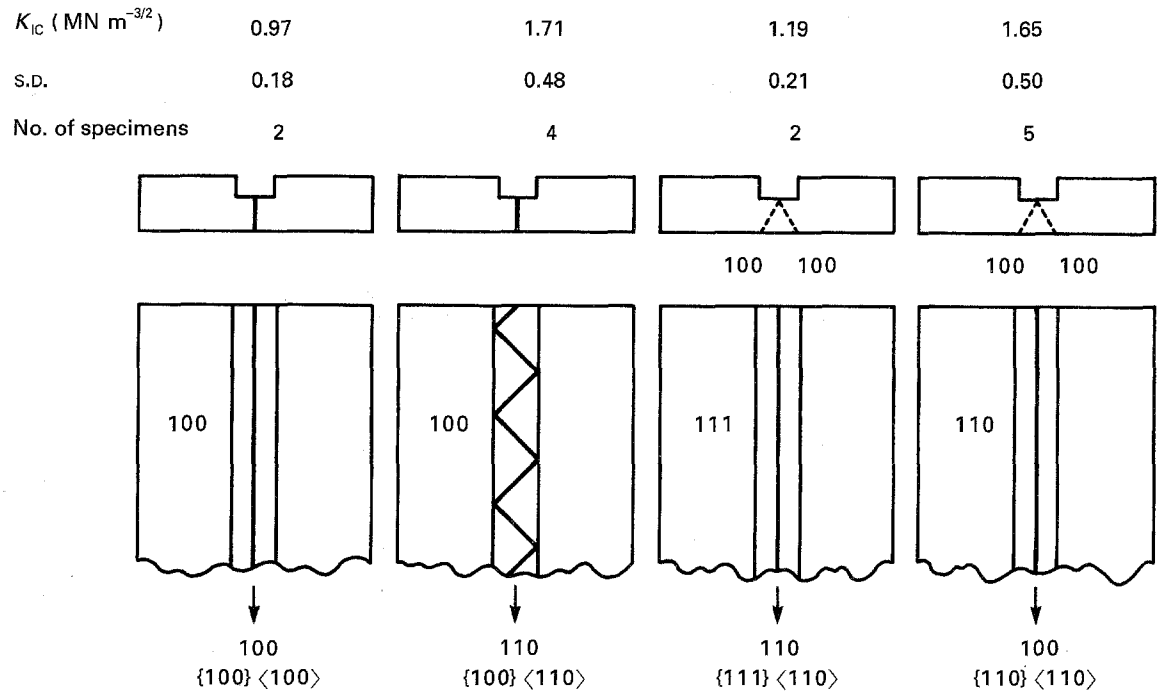

Figure 1 Summary of DCB fracture toughness results. Both plane and cross-sectional views of the different DCB specimens and the resultant crack-propagation characteristics for the different orientations are shown along with the fracture toughness, their standard deviations and the number of specimens used.

flaws, i.e. from the machining operations noted above, as well as from Vicker's indents (under loads of typically 2.5, 5, or 20 kg), were used. All tests were at $\sim 22\,^{\circ}C$, $\sim 50\%$ relative humidity.

3. Results

The results of the DCB tests on the four different crystal orientations studied are summarized in Fig. 1. The lowest $K_{IC} \sim 1$ MPa m$^{1/2}$, was observed for specimens where failure on $\{1\,0\,0\}$ planes was the expected fracture path, i.e. straight fracture nominally down the centre of the groove and perpendicular to the two major surfaces of the DCB specimen. The remaining specimens also failed mainly on or close to $\{1\,0\,0\}$ planes, but gave higher K_{IC} values. Such $\{1\,0\,0\}$ fracture occurred despite the fact that these fracture planes were either not normal to the major planes of the DCB specimens or not parallel with the specimen axis. (As discussed elsewhere [4] this latter, zigzag, $\{1\,0\,0\}$ failure pattern only occurred at lower velocities, e.g. below $\sim 5 \times 10^{-3}$ m s^{-1}. Above this velocity, an approximately flat fracture on $\{1\,1\,0\}$ planes was observed.)

Examples of fracture origins in strength tests from machining flaws are shown in Fig. 2 (see also [1, 2, 5]). Results of the fracture toughness calculated from such fractography, along with the overall summary of the fracture mode of crystals of various orientations tested, are shown (along with other K_{IC} data) in Table I. Fracture initiated on either $\{1\,0\,0\}$ or $\{1\,1\,0\}$ planes and subsequently propagated on either, or a combination, of these, depending upon orientations (Fig. 3). Different fracture modes resulted from the same tensile axis orientation of test specimens with different tensile surfaces. However, either substantially, or totally, $\{1\,0\,0\}$ fracture with a $\langle 1\,0\,0\rangle$ tensile axis and $\{1\,1\,0\}$ fracture with a $\langle 1\,1\,0\rangle$ tensile axis, was obtained. There were a few individual specimens that had substantially higher than average K_{IC} values, i.e. beyond the standard deviation. However, these occurred with specimens having $\{1\,1\,0\}$ tensile surfaces and either $\langle 1\,0\,0\rangle$ or $\langle 1\,1\,0\rangle$ tensile axis where the flaws from which failure initiated were not on the plane on which fracture propagated, hence these values were excluded. These resulted in, respectively, two and three different specimens having a fracture toughness of 1.2–1.9 MPa m$^{1/2}$. Only one set of specimens ($\{1\,1\,0\}\langle 1\,1\,1\rangle$) gave a fracture toughness significantly greater than 1 MPa m$^{1/2}$. Overall, 27 specimens failing from machining flaws with resultant fracture on $\{1\,0\,0\}$ planes gave an average K_{IC} of 0.8 ± 0.1 MPa m$^{1/2}$, while 19 specimens failing from machining flaws with resultant fracture on $\{1\,1\,0\}$ planes gave an average K_{IC} of 0.9 ± 0.1 MPa m$^{1/2}$. The few biaxial tests gave somewhat higher values (Table I).

Specimens that had been indented prior to flexure testing were found by fractography to have failed from fairly well-shaped indentation flaws (Fig. 4). The K_{IC} calculated from their flaw sizes and strengths ranged from the same K_{IC} as found for the DCB tests, as well as from fractography on specimens failing from natural machining flaws, to higher values (Table I). Again, the overall fracture mode varied (e.g. Fig. 5) similar to flexure specimens failing from machining flaws. The most extreme K_{IC} values were from crack propagation on a different plane from the indentation crack (Fig. 5b). Variable hackle-like features also commonly originated from indentation crack boundaries (Fig. 5), but in contrast to this, only occurring rarely, and then much less extensively, from machining flaws.

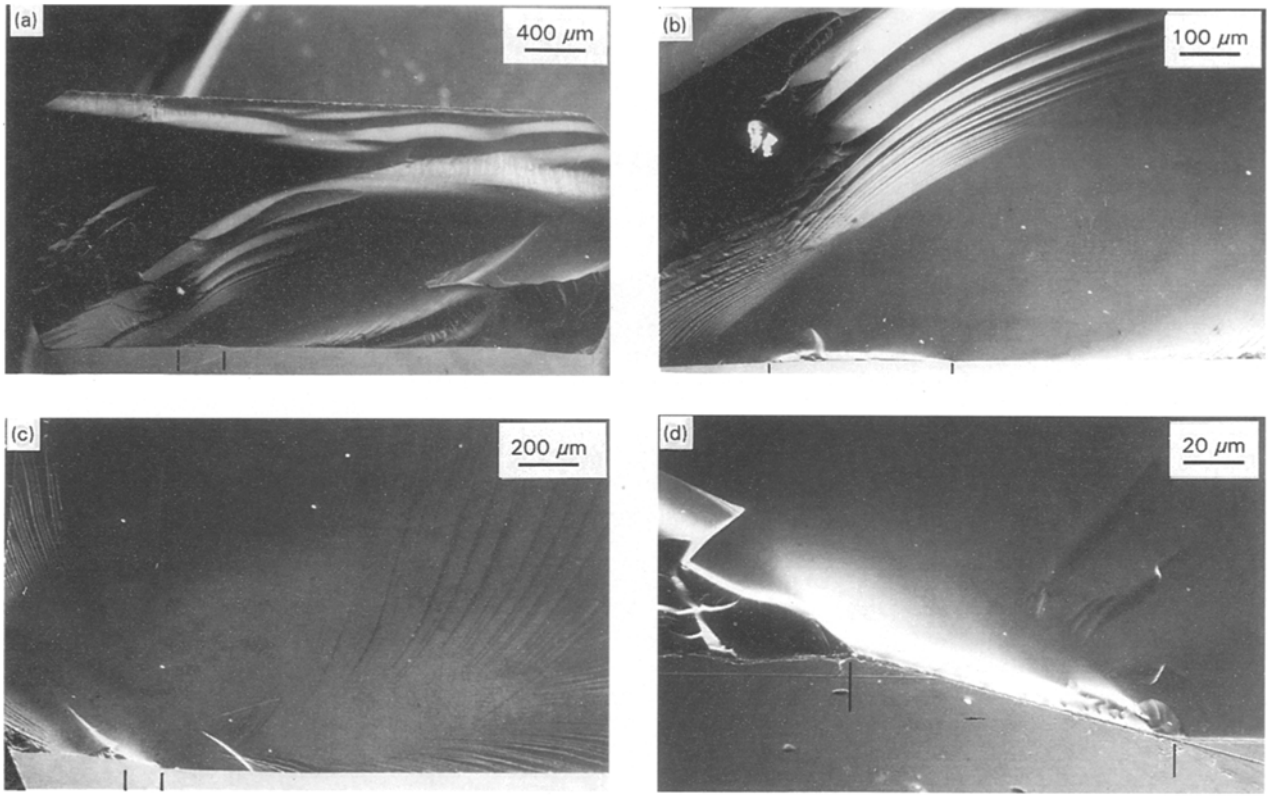

Figure 2 Examples of machining flaw origins in stoichiometric $MgAl_2O_4$ crystals with a {1 1 1} tensile surface and a {1 1 0} tensile axis. (a) Lower and (b) higher magnifications of a specimen in which the fracture origin was in the (1 1 0) plane resulting in a fracture toughness of 1.0 MPa $m^{1/2}$ from a resultant strength of ~172 MPa (22 400 p.s.i.). (c) Lower and (d) higher magnifications of a specimen of the same orientation in which the failure initiating flaw is not on the {1 1 0} plane, yielding a substantially higher fracture toughness of 1.5 MPa $m^{1/2}$ and strength ~260 MPa (37 200 p.s.i.).

TABLE I Comparison of K_{IC} values from DCB tests and machining or indent flaws[a]

Specimen		K_{IC} (MPam$^{-1/2}$) from				
Tensile surface	Tensile axis	DCB tests[b]	Machine flaws[c]		Flaws from intends[c]	
					This study[d]	[19][e]
{1 0 0}	⟨1 0 0⟩	1.0 ± 0.2 (2)	0.8 ± 0.1 (5)	1.1 ± 0.1[f] (4)	1.5 (1)	1.18 ± 0.05
{1 1 0}	⟨1 0 0⟩		0.8 ± 0.1 (26)		1.3 ± 0.2 (8)	
{1 0 0}	⟨1 1 0⟩	1.7 ± 0.5 (4)	0.9 ± 0.1 (7)		1.0 (2)	
{1 1 0}	⟨1 1 0⟩	1.6 ± 0.5 (5)	0.9 ± 0.2 (7)		1.6 ± 0.4 (4)	
{1 1 1}	⟨1 1 0⟩	1.2 ± 0.2 (2)	0.9 ± 0.1 (5)	1.1 ± 0.2[f] (4)		1.90 ± 0.06
{1 1 0}	⟨1 1 1⟩		1.8 ± 0.3 (4)			
{1 1 2}	⟨1 1 2⟩					1.54 ± 0.08

[a] Number of tests shown in parentheses.
[b] Shown here for the equivalent flexure bar orientation, i.e. for the tensile stress normal to the intended fracture plane.
[c] Values as measured, i.e. uncorrected for slow crack growth, which would, on average, increase K_{IC} by ~ 30% (specifically ~ 34% and 26%, respectively, for {1 0 0} and {1 1 0} fracture, showing for elastic differences of ~ 3% betwen 22 and − 196 °C).
[d] Specimens tested as indented (Vickers).
[e] Specimens tested after the indentation (Knoop) itself was machined off the specimen. Values based on 3–6 tests for each orientation.
[f] Biaxial tests, the tensile axis designation in this case refers to that of the plane of fracture (only approximate for the lower, ⟨1 1 0⟩, set of samples).

4. Discussion

Indentation fracture values from this study (Table I) would be reduced by ~15% when corrected for greater flaw depth to specimen thickness effects [9], bringing these values somewhat closer to those for machining flaws. However, with or without such corrections, the indentation fracture values of this study are in the same range as those of Stewart and Bradt [6] (using Knoop indents which were machined off, Table I). In turn, these values are consistent with those of 1.3–1.7 MPa $m^{1/2}$ measured by indentation techniques on stoichiometric crystal specimens of unspecified orientation (but presumably for {1 0 0} fracture) by other investigators [10, 11]. Results for Verneuil

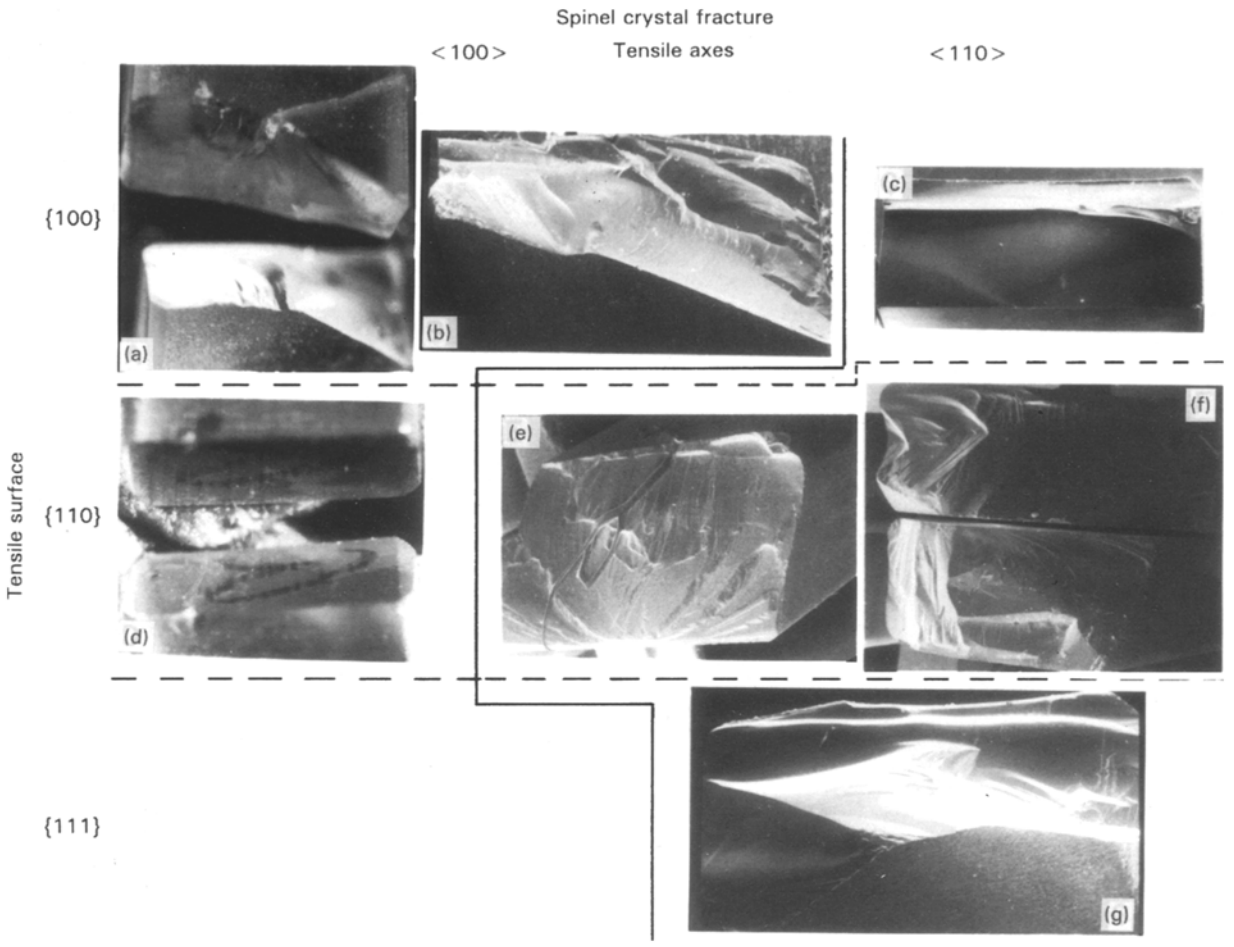

Figure 3 Examples of overall fracture mode for spinel crystals of various tensile surfaces and orientations as indicated.

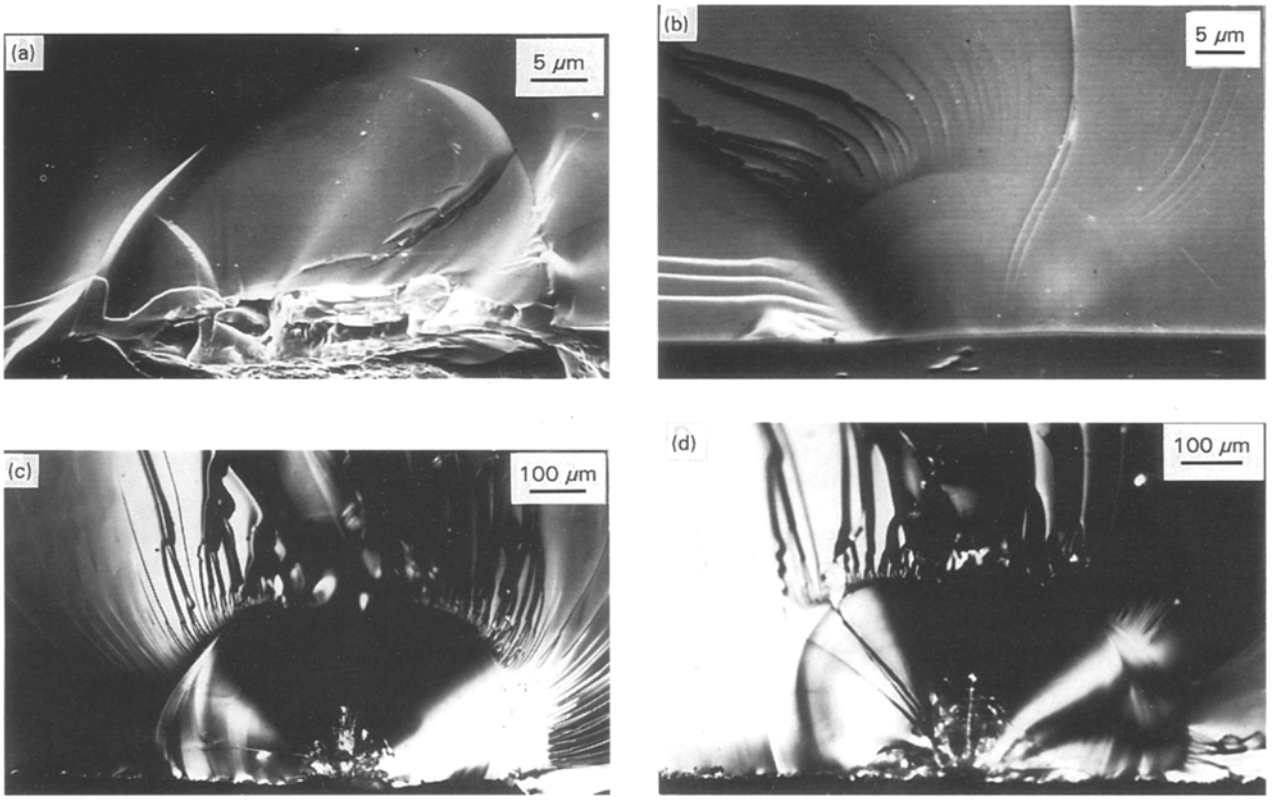

Figure 4 Examples of indent fracture origins in stoichiometric $MgAl_2O_4$. K_{IC} (MPa $m^{1/2}$) and the tensile surfaces and tensile axis: (a) 1.0, {100}⟨100⟩; (b) 1.0, {100}⟨110⟩; (c) 1.2, {110}⟨110⟩; (d) 1.4, {110}⟨110⟩.

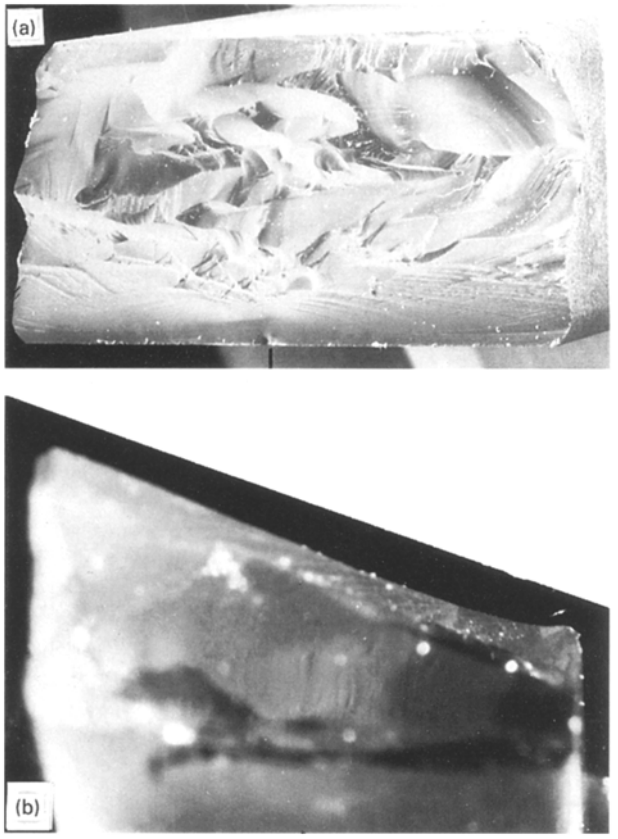

Figure 5 Overall fracture mode from stoichiometric $MgAl_2O_4$ crystals failing from hardness indents. (a) $K_{Ic} = 0.9\ MPa\,m^{1/2}$, $\{1\,1\,0\}\langle 1\,1\,1\rangle$, $\sigma_f = 28.3 \times 10^3$ p.s.i.; (b) $K_{Ic} = 1.5\ MPa\,m^{1/2}$, $\{1\,1\,0\}\langle 1\,1\,1\rangle$, $\sigma_f = 26.6 \times 10^3$ p.s.i.

grown $3Al_2O_3 \cdot 1MgO$ crystals oriented for normal fracture on $\{1\,0\,0\}$ planes are also in a similar range, i.e. $1.5\ MPa\,m^{1/2}$ (using a double torsion–load relaxation method for as-grown, unprecipitated crystals) [12] and 1.5 and $1.9\ MPa\,m^{1/2}$, respectively, using notched-beam (NB) and indentation (Vicker's, 2 kg) [13]. The indentation values cover the broadest range, even for normal $\{1\,0\,0\}$ fracture, but show no trend with whether or not the indent was machined from the specimen before it was fractured [4, 9, 10]; indicating the presence or absence of the actual indent for testing of these materials does not appear to be critical. The values from indentation tests, while tending to be somewhat higher than for other tests for the same fracture plane, generally agree with the relative K_{IC} trend, i.e. K_{IC} being lowest for $\{1\,0\,0\}$ fracture.

In the DCB tests, only the $\{1\,0\,0\}\langle 1\,0\,0\rangle$ oriented samples propagated in the proper fashion, i.e. along a fracture surface both nominally parallel with the axis, and perpendicular to the plane, of the specimen. These values agree well with other tests for the same fracture plane. The other three DCB specimen orientations, while each favouring failure on a $\{1\,1\,0\}$ surface, fractured on or very close to a $\{1\,0\,0\}$ surface. While these results correctly indicate that the $\{1\,1\,0\}$ plane is less favoured for fracture, i.e. has a higher K_{IC}, the differential K_{IC} implied is misleadingly high for at least three reasons. First, as previously noted and discussed in more detail elsewhere [9], the $\{1\,0\,0\}\langle 1\,1\,0\rangle$ DCB specimen did result in crack propagation on essentially the $\{1\,1\,0\}$ plane at higher velocities, indicating a potentially closer balance between fracture on the $\{1\,0\,0\}$ and $\{1\,1\,0\}$ plane than the $\sim 70\%$ higher DCB K_{IC} values for this latter orientation relative to the $\{1\,0\,0\}\langle 1\,0\,0\rangle$ DCB results. Secondly, the three specimens failing on $\{1\,0\,0\}$ planes that were either not parallel to the long direction of the DCB specimen or perpendicular to its plane, need to be corrected (to the extent possible) for their non-normal behaviour. A minimal correction would be based on the actual areas created, which in this case would typically be $\sim 40\%$ greater. Because K_{IC} depends upon the square root of γ, which in turn depends on the area created, this would mean that the minimum correction would be $\sim 20\%$. This would reduce $\{1\,1\,0\}$ K_{IC} to between $\sim 0\%$ and $\sim 40\%$ greater than that for the $\{1\,0\,0\}$ plane. However, this correction is minimal because it involves mixed-mode failure which typically requires corrections greater than those based on surface area alone.

Most extensive and self consistent were the values from fractography of strength tests of single crystal bars. While reasons for the differences between the uniaxial and (the much more limited) biaxial crystal tests are not fully known (some variation in the fracture plane, especially for $\{1\,1\,0\}$ fracture may be a factor), the differences are limited. Combining the uniaxial and biaxial fractography data of this study, as well as recent 22 °C (for comparison with those in liquid nitrogen) tests [5] giving two values for $\{1\,0\,0\}$ fracture (averaging $1.1 \pm 0.2\ MPa\,m^{-1/2}$) and seven values for $\{1\,1\,0\}$ fracture (averaging $1.3 \pm 0.2\ MPa\,m^{-1/2}$), gave overall averages of $1.0 \pm 0.2\ MPa\,m^{-1/2}$ for 31 $\{1\,1\,0\}$ fractures and $0.9 \pm 0.2\ MPa\,m^{-1/2}$ for 34 $\{1\,0\,0\}$ fractures. These overall average values from fractography are even more consistent with data from other tests without complications, e.g. DCB tests for $\{1\,0\,0\}$ fracture. All of these values are also consistent with K_{IC} values from machining flaw origins in single grains in large-grain $MgAl_2O_4$ bodies [14].

A key issue for fractographically obtained (as well as some other) data is the possible impact of slow crack growth (SCG) that may have occurred. Recent tests [5] show that SCG does occur in $MgAl_2O_4$ crystals, with strengths of crystal bars with a $\langle 1\,0\,0\rangle$ tensile axis in liquid nitrogen being $37\% \pm 6\%$ greater than for testing in air at 22 °C. Similarly, the strengths of crystal bars with $\langle 1\,1\,0\rangle$ tensile axis was $26\% \pm 6\%$ greater. Evaluation confirmed that the flaw boundaries observed in fractography are those of the original machining flaws; i.e. not reflecting SCG (in other words, K_{IC} calculated from flaw dimensions on strengths were higher, e.g. by $\sim 30\%$, than at 22° C). Thus, the fractographically determined K_{IC} values should be scaled with the strength increases in liquid nitrogen (reduced by $\sim 3\%$ to allow for increased Young's moduli at –196 °C). The overall K_{IC} averages for $\{1\,0\,0\}$ and $\{1\,1\,0\}$ fracture would be increased, respectively, to ~ 1.21 and $1.28\ MPa\,m^{-1/2}$. Thus, the gap between $\{1\,1\,0\}$ and $\{1\,0\,0\}$ fracture is reduced, but the order is consistent. The K_{IC} value for $\{1\,1\,0\}$

fracture being similar to, but somewhat greater than for {1 1 0} fracture of $MgAl_2O_4$, is supported by theoretical results. Thus, application of Gilman's theory of crystal fracture energies [15] to $MgAl_2O_4$ and MgO, predicts [4] {1 1 0}/{1 0 0} K_{IC} ratios of 1.3–1.4 and 1.8, respectively, i.e. a lower ratio for $MgAl_2O_4$ than MgO.

Other experimental results also support the relative closeness of {1 0 0} and {1 1 0} K_{IC} values for $MgAl_2O_4$, e.g. *vis-à-vis* MgO. Thus, fracture initiated in $MgAl_2O_4$ on the {1 0 0} plane normal to the tensile axis can branch off on to favourably oriented {1 1 0} planes. Studies of other single crystals have shown that crack branching can readily occur on to lower K_{IC} planes from higher K_{IC} planes, e.g. from {1 1 0} on to {1 0 0} for MgO [1, 2, 15], but not the reverse, implying a much closer balance in $MgAl_2O_4$. Further, extensive $MgAl_2O_4$ failure has been observed to occur on both {1 0 0} and {1 1 0} planes in properly oriented samples. Again, this implies that the K_{IC}s are similar, because in other crystal systems, e.g. MgO, where the secondary {1 1 0} cleavage plane apparently has a higher K_{IC}, large-scale (i.e. macro) failure is not observed to occur on this plane, only on the {1 0 0} plane. Finally, it should be noted that while the K_{IC} observed in the $MgAl_2O_4$ flexure samples was not dependent upon the orientation about a common tensile axis, (i.e. on what tensile surface the specimens had), the crack propagation character clearly was dependent on it. As noted above, in proper orientations, cleavage failure could be obtained on either the {1 1 0} or {1 0 0} planes. However, other orientations about these respective tensile axes gave more complex failure, indicating considerable limitations on obtaining large-scale fracture on either of these planes, especially the {1 1 0} plane. This, combined with the apparent greater sensitivity of the DCB tests to elastic anisotropy (discussed below), appears to be a reason for higher values and less failure on the {1 1 0} plane in such tests.

Because fractographic methods are not nearly as widely used as other tests such as DCB, notched-beam (NB) and indentation tests, it is useful to compare these further. First, it is important to remember that DCB tests did not function properly for other than {1 0 0} fracture, and other larger scale crack tests would be expected to have similar problems, which were not encountered in fractography of strength test specimens. Second, fractography results have been extensively shown to agree with other, e.g. DCB, results for polycrystalline bodies when other factors such as microstructural stress are not significant in fracture [16–18].

The third reason for accepting the fractography results where they differ from other tests is the relationship between single crystal and polycrystalline values. Reported polycrystalline $MgAl_2O_4$ K_{IC} values measured by various techniques, e.g. both indentation fracture [19] and DCB [20], fractography [16], and various notched beam methods [21–23] range from 1.4–2.2 MPa $m^{1/2}$, averaging $\sim 1.9 \pm 0.2$ MPa $m^{1/2}$. This includes values for press-forged $3Al_2O_3 \cdot 1MgO$ crystals that were thus polycrystalline (but without precipitation) in good agreement with other theoretically dense polycrystalline spinal bodies. Comparing $MgAl_2O_4$ single-crystal values, especially for the easiest fracturing plane (i.e. {1 0 0}) to polycrystalline values indicates lower polycrystalline to single-crystal K_{IC} ratios compared to the other few materials for which suitable single and polycrystalline materials are available. Thus, a compilation [24] of such data for Al_2O_3 and MgO shows that the polycrystalline to the easiest single-crystal fracture (p/c) K_{IC} ratios are, respectively, in the range of > 1.5 to < 2.5 and $\geqslant 2.4$ to $\leqslant 3.2$. The latter range for MgO seems particularly pertinent for comparison, because it has the same primary ({1 0 0}) and secondary ({1 1 0}) cleavage planes as $MgAl_2O_4$, as well as similar Young's modulus (but somewhat less elastic anisotropy) to that of $MgAl_2O_4$ [3]. Similarly, fairly comprehensive studies of fracture of CaF_2 single crystals (i.e. {1 1 1} cleavage), and press-forged and dense hot-pressed bodies [19, 20] give similar p/c K_{IC} ratios of 1.7–2.7. For $MgAl_2O_4$, the p/c K_{IC} ratios from indentation tests range from $\leqslant 1$ to < 1.9, i.e. abnormally low to somewhat low. At the other extreme, DCB tests (for properly oriented tests, problems with other orientations were discussed earlier) give p/c K_{IC} ratios of $\geqslant 1.4$ to $\leqslant 2.2$, very similar, if not identical, to the fractography results, and fairly consistent with Al_2O_3 and MgO ratios. The limited DT and NB data are in between these extremes, i.e. with ratios in the range of 1.1 to < 1.5. The only other data indicating such low polycrystalline to single-crystal (easiest fracture) K_{IC} ratios, are some limited data for SiC. Limited measurements of single crystals using NB [24], indentation, or both [25] techniques, give K_{IC} of $\sim 3.2 \pm 0.2$ MPa $m^{1/2}$, identical or very close to widely determined polycrystalline values, i.e. p/c K_{IC} ratios of ~ 1. On the other hand, Rice [18, 26] has reported K_{IC} values for easy fracture of SiC crystals based on limited fractographic studies of ~ 1.6–2.1 MPa $m^{1/2}$, thus giving p/c K_{IC} ratios of ~ 1.5 to $\geqslant 2$. Thus, fractographic results have given more consistent p/c K_{IC} values than other techniques.

Another factor that needs to be considered in applying a K_{IC} test to single crystals is the validity of the test assumptions to the crystal studied. The indentation test presents significant uncertainties, which are probably partly related to variations in the indentation fracture method. The indentation method basically assumes that the material is isotropic in its deformation, elastic stresses and fracture characteristics. However, single crystals often deviate substantially from isotropy of deformation, and can often have considerable elastic anisotropy. Such isotropy–symmetry factors, at a minimum may effect the "calibration" (i.e. proportionality constant) needed for the direct indentation test, and hence could be a major reason for the high (1.9 MPa $m^{1/2}$) values of Lewis *et al.* [13]. These factors could also be significant for the indentation fracture tests giving high values in this and other single-crystal studies [8, 27, 28]. Effects of deviations from isotropy may, in turn, influence factors such as crack shape, curvature, and residual stresses and resultant propagation (e.g. variable hackle-like

features originating from the indentation crack boundaries (Fig. 5). Further, depending on both the crystal system and the orientation of specific specimens, resultant fracture surfaces may not be symmetric with the indent. This may even be a problem for indents oriented to introduce cracks on secondary fracture planes because of frequent propensities to branch off such planes, especially with larger cracks. This was a problem for some indentation fracture tests, e.g. Fig. 5b.

The NB tests, which often give broader variations (usually higher values than other tests) [29–31] assume a sharp, essentially slit crack at the base of the notch, which is seldom verified by fractography. Where such verification has been made (in glass [30], and ZrO_2 crystals [31]), failure is often found to occur from cracks approaching a half-penny instead of a slit character, with the former giving K_{IC} values up to twice those of the latter, as expected. Such crack variations are quite likely in NB tests of single crystals (especially for fracture along easy cleavage planes). Thus, NB single-crystal tests need to be considered with caution, especially when they are higher than for other tests. NB-tested specimens should be fractographically examined to determine the failure-initiating flaw character.

Finally, elastic anisotropy of $MgAl_2O_4$ crystals may be a factor in DCB and related tests. As discussed, for example, by Walsh [32] and Green *et al.* [33], elastic anisotropy in orthotropic materials (as would be the case for at least some $MgAl_2O_4$ orientations) can have significant affects on DCB (and probably DT) results, substantially more so than on flexure or tensile results. Applying the simplified analysis proposed by Green *et al.* for such orthotropic materials to $MgAl_2O_4$ would predict about a 20% difference between a DCB specimen oriented with its length in a $\langle 1\,0\,0 \rangle$ versus a $\langle 1\,1\,0 \rangle$ direction. The $\langle 1\,0\,0 \rangle$ values are about 10% higher than those obtained if the material were isotropic, and the $\langle 1\,1\,0 \rangle$ values are $\sim$10% lower than the isotropic values. Thus, the true difference between $\{1\,0\,0\}$ and $\{1\,1\,0\}$ K_{IC} probably is only of the order of 10–20% (i.e. reasonably consistent with the values calculated from machining flaw fracture origins) from flexure testing.

5. Conclusion

K_{IC} values calculated for failure from machining flaws in stoichiometric $MgAl_2O_4$ crystals indicate a close balance between K_{IC} for the $\{1\,0\,0\}$ and $\{1\,1\,0\}$ planes, consistent with both of these being cleavage planes. The overall values for these planes are, respectively, ~ 1.2 and ~ 1.3 MPa m$^{1/2}$, i.e. indicating somewhat easier failure on the $\{1\,0\,0\}$, as do theoretical considerations and other experimental observations. DCB results agreed well with the machining flaw results for fracture on the $\{1\,0\,0\}$ planes, but gave substantially higher values for attempted fracture on the $\{1\,0\,0\}$ plane. These values are high because DCB specimens actually fractured on the $\{1\,1\,0\}$ plane only under special circumstances, normally failing on $\{1\,0\,0\}$. Area or mixed-mode corrections account for at least part of these differences. However, greater sensitivity of the DCB-type tests to elastic anisotropy (e.g. relative to flexure testing) may also be a factor. Greater favouring of failure on $\{1\,0\,0\}$ rather than $\{1\,1\,0\}$ in DCB tests is attributed to slightly higher $\{1\,1\,0\}$ versus $\{1\,0\,0\}$ K_{IC} and the attendant greater difficulty of large-scale crack propagation on $\{1\,1\,0\}$ versus $\{1\,0\,0\}$.

Other conventional K_{IC} tests often present similar anisotropy and scale of crack-propagation problems in single crystals, especially indentation methods where isotropy–symmetry requirements may often not be fully satisfied. Thus, indentation-based methods of determining K_{IC}, both in this and other studies, generally resulted in values ranging from approximately the same as those found from K_{IC} calculated from machining flaw fracture origins, to the highest values observed. The higher indentation K_{IC} values appear to occur, whether or not the indentation had been machined off in order to minimize residual stresses from the indent itself. An important reason why the indentation, as well as other (including some DCB), values are felt to be too high is that they are too close to, e.g. actually overlapping, polycrystalline results. Thus, fractographic methods of determining K_{IC} deserve more attention and the use of conventional methods deserves more caution and analysis when applied to single crystals.

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