The Toughness of Polycrystalline MgAl_2O_4

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多結晶 MgAl_2O_4 の破壊靱性値

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The effect of the measurement technique on the apparent fracture toughness, or K_{IC} value of a transparent polycrystalline MgAl_2O_4 spinel was investigated. Results from macronotched specimens are compared with those of microflaw techniques from room temperature to 1000°C. Contributions of the process zone wake in terms of grain bridging and interlocking frictional effects are qualitatively addressed and the effects of grain size are also discussed.

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1. Introduction

The importance of the crack tip frontal process zone and its following wake on the toughness of ceramics has been recognized through fracture mechanics studies of different materials which develop an extensive process zone as a consequence of their susceptibility to stress-induced microcracking and/or phase transformations in the vicinity of the crack tip. The rising R -curve behavior of increasing toughness parameters accompanying incremental crack growth, which results from the development of the process zone and the wake, have been reported and studied. Measured toughness values from test specimens which support a fully developed process zone and an extensive wake are in many cases considerably larger than for specimens without a fully developed process zone wake.¹⁾⁻¹⁴⁾

The rising R -curve behavior of ceramics, as a consequence of the process zone and its wake, implies that the measured toughness values may be influenced by the size of the test specimen, the geometry of the crack tip, the particular test method, etc., as well as the characteristic microstructure of the ceramic materials.¹⁴⁾ For example, the stress shielding effect of the process zone wake on the main crack may be expected to be different in macro-notched specimens such as the double cantilever beam, the double torsion plate,

the chevron-notched short bar, the single edge notched bend bar, etc. than in micro-flaw test specimens with microhardness indentation induced flaws or cracks. This is because the dimensions of the process zone and the wake, its length, its thickness and their geometry are different in macro-notched and micro-flawed specimens.

One objective of this study is to investigate and report the effect of the testing method on the measured toughness in relation to the crack tip process zone and its wake using a transparent stoichiometric polycrystalline magnesium aluminate spinel (MgAl_2O_4) at room temperature and at elevated temperatures.

2. Experimental procedure

A commercial hot-pressed transparent polycrystalline spinel, MgAl_2O_4 (Coors Ceramics, Golden, CO, USA), was used as the test material. As a consequence of the processing, grain sizes ranged from about 2 to 400 μm in diameter as observed on a polished surface. The toughness values of this material were measured by four different test methods, including the straight-through notched (STN) and chevron-notched (CHV) 3-point bend bar methods which are representative of macronotched test specimens and the Vickers indentation (VIM) and the controlled surface flaw (CSF) methods which are characteristic of micro-flawed test specimens. Toughness measurements using the first three methods were completed in the present study, while the toughness values previously reported by

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one of the authors (RCB) were determined through a fourth method (controlled surface flaw).¹⁵⁾ Prior to testing, the as-received spinel was annealed at 1400°C for 1 hour in air to reduce any residual stresses that may have existed in the billets. The billets were then diamond machined to the appropriate test specimen dimensions. The dimensions of the chevron-notched bend bar specimens are shown in Fig. 1. The chevron notch was sawed using a 0.3mm thick diamond blade. The $K_{\text{IC}}(\text{CHV})$ toughness was determined from the maximum fracture load (P_{max}) during steady-state crack extension, using¹⁶⁾⁻¹⁸⁾

$$K_{\text{IC}}(\text{CHV}) = \frac{P_{\text{max}} Y_{\text{min}}}{B\sqrt{W}} \quad (1)$$

where B and W are the thickness and the width of the specimen, and Y_{min} is the minimum value of the dimensionless stress intensity factor, which was determined using Bluhm's slice model with a shear correction factor, $k=1.8$.¹⁸⁾ The $K_{\text{IC}}(\text{CHV})$ measurements were completed over a temperature range from room temperature to 1000°C in air at a 0.1 mm/min crosshead speed of the commercial testing machine (Instron Corp., Canton, MA., USA). In this instance and throughout the remainder of this paper, the term K_{IC} will be used to designate the toughness measurement even though the measured K_{IC} values may not be true K_{IC} values, independent of the test method and crack length.

The overall dimensions of the straight-through macro-notched 3-point bend bars were the same as those of the chevron-notched specimens, except for the notch geometry. The straight-through notch was sawed with the same 0.3mm thick diamond blade to a relative depth of $(a/W)=0.5$, followed by extending the sawed notch about 100 to 150 μm using a thin razor blade and SiC abrasive grains (60 and 20 μm). The radius of the sharp notch tip was observed to be less than 15 μm . The fracture load (P_c) was measured at room temperature using a crosshead speed of 5 mm/min

to prevent the subcritical slow crack growth prior to the failure. The fracture toughness, $K_{\text{IC}}(\text{STN})$, was then calculated using the formula in ASTM E399. The exact notch length at failure was determined using a microscope and observing the actual fracture surface.

The specimens for the Vickers indentation method (VIM) were $6 \times 6 \times 25 \text{ mm}^3$ bars, one of the surface being polished through 0.3 μm with an Al_2O_3 powder. The induced indentation radial crack length (c) and the impression half-diagonal (a) at room temperature were measured using the microscope on the microhardness tester (Shimadzu, Kyoto, Japan) immediately after removal of the indentation load, $P_{\text{ind}}=9.81 \text{ N}$. The fracture toughness for the Vickers indentation technique, $K_{\text{IC}}(\text{VIM})$ was then determined using:¹⁹⁾

$$K_{\text{IC}}(\text{VIM}) = 0.02 E^{1/2} P_{\text{ind}}^{1/2} a^{-1/2} (c/a)^{-3/2} \quad (2)$$

where E is Young's modulus, 258 GPa for polycrystalline spinel at room temperature.²⁰⁾ To examine the effects of the presence of grain boundaries on the indentation radial crack length, i.e., on the measured $K_{\text{IC}}(\text{VIM})$ value, a number of different measurements were made with the Vickers indenter tip directly in the center of different size individual grains with diameters (d_g) of 300, 100, 50, 20 μm , and then also actually on a grain boundary ($d_g=0$). The average lengths of (a) and (c) obtained from ten separate indentations for each grain diameter were then used to calculate the $K_{\text{IC}}(\text{VIM})$ values for each of the grain sizes. The details of the controlled surface flaw measurements are summarized elsewhere.^{15), 21)}

3. Result and discussion

Vickers indentation impressions and their radial cracks are shown in Fig. 2. for a 50 μm grain and for a 300 μm diameter grain. Interactions between the indentation produced cracks and the grain boundaries inhibit crack extension, and actually arrest or pin the radial cracks on some occasions. This crack/grain boundaries interaction results in much shorter radial crack length and higher apparent fracture toughness values. The $K_{\text{IC}}(\text{VIM})$ toughness values at room temperature are plotted versus the indentation measurement grain diameter (d_g) in Fig. 3. The toughness values for the cleavage of single crystal spinel on the different crystallographic planes ($\{100\}$, $\{110\}$, and $\{111\}$) as measured by the controlled surface flaw method²¹⁾ and the $K_{\text{IC}}(\text{CHV})=1.95 \pm 0.05$ and $K_{\text{IC}}(\text{STN})=1.27 \pm 0.02 \text{ MPa}\cdot\text{m}^{1/2}$ values are also included for comparison. The average values of ($2a \text{ } \mu\text{m}$, $2c \text{ } \mu\text{m}$) for the different diameter grains are (34.3, 111),

Fig. 1. The three-point-bend specimen with a chevron notch.

Fig. 2. Vickers indentation impressions in a small grain ($\approx 50 \mu\text{m}$ diameter) and a large grain ($\approx 300 \mu\text{m}$ diameter), bottom.

Fig. 3. Fracture toughness versus temperature for the chevron-notched specimen (CHV) and by the controlled surface flaw method (CSF).

(35.5, 109), (34.4, 96), (34.8, 91) and (35.0, 88) for (d_g) $\approx$ 300, 100, 50, 20, and 0 μm , respectively. The apparent $K_{IC}(\text{VIM})$ value increases with a decrease in the individual test grain size, and eventually approaches the $K_{IC}(\text{CHV})$ toughness value for this polycrystalline spinel. In the limit of the largest grain size, the measured $K_{IC}(\text{VIM})$ value is intermediate to the $K_{IC}(100)$ and $K_{IC}(110)$ toughness values for the cleavage of

single crystal spinel.

The apparent $K_{IC}(\text{VIM})$ toughness value varies as a function of the grain diameter in which the indentation is made. This may be expected since the indentation fracture mode should be similar to that for a single crystal when $(2c/d_g) \ll 1$, i.e., for a very large grain size, but should approach the value for polycrystalline spinel when $(2c/d_g) \gg 1$. It is also of interest to note that the spontaneous propagation of these indentation radial cracks in individual grains which appear to be arbitrary crystallographic directions is similar to that for the weakest or lowest toughness cleavage plane, the {100} for MgAl_2O_4 spinel, as may be inferred from the $K_{IC}(\text{VIM})$ at $d_g = 300 \mu\text{m}$ in Fig. 3. Another interesting experimental result depicted in Fig. 3, is the difference between the $K_{IC}(\text{CHV})$ and $K_{IC}(\text{STN})$ toughness values, where $K_{IC}(\text{CHV}) \approx 1.5 K_{IC}(\text{STN})$. The fracture mechanisms or processes associated with this apparent discrepancy in toughness measurements will be addressed in a later section.

The results in Fig. 3, are also an indication of the effect of grain size on the toughness of polycrystalline MgAl_2O_4 spinel. Several investigators^{(22), (23)} have presented polycrystalline ceramic toughness/grain size plots of a sigmoidal shape, not unlike the $K_{IC}(\text{VIM})$ results in Fig. 3, where the polycrystalline toughness exceeds the single crystal toughness. In general, the results of this study support that general trend, but only if the single crystal toughness is interpreted to be the lowest toughness cleavage plane. It is evident that fracture on "non-easy" cleaving crystallographic planes such as the {111} in spinel, may be of a comparable toughness to that of the material in polycrystalline form, for the measurements of $K_{IC}(\text{CHV})$ and that for cleavage $K_{IC}(\{111\})$ are virtually equivalent in this instance. Since fracture on crystallographic planes other than the {100}, {110}, and {111} may also be expected to occur in certain circumstances, it is not unreasonable to speculate that there may even exist some single crystal cleavage toughnesses for MgAl_2O_4 spinel which may exceed the polycrystalline values. One can further speculate that the same might occur for other ceramic materials.

The temperature dependence of the $K_{IC}(\text{CHV})$ toughness between room temperature and 1000°C is shown as the data points from individual measurements in Fig. 4. The $K_{IC}(\text{CHV})$ values decrease monotonically with increasing temperature from 1.95 $\text{MPa} \cdot \text{m}^{1/2}$ at room temperature to 1.29 $\text{MPa} \cdot \text{m}^{1/2}$ at 1000°C. In Fig. 4, the fracture toughnesses previously obtained using the

Fig. 4 Fracture toughness versus temperature for the chevron-notched specimen (CHV) and by the controlled surface flow method (CSF).

Fig. 5. Load-loadpoint displacement relations of spinel at two different test temperature.

controlled surface flow method, $K_{Ic}(\text{CSF})$, are represented by the broad band.^{15), 20)} It is evident in Fig. 4. that there exists excellent agreement between $K_{Ic}(\text{CHV})$ and $K_{Ic}(\text{CSF})$ values in spite of the differences in the crack sizes and crack tip geometries during the two different tests.

Crack propagation in the chevron-notched 3-point bend bar specimens was always stable, two examples of which are shown in Fig. 5. as load (P) versus loadpoint displacement (u) curves, one at room temperature and one at 1000 $^{\circ}\text{C}$. In this instance the loadpoint displacement was determined from the crosshead displacement of the testing machine and the compliances of the testing machine and the loading fixtures. The extensive inelastic contribution to the crack propagation process causes the (P - u) curve at 1000 $^{\circ}\text{C}$ to be substantially broader than the (P - u) curve at room temperature. The work of fracture, γ_{wof} , a common nonlinear fracture surface energy parameter, can be determined directly from the area under the (P - u) curves using conventional methods. In ceramic materials containing a microcracked process zone and wake, $2\gamma_{\text{wof}}$ can be expressed by²⁴⁾

$$2\gamma_{\text{wof}} = \int_0^s \left[2\gamma_s + \frac{\partial \Gamma_m}{\partial A} + \frac{\partial U_{\text{re}}}{\partial A} + \frac{\partial U_d}{\partial A} \right] dA/S \quad (3a)$$

OR

$$2\gamma_{\text{wof}} = K_{Ic}^0/E' + \langle \partial U_{\text{nl}}/\partial A \rangle \quad (3b)$$

where γ_s , $\partial \Gamma_m$, ∂U_{re} , ∂U_d , and ∂U_{nl} are, respectively, the elastic surface energy of the main crack, the energy consumption by stress-induced microcracks, the residual elastic strain energy stored in the process zone wake, the inelastic energy dissipation and the nonlinear energy consumption during the incremental increase of the main crack surface (dA). The (S) term represents the total fracture surface area. In Eq. (3b), $2\gamma_s$ can be assumed to be equal to the critical strain energy release rate, $g_c (= K_{Ic}^0/E')$, where K_{Ic}^0 is the fracture toughness of a specimen without any wake, E' is $E/(1-\nu^2)$ where E is the modulus of elasticity and ν is Poisson's ratio, respectively:

$$\langle \partial U_{\text{nl}}/\partial A \rangle = \int_0^s \left[\frac{\partial \Gamma_m}{\partial A} + \frac{\partial U_{\text{re}}}{\partial A} + \frac{\partial U_d}{\partial A} \right] dA/S$$

Rewriting (3b) yields:

$$\langle \partial U_{\text{nl}}/\partial A \rangle / (K_{Ic}^0/E') = \left[\frac{2\gamma_{\text{wof}}}{K_{Ic}^0/E'} \right] - 1 \quad (4)$$

where the right-hand side of Eq. (4) defines the nonlinear energy consumption relative to the elastic energy consumption, K_{Ic}^0/E' . This is presented in Fig. 6. versus temperature to 1000 $^{\circ}\text{C}$, where K_{Ic}^0 and E' were approximated by $K_{Ic} (= K_{Ic}(\text{CHV})$ in Fig. 4.) and E as a function of temperature,²⁰⁾ respectively. This approximation would be enough to demonstrate the dependence of the nonlinear energy consumption on temperature. It is evident that the nonlinear energy consumption increases rapidly at higher temperatures over the region which corresponds to the steep decrease on K_{Ic} shown in Fig. 4. and also to the broadening of the (P - u) curve depicted in Fig. 5. This presence of a significant nonlinear energy consumption at elevated temperatures suggests that inelastic microcracking in the frontal process zone and grain bridging in the wake region may be occurring in this polycrystalline spinel during steady-state crack propagation. In addition, it is notable that the nonlinear contribu-

Fig. 6. The dependence of nonlinear energy consumption ($2\gamma_{\text{wof}}/(K_{Ic}^0/E')$).

tion to energy consumption is not zero ($2\gamma_{\text{wof}} \approx 2.5 \cdot (K_{\text{Ic}}^2/E)$), even at room temperature, which implies that the significant inelastic microcracking and the grain bridging processes are probably occurring in the crack tip region as well as along the fracture surfaces, even at room temperature.

Detailed examination of Figs 3-6 on the basis of the *R*-curve concept reveals several interesting features concerning nonlinear fracture processes in the polycrystalline MgAl_2O_4 spinel. At room temperature, the $K_{\text{Ic}}(\text{CHV})$ toughness of the specimen with a well-extended wake is about 50% larger than the $K_{\text{Ic}}(\text{STN})$ fracture toughness value which is supposedly not influenced by the presence of the process zone wake. This is similar to the toughness increment $K_{\text{Ic}}(\text{CHV}) \approx 1.3 \cdot K_{\text{Ic}}(\text{STN})$ which has been observed for a dense polycrystalline Al_2O_3 .²⁵⁾ The MgAl_2O_4 fracture toughness increment, $\Delta K = K_{\text{Ic}}(\text{CHV}) - K_{\text{Ic}}(\text{STN}) = 1.95 - 1.27 = 0.68 \text{ MPa} \cdot \text{m}^{1/2}$, could possibly result from the stress shielding of the main crack by the process zone wake which develops during the steady-state crack extension in the chevron-notched specimen. The increment of ΔK for this polycrystalline MgAl_2O_4 spinel can perhaps be qualitatively explained by the mechanisms of grain bridging and interlocking, or perhaps even surface forces between the fracture surfaces in the process zone wake developed behind the main crack tip. In fact, the chevron-notched specimens did not separate into two distinct pieces at the end of steady-state crack growth, but rather the two pieces remained joined as one via a weak interaction between the two fracture surfaces of the test specimen. The optical micrographs of polished side surfaces of the fractured specimen are shown in Fig. 7., where the grain bridges and the stress-induced micro-

racks in the wake region are clearly seen. These experimental facts support the concept of grain bridging and interlocking as a possible mechanism for the incremental ΔK observed for the chevron-notched specimens.

In Fig. 3, the $K_{\text{Ic}}(\text{VIM})$ at $(2c/d_g) \rightarrow \infty$ approaches the $K_{\text{Ic}}(\text{CHV})$ value, the toughness for a polycrystalline specimen with a well-extended process zone and wake. This experimental results suggest that a reasonably well-developed process zone wake may also be associated with the formation of the Vickers radial cracks in the finer grain size specimens. Initially, this may appear puzzling, as it is somewhat difficult to visualize a well-developed microcrack process zone with an accompanying wake in accordance with the formation of these small indentation radial cracks, for these indentation cracks are surrounded by only a few grain facets. Even if there are grain boundary microcracks along the indentation radial cracks, the dimensions of those microcracks would probably be of the same order of size of the radial cracks ($2c$), requiring the microcrack density to be quite low. The agreement between $K_{\text{Ic}}(\text{CHV})$ and $K_{\text{Ic}}(\text{VIM})$ at $(2c/d_g) \rightarrow \infty$ could also occur if the stress-induced microcracks around the small Vickers radial crack have much stronger shielding effect on the stress intensity than those of a macro-notched specimen. It is also possible that the extent of the microcracked shielding zone is so small that it becomes fully developed even in the vicinity of these small indentation flaws.

From Fig. 4., it is quite evident that for the controlled surface flaw specimens the process zone wake must be fully developed during the course of the Knoop microhardness indentation and the fracture test even at room temperature.^{15), 20)}

Fig. 7. Optical micrographs of the wake region of the fractured specimen.

Of course, the process zone wake in the chevron-notched specimens becomes fully developed at each testing temperature. The basic nature of the process zone wakes formed at room temperature and at elevated temperature could be different because of the marked nonlinear energy consumption at elevated temperatures, as illustrated in Figs. 5. and 6. However, in spite of those differences for the wake formation between the controlled surface flaw specimen and chevron-notched specimens, there exists quite excellent agreement between the $K_{\text{Ic}}(\text{CHV})$ and $K_{\text{Ic}}(\text{CSF})$ values over this extensive temperature range. It is also interesting that the spread of values, or the range of the controlled surface flaw results is much greater than that for the chevron-notched specimens. This is probably a consequence of the multiple grain averaging effects of the much larger cracks in the chevron-notched specimens.

It is also of value to directly compare the polycrystalline spinel toughness results in Figs. 4-6 with those of Stewart and Bradt²¹⁾ for spinel single crystals at elevated temperatures. Similar to Fig. 6., single crystals of MgAl_2O_4 also exhibit a rapidly increasing toughness contribution with increasing temperature for all crystallographic orientations, but initiating at a much higher temperature range, about 900°-1000°C. Single crystal and polycrystalline specimens both exhibit substantial inelastic contributions to fracture at elevated temperatures; however, the inelastic effects in the two forms of spinel initiate at different temperatures and result in opposite trends in the " K_{Ic} " measurements at high temperatures. In the case of the dislocation plastic flow processes in the single crystals, the fracture toughness increases at elevated temperatures. However, for the microcracking exhibited by the polycrystalline specimens the apparent toughness decreases, even though there is an accompanying increase in the nonlinear energy consumption ($<\partial U_{\text{nl}}/\partial A>/K_{\text{Ic}}^0/E'$) for the complete fracture process. One possibility is that a mechanism which is related to the microcracking phenomenon may promote the linking together of the microcracks to the main crack, thus offsetting any substantial contribution to a real toughness increase.

4. Conclusions

The toughness of polycrystalline MgAl_2O_4 spinel is about the same as that for the cleavage of single crystal spinel on the {100}, {110}, and {111} planes, but exhibits a dependence on the measurement technique. This latter effect is related to the development of the process zone and

its wake and their attendant inelastic effects in the experimental measurements.

The process zone wake of polycrystalline spinel results in $K_{\text{Ic}}(\text{CHV}) \approx 1.5 \cdot K_{\text{Ic}}(\text{STN})$. However, the toughness values determined using the indentation microflaw method with Vickers indentations and also by the controlled surface flaw method agree with the $K_{\text{Ic}}(\text{CHV})$ values over a wide range of temperatures. This suggests that the size of the process zone and its wake are probably only a few grain diameters in extent and that interactions may be very intense across the newly fractured surfaces. The effect of the process zone wake on the incremental $K_{\text{Ic}}(\text{CHV})$ can be qualitatively understood by considering that a component of the stress shielding of the main crack tip exists due to grain bridging and interlocking forces acting across the newly created fracture surfaces.

When the inelastic contributions to the fracture toughness are examined through analysis of the $K_{\text{Ic}}(\text{CHV})$ measurements at elevated temperatures, it is evident that a substantial inelastic contribution rapidly increases or develops at temperatures as low as about 600°C for polycrystalline spinel. However, this increased inelastic energy consumption does not increase the apparent fracture toughness, which rapidly decreases at elevated temperatures.

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