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Short communication

Fracture toughness of acid corroded Y-α-SiAlON ceramics

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Abstract

To unequivocally identify the fundamental mechanism of toughening in an acid corroded Y-α-SiAlON microstructure, an attempt was made in the present work to understand the mode of crack propagation due to indentation fracture in such ceramics deliberately made devoid of amorphous phase after acid treatment. The results established that toughening in such case was governed by grain bridging mechanisms.

Keywords: Engineering ceramics; Acid corrosion; Mechanical behavior

1. Introduction

During last decade, contribution towards improving fracture toughness in otherwise excellent hardness of the α-SiAlON ceramics (α-sialon) has remained as the point of attraction for many workers [1–7]. Among the various approaches, attempts to obtain single phase in situ toughened α-sialon have been reported to exhibit the best optimized combination of these two mechanical properties by the production of elongated grains of the hard phase itself in microstructure [1]. However, the unequivocal identification of the dominant toughening mechanism is far from complete. Toughening mechanisms were proposed to be grain pull-out in self-reinforced Ca-α-sialon [2], high proportion of elongated grains in both Y- and Ca-containing α-sialon [3], crack deflection in self reinforced Li-α-sialon [4], a combination of crack deflection, grain pull-out and debonding caused by the elongated grains in hot pressed Yb-α-sialon [5], the elongated grains in gas pressure sintered Dy-α-sialon [6] and crack bridging in pressureless sintered self-reinforced Y-α-sialon [7]. Attempts to identify the dominant toughening mechanisms unequivocally were far from significant [4,7]. The typical reported [2–7] toughness values covered the range of ~6.2–11 MPa $m^{0.5}$. The particular mode of crack propagation which will be applicable is determined mainly by the differences in grain geometries those arise from its composition, processing techniques, etc. However, in all above cases reported in literature [2–7] the typical microstructure contained some amorphous phases in the grain boundary and triple point region in the matrix phase and the measurement of fracture toughness is done in such microstructures. The crack propagation in majority of such cases is seen to occur through the amorphous phases in grain boundaries. Therefore, the basic objective of the present work was to critically examine the mode of crack propagation due to indentation fracture after removal of such grain boundary amorphous phase so that the fundamental mechanism of toughening in a α-sialon microstructure in such cases can be unequivocally identified. There is no doubt that such knowledge will be very useful to develop in future better, microstructurally engineered α-sialon ceramics for structural applications.

2. Experimental

A composition corresponding to α-sialon ($M_x^{p+}Si_{12-m-n}Al_{m+n}O_nN_{16-n}$, where $x=m/p$, in this case $M=$yttrium) with $m=1.3$, $n=1.4$ and Y-content little above theoretical value of x [8] was taken from Si_3N_4–$Al_2O_3\cdot AlN$ $YN\cdot 3AlN$ system. The starting powders silicon nitride (Si_3N_4, UBE E10, UBE Industries, Yamaguchi, Japan), aluminum nitride (AlN, Grade

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C, H. C. Starck, Goslar, Germany), alumina (Al_2O_3, Aluminalux 49SG, Alcoa, Pittsburgh, PA), and yttria (Y_2O_3, fine-grade, H. C. Starck) in an appropriate weight ratio were attrition milled with Si_3N_4 balls in an acetone medium. The obtained powder was dried, sieved and pressed under an isostatic pressure of 250 MPa into green shapes. The green billets were fired at 1800 °C under 0.2 MPa nitrogen gas pressure in an electrical resistance furnace. The phases were analyzed by XRD using CuK_α radiation. The samples were finished up to 1 μm by diamond grinding and polishing. Surface of the polished samples were leached by HCl solution by keeping the samples immersed in the acid solution for 90 days. These samples were used after ultrasonically cleaning in pure water followed by drying. The fracture toughness (K_{IC}) was measured on these samples by the well known indentation fracture method at a load of ~98.1 N using a Vicker's diamond indenter and by using the following equation of Anstis et al. [9]:

$$K_{IC} = 0.016\,(P/D^{1.5})(E/H)^{0.5} \quad (1)$$

where, P is the applied indentation load, D is the characteristic crack parameter, E is Young's modulus and H is the measured hardness of the sample at the load P. The characteristic crack parameter D is calculated as the average crack length plus half of the average indentation diagonal where the crack length is measured from the indentation corner.

3. Results and discussions

The sintered material was characterized with the help of XRD which showed Y-α-sialon as the single crystalline phase (Fig. 1a). SEM of sintered Y-α-sialon is presented in Fig. 1b. The BSE image shows the presence of the amorphous phase as a bright white phase distributed in the matrix which is enriched in element of higher atomic weight, in this case, yttrium. The crystalline Y-α-sialon grains in different geometries contain lesser yttrium and hence are showing a lesser contrast. The FESEM image of the same material (Fig. 1c and d) after acid treatment shows the removal of the amorphous phase as is marked by the void pockets created in the microstructure, typically at the triple grain junctions. Enlarged version of such acid leached images has been used in image analysis. It is evident that majority of Y-α-sialon grains had grown into elongated shapes with an average length of around 4 μm, extending up to ~7 μm maximum. The average

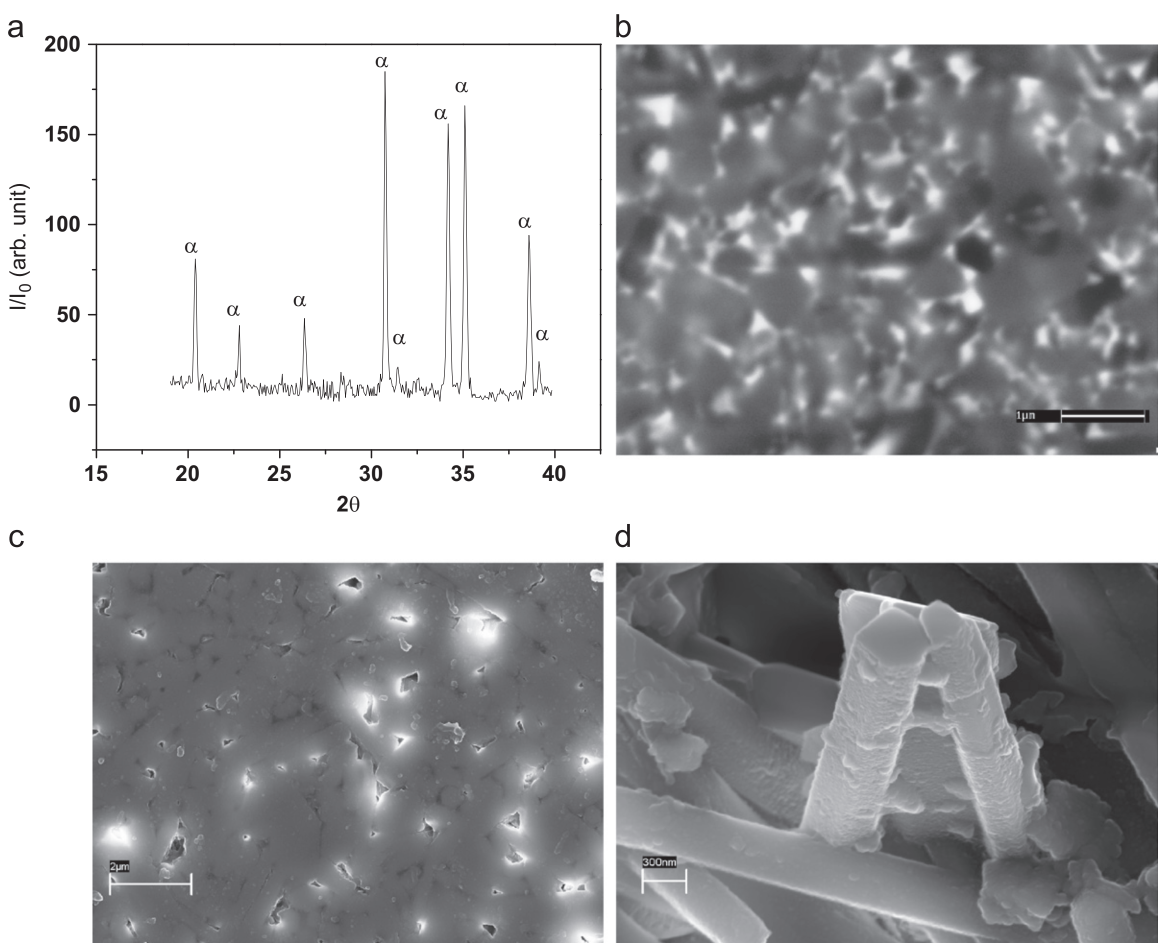

Fig. 1. (a) XRD pattern of the starting sintered (1800 °C, 2 h, 0.2 MPaN_2 overpressure) material; (b) BSE–SEM image of the polished surface of α-sialon; (c) FESEM image of acid leached surface and (d) the elongated grains of α-sialon as observed under high magnification FESEM after removal of amorphous phase.

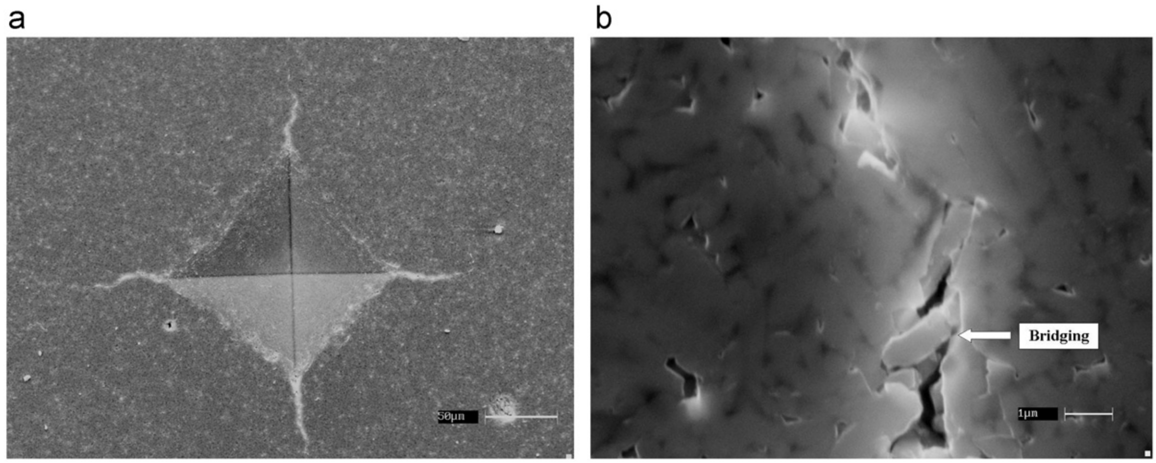

Fig. 2. (a) Indentation fracture as produced in acid leached surface and (b) typical crack propagation as obstructed by the bridging grains in α-sialon.

volume fraction of elongated grains as obtained from image analysis of at least five representative images was ~0.23 ± 0.03 while that of aspect ratio was measured as ~4.1 ± 0.57. The grains with an aspect ratio of larger than three were considered to avoid the alignment effect on a 2-D image. These values were utilized in estimating toughness values in two separate cases: (a) when crack bridging is the dominant mechanism, and (b) when aspect ratio and volume fraction of elongated grain is the dominant mechanism. An illustrative picture of indentation fracture is shown in Fig. 2a while an enlarged view of a typical indentation crack is shown in Fig. 2b. The average fracture toughness was measured as 6.95 ± 0.39 MPa $m^{0.5}$.

The toughening due to the crack bridging mechanism is given by [7]

$$K_{IC} = K_m(1-V) + \{4E\tau Vu(1-v^2)^{-1}K_m^{-1}\}ld^{-1} \quad (2)$$

In Eq. (2), K_m is the fracture toughness of the equiaxed sialon matrix grains, V the volume fraction of the elongated grains, u the grain pull-out length, τ the sliding friction stress, E the Young's modulus, v the Poisson's ratio and l/d is the grains aspect ratio. The Young's modulus of fully dense α-sialon has been measured in our case to be ranging from 298 to 336 GPa. Since leaching out of the grain boundary phase might create porosity in the open surface (the observed depth of corrosion layer in this case is ranging between 10 and 15 µm against the depth of penetration of the diamond indenter to be around 30 µm) and since elastic modulus varies with porosity [10,11], the average value of E has been considered from the lower side of the measured range to be 300 GPa. Now assuming the following other available values of α-sialon [7,12], $v = 0.22$, $\tau = 50$ MPa, $K_m = 2.6$ MPa $m^{0.5}$, and $V = 0.23$ as measured from microstructure and $u = 0.2$ µm as measured from conventional fractography of three-point bend test samples, the fracture toughness is predicted to be 6.59 MPa $m^{0.5}$. The predicted fracture toughness value corroborated closely to the average experimental data.

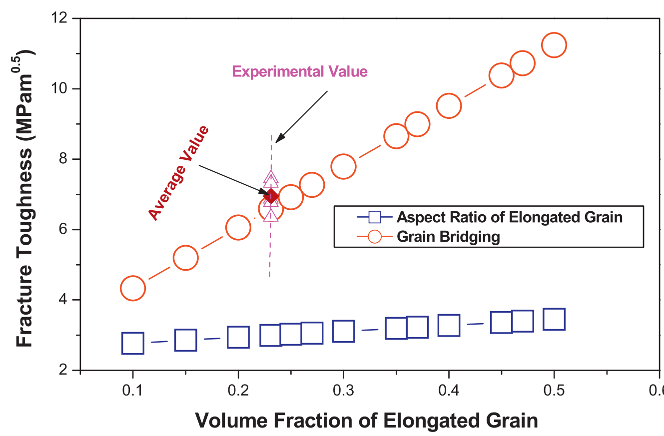

Fig.3. Predicted fracture toughness as a function of elongated grains.

On the other hand, the toughening due to the aspect ratio and volume fraction of elongated grains is given by [7,13]

$$K_{IC} = \{1 + V(0.6 + 0.014ld^{-1} - 0.0004l^2d^{-2})\}K_m \quad (3)$$

Applying the relevant values of V, (l/d) and K_m the calculated value of K_{IC} appears to be 2.99 MPa $m^{0.5}$. This is evidently much lower than the average experimental value of 6.95 MPa $m^{0.5}$ found in the present work.

The plot of fracture toughness as a function of volume fraction of elongated grains (Fig. 3) clearly demonstrate that the individual K_{IC} values as well as the average values fall close to the trend predicted according to Eq. (2) rather than that predicted according to Eq. (3). Therefore, we suggest that the fracture toughness improvement in the present dense Y-α-sialon ceramics after removal of amorphous phase was governed by grain bridging mechanisms operative across the high aspect ratio elongated grains which provide a suitable energy dissipation process. To the best of our knowledge this is the first experimental unequivocal observation that the fracture toughness of pure α-sialon grains devoid of amorphous phase is controlled by grain bridging mechanisms.

4. Conclusions

The present results unequivocally established that in dense Y-α-sialon ceramics with deliberately eliminated amorphous phase typically at the triple point junctions by acid treatment, the dominant toughening mechanism was grain bridging.

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