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FLAW TOLERANT Al_2O_3-Al_2TiO_5 COMPOSITES

Nitin P. Padture,[1] Stephen J. Bennison,[2] Julie L. Runyan,[2] Jürgen Rödel,[2] Helen M. Chan,[1] Brian R. Lawn[2]
[1] Department of Materials Science & Engineering, Lehigh University Bethlehem, PA 18015
[2] Ceramics Division, National Institute of Standards & Technology Gaithersburg, MD 20899

ABSTRACT

Fabrication of particulate-reinforced Al_2O_3-Al_2TiO_5 composites, with controlled homogeneous and heterogeneous microstructures, is described. The degree of flaw-tolerance in these composites, as determined by indentation-strength testing, is enhanced by the strong thermal expansion anisotropy stresses associated with the Al_2TiO_5 particles and by the scale of the microstructure. In situ scanning electron microscope observations of bridging grains at propagating cracks in the microstructure are analyzed. The role of key microstructural elements in the underlying toughness-curve behavior responsible for the flaw-tolerance is discussed.

INTRODUCTION

Researchers in recent years have demonstrated R-curves, i.e. an increasing crack resistance with crack size, in Al_2O_3-based ceramics [1-3]. A most important manifestation of this property is "flaw tolerance", i.e. near-invariance in strength over a broad range of flaw sizes. The major toughening mechanism responsible for R-curve behavior in Al_2O_3 ceramics has been identified as grain-localized bridging of the crack-wake interface [2,4]. The bridging grains are "clamped" in the matrix by internal compressive stresses from thermal expansion anisotropy (TEA), which augment frictional closure tractions from pullout during crack propagation [5]. Microstructural scaling parameters appear as additional important variables in the ensuing toughness equation [5].

The present paper reports on processing strategies to optimize the effect of TEA stresses and microstructural scaling on

the R-curve. TEA in monophase Al_2O_3 is relatively small, but can be enhanced by the incorporation of a suitable second phase. Al_2TiO_5 was chosen in this study because it has large TEA; it also has low solubility in Al_2O_3, and does not form intermediate phases [6-8]. Estimates of the internal stress in Al_2O_3-Al_2TiO_5 yield ≈ 3 GPa [8], greater than in Al_2O_3 by a factor ≈ 10.

The simplest way to scale up ceramic microstructures is by heat treatments [8,9,11]. However, coarsening of ceramics containing Al_2TiO_5 is severely restricted by an enhanced tendency to spontaneous microcracking [8]. To circumvent this problem scaling was achieved through controlled additions of large Al_2O_3 grains within an otherwise fine-grained Al_2O_3-Al_2TiO_5 matrix.

It will be seen in the subsequent sections that the tailored Al_2O_3-Al_2TiO_5 composites show profoundly enhanced flaw tolerance, hence stronger R-curves.

EXPERIMENTAL

Conventional ceramic processing routes were used to fabricate 80/20 vol% Al_2O_3/Al_2TiO_5 composites. Powders [1] of high purity α-Al_2O_3 (0.5 μm) and β-Al_2TiO_5 (crystallites 1-5 μm, agglomerates 10-20 μm) were mixed in a class A-100 clean-room, dispersed in methanol and stir-dried. Three batches were prepared: **A**, **homogeneous**, with a pre-drying ball-milling stage (Y-TZP balls, 24 hr in methanol) to remove the agglomerates; **B**, **heterogeneous**, with spray-dried Al_2O_3 agglomerates (25 μm) added to the ball-milled mix before drying; **C**, **heterogeneous**, similar to **A** but without ball milling, to retain Al_2TiO_5 agglomerates (10-20 μm).

Disks 4 mm thick were uniaxially pressed at 50-60 MPa followed by wet-bag isostatic pressing at 350 MPa. These disks were calcined at 1050°C for 12 hr, then sintered at 1550-1600°C for 1-17 hr. Microstructures were examined in the SEM and characterized using routine ceramographic procedures.

Prospective tensile sides of 20 mm diam. disks were polished to 1 μm, followed by indentation at the face center with a Vickers diamond at loads from 3 to 300 N. The indented specimens were then broken in biaxial flexure and the strengths determined. Details of this method are described elsewhere [3].

Compact-tension geometry fracture specimens were machined from 50 mm diameter disks for crack-interface examinations. These examinations were made **in situ** using a mechanical loading fixture housed in an SEM [10].

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[1] α-Al_2O_3, Sumitomo AKP-HP; β-Al_2TiO_5, Trans-Tech.

RESULTS

Figure 1 shows the microstructures of materials **A**, **B** and **C**. The base size of the matrix Al_2O_3 grains is consistently $\simeq 6\ \mu m$. In material **A**, fig. 1a, the Al_2TiO_5 grains are homogeneously distributed, but are about half the size of the Al_2O_3 grains. In material **B** and **C**, figs. 1b and 1c, distributions of relatively large heterogeneities are evident: Al_2O_3 agglomerates in **B**; Al_2O_3 **and** Al_2TiO_5 agglomerates in **C**. Densities in all cases exceed 98% of the theoretical limit.

Figure 2 shows data for strength against indentation load for the three composites, together with a comparative curve computed for 6 μm pure Al_2O_3 from an earlier study [11]. The composites show **significantly** lower strength sensitivity to starter flaw size, i.e. greater flaw tolerance, than the base Al_2O_3. Further, the sensitivity decreases in the composite sequence **A→B→C**, i.e. with more pronounced heterogeneity.

Figure 3 shows SEM micrographs of a bridging site taken at two stages of monotonic loading, in material **B**. Grain-facet frictional contacts at points **P** and **Q** act on the large (alumina) grain. Highly active bridge sites of this kind were significantly more prevalent in the composites than in the base Al_2O_3.

DISCUSSION AND CONCLUSION

We conclude from the above results that Al_2O_3-based composites can show considerably enhanced flaw tolerance relative to base Al_2O_3. This enhanced tolerance is a manifestation of a more pronounced R-curve [4], resulting in turn from stronger grain-interlock bridging.

Specifically, it is evident that high residual TEA stresses and heterogeneity are key factors in the increased flaw tolerance. Simplistically, internal stresses contribute by augmenting the frictional tractions at sliding grain facets during pullout [5]. Heterogeneity increases the pullout distance at the larger individual grains. Predictive modelling of the resultant enhanced bridging in two-phase ceramics should provide useful guidelines for improved microstructural design of structural ceramics [5,12].

We note finally that the approach advocated here of including controlled heterogeneous stress centers to augment bridging is in strong violation of conventional ceramics processing wisdom, where one seeks to produce ultra-fine, defect-free microstructures. Ours is a philosophy of **containing** cracks, not of eliminating them. In the same context, it may be pointed out that those very same internal stress sources that enhance long-crack toughness also may lead to deleterious short-crack properties like wear, spontaneous microcracking and fatigue [12].

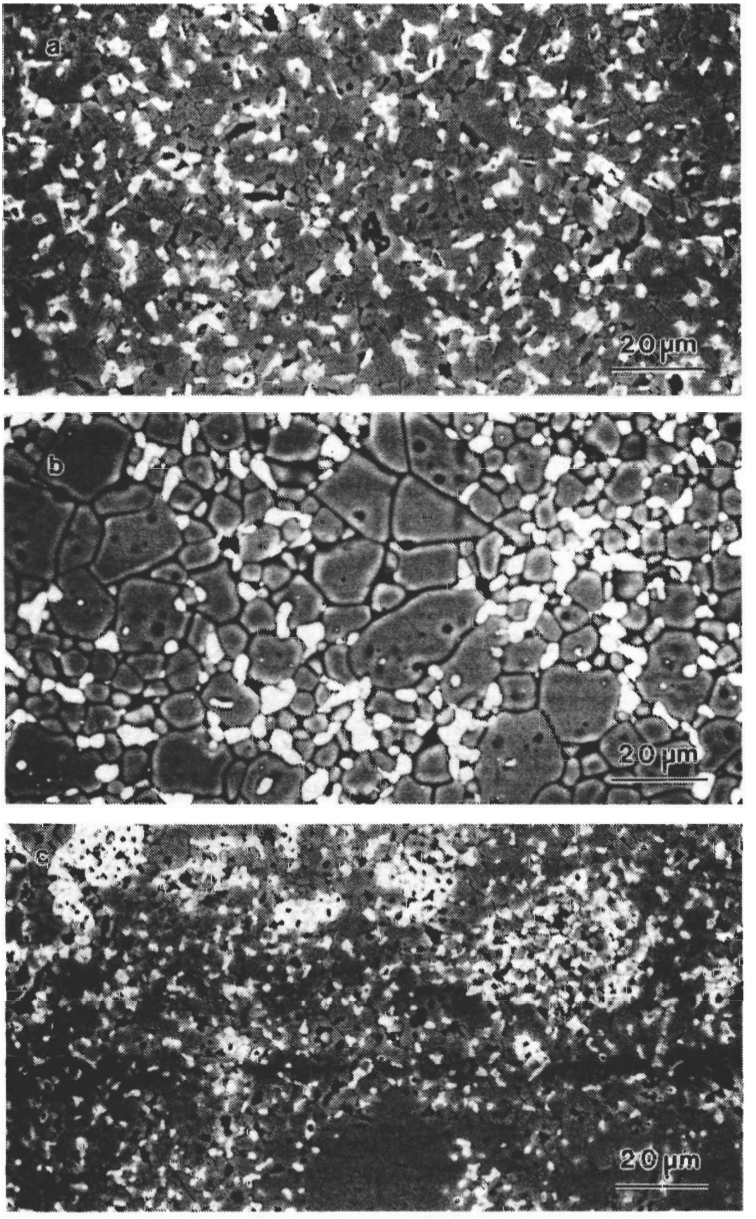

Figure 1. SEM microstructures of composites of 80% Al_2O_3 (dark grains) and 20% Al_2TiO_5 (light grains): (a) homogeneous material **A**, (b) heterogeneous material **B**; (c) heterogeneous material **C**. Samples polished to 1 μm and thermally etched.

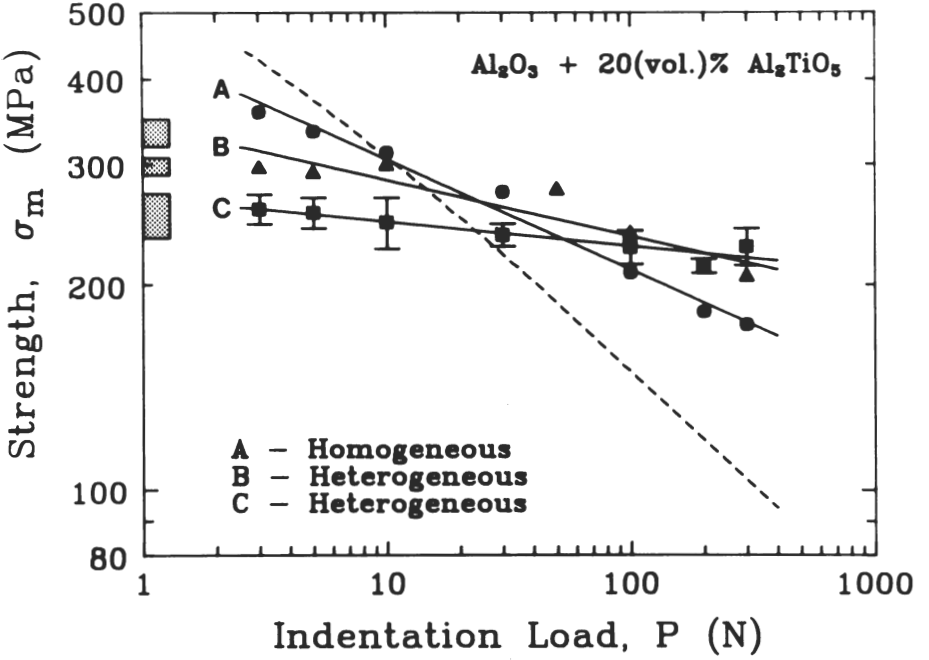

Figure 2. Indentation load versus strength plots. Data with solid curves for composite materials **A**, **B** and **C**. Error bars omitted from **A** and **B** data for clarity. Shaded area to left represents failures from natural flaws. Comparative dashed curve for Al_2O_3 of same grain size.

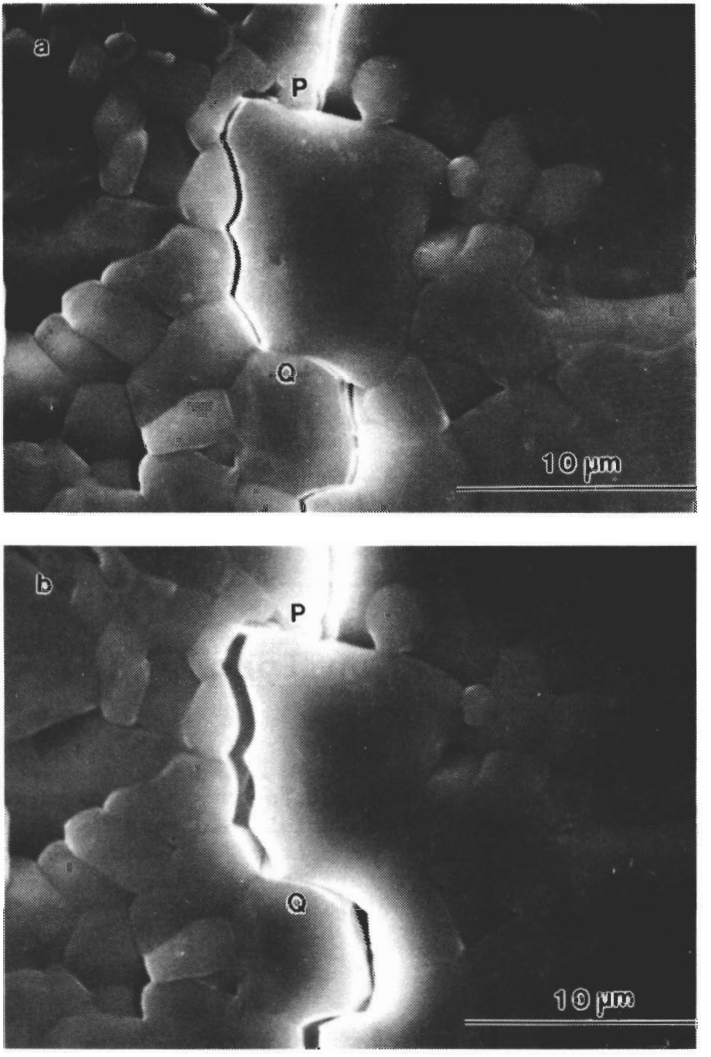

Figure 3. Bridging site at large alumina grain in composite **B**, showing frictional contact points at **P** and **Q**. Micrographs (a) and (b) taken **in situ** at successive increasing loads in SEM.

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