

Toughening in zirconia-toughened alumina composites with non-transforming zirconia

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Recently, microcrack toughening has been observed in some two-component ceramic systems having substantial thermal expansion mismatch (and consequently residual thermal stresses), in which neither component undergoes a phase transformation. In such systems toughening would arise from stress-induced microcracking in the vicinity of a propagating crack. Zdaniewski [1, 2] described the residual stress-induced microcracking as a principal toughening mechanism in TiB_2 -AlN composites, where the difference in the coefficients of thermal expansion (CTEs) of the components varied from $(1.5 \text{ to } 5.0) \times 10^{-6} \text{ }^\circ\text{C}^{-1}$. Faber *et al.* [3] observed a four-fold toughness increase in glass-ceramic systems, following the increase of volume fraction of the dispersed Al_2O_3 particles from 0 to 30%. Cai *et al.* [4] examined the SiC/TiB_2 (15 vol % TiB_2) systems in which toughening exceeded about 100% over the monolithic SiC matrix material. Magley *et al.* [5] determined experimentally the residual stresses within grain boundaries between phases in the SiC/TiB_2 composite as being of the order of up to 400 MPa with the difference of CTE of $2.3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$. Cai *et al.* [4] were the first to provide direct microscopic evidence (by transmission electron microscopy) of the presence of very fine microcracks at the SiC/TiB_2 grain boundaries.

In the present work an increase in fracture toughness of zirconia-toughened aluminium (ZTA) composites containing non-transforming zirconia was observed. It is well known that in such a system the tetragonal-to-monoclinic phase transformation in ZrO_2 can be suppressed by the appropriate choice of grain size and/or amount of stabilizer added to the ZrO_2 dispersed phase [6, 7]. Therefore, an increase in fracture toughness of ZTA materials with non-transforming ZrO_2 cannot be attributed to stress-induced phase transformation, or to the presence of microcracks produced by spontaneous transformation occurring upon cooling from sintering temperatures [8]. Stress-induced microcracking is then probably the dominant toughening mechanism.

ZTA compositions, with ZrO_2 content varying from 0 to 30 vol %, were wet-processed from dense aqueous suspensions using commercial powders. Alumina (Reynolds, RCHP-DBM) had a reported average particle size of $0.48 \mu\text{m}$. ZrO_2 powder (3Y-TZ from Tosoh, formerly Toyo Soda), containing 3 mol % Y_2O_3 as a stabilizer, was attrition milled

(Union Processes, model 1-S) for 4 h and centrifuged (Cont. Flaw Centrifuge IEC model CU 5000, from Damon) to all-submicrometre size ($d_{50} = 0.19 \mu\text{m}$). Individual aqueous suspensions (77 wt % solids) of the components were electrostatically stabilized using HNO_3 . These slurries were then mixed in appropriate proportions, homogenized in a planetary mixer (Turbula model 2TC) for 12 h with pH adjustments where necessary, and ultrasonically agitated (Heat Systems model W-385) for 10 min at 90% output power before casting into large plate forms ($10 \text{ cm} \times 8 \text{ cm} \times 0.5 \text{ cm}$). After drying, the samples were sintered in air at $1550 \text{ }^\circ\text{C}$ for 3 h to densities of the order of 99% theoretical.

The three-point bending strength was measured on $1.5 \text{ mm} \times 2 \text{ mm} \times 20 \text{ mm}$ samples cut from sintered plates and prepared according to US Military Standard 1942. The measurements were performed in air at room temperature (Instron model 1125) at a crosshead speed of 0.2 mm min^{-1} . A minimum of ten samples were tested for each ZTA composition. The fracture toughness was measured by the indentation technique (Leco model V-100-A2) on small samples from each composition, which were diamond polished to $1 \mu\text{m}$ finish and thermally etched at $1400 \text{ }^\circ\text{C}$ for 5 min. A load of 196 N was used in all cases to produce a suitable pattern. The K_{IC} -values were calculated according to Marshall and Evans [9].

After the indentation tests, selected samples were sputtered with Au-Pd and observed under the scanning electron microscope (SEM) in a secondary-electron mode (Jeol model 6000). The X-ray diffraction analysis (Phillips model PW 1840) were performed at room temperature on as-machined surfaces, coarsely abraded surfaces (60-grit) and fracture surfaces, after fracture stress measurements. All of these diffraction patterns revealed that there was essentially no monoclinic ZrO_2 phase present within the range of compositions examined (Fig. 1).

Fracture stress and fracture toughness values are shown in Fig. 2 as a function of the zirconia content. The strengthening occurs for compositions with up to 10 vol % ZrO_2 , corresponding to microstructures in which mostly singular grains of ZrO_2 can be found at the interstices of larger Al_2O_3 grains. The distribution of zirconia grains depends on the conditions of codispersion of component powder mixtures in aqueous suspension during wet processing, where the best dispersions usually occur at small ZrO_2

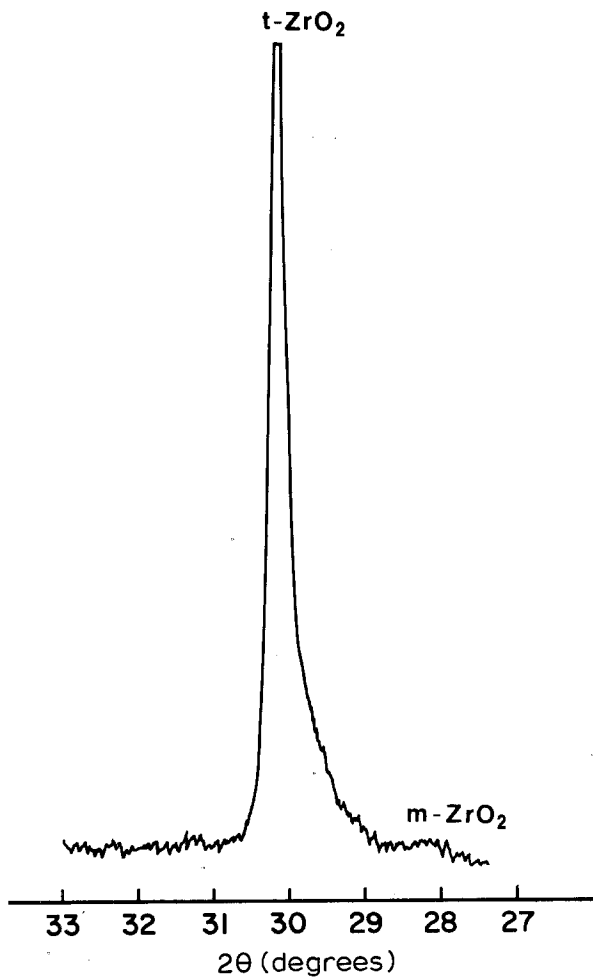


Figure 1 Typical X-ray diffraction pattern obtained from both fracture surfaces and lateral surfaces of modulus-of-rupture bars made of ZTA composites with various ZrO₂ contents.

content [10–12]. The increase in ZrO₂ content leads to an increasing degree of flocculation of composite suspensions and formation of ZrO₂ grain clusters within the final composite microstructures [10]. These clusters are also effective in hindering the Al₂O₃ grain growth upon sintering, therefore the strength of the compositions containing 20 and 30 vol % ZrO₂ remains practically the same as with 10 vol % ZrO₂.

The fracture toughness increases in the range of ZTA compositions with ZrO₂ content varying from 0 to 30 vol %, as seen in Fig. 2. With the increasing zirconia content, both the number and size of ZrO₂ clusters also increase, with the consequent increase in the number of potential sites of microcrack formation [13, 14]. These microcracks form under load when the tensile stress field at the tip of a propagating crack adds to the residual tensile stress generated by the thermal expansion mismatch between the phases (Fig. 3). That this toughening mechanism is dominant is further supported by the fact that, in addition to the absence of phase transformation of zirconia under load and spontaneous microcracking upon cooling, systematic observations of indentation crack paths in various ZTA compositions did not reveal a change in tortuosity, discarding crack deflection as a significant contributor to the increase in fracture toughness. This is also valid for crack bridging [15].

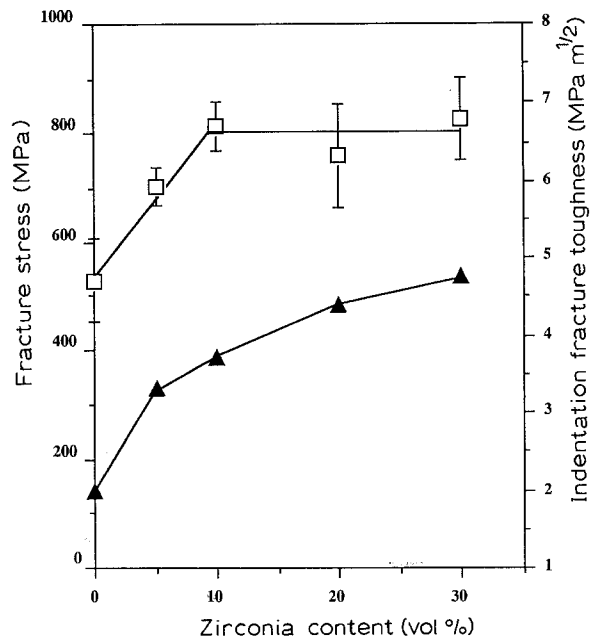


Figure 2 The values of (□) three-point bending strength and (▲) indentation fracture toughness versus ZrO₂ content in the ZTA composites examined. Error bars for toughness (equal to 1 s.d.) do not exceed the size of the symbol.

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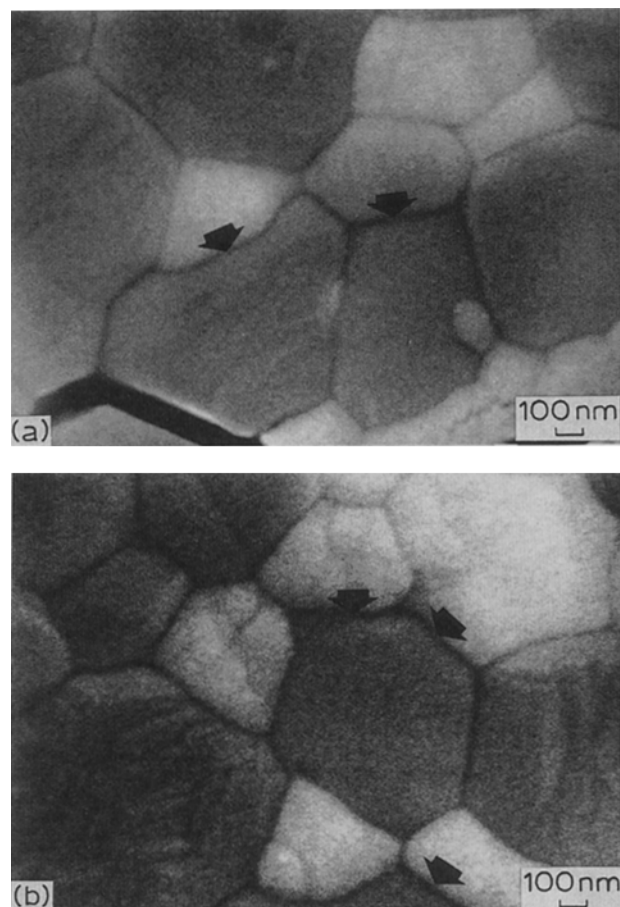


Figure 3 Microcracks between ZrO₂ and Al₂O₃ grains seen under the high-resolution SEM: (a) close to the main crack and (b) at a distance of a few μm from the main crack.

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