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Bifurcation in alumina plates produced by a phase transformation in central, alumina/zirconia thin layers

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Abstract

Alumina plates with a center, thin layer composed of Al_2O_3 and ZrO_2 (equal volume fractions) have been produced by sequential slip casting. Central layers as thin as 4 μm were fabricated using a low solid content slurry, but were observed to segregate the two oxides due to segregation of the consolidated layers during slip casting. Thicker layers were homogenous. The ZrO_2 tetragonal to monoclinic transformation generated residual, compressive stresses in the thin, center layer. Edge cracking and crack bifurcation was observed when the compressive layer thickness was $\geqslant$ 25 μm.

Keywords: Al_2O_3; Composites; Laminates; Mechanical properties; Slip casting; ZrO_2

1. Introduction

Bifurcation is a new phenomenon where a crack changes its direction after it enters a thin, compressive layer, provided that magnitude of the compressive stress times the layer thickness is greater than a critical value.[1] When placed in bending, the crack propagates through the stress-free layer and enters the compressive layer to extend along its center line. A new crack must reinitiate at a higher load to propagate through the next stress-free layer. In a multi-layered body, crack bifurcation occurs at each thin, compressive layer. Crack bifurcation along each compressive layer gives rise to a series of step-like drops in the stress-strain response instead of a single, catastrophic drop characteristic of brittle materials.[2] When the crack deflection phenomenon is present, the nominal failure strain is substantially higher than the failure strain measured under uniaxial tension. Residual compressions developed inside thin mullite–alumina layers in an alumina/mullite–alumina multilayer have been found to generate a threshold strength that ceramics materials with defects as big as 1 mm must overcome before catastrophic failure occurs.[3]

In the first bifurcation experiments, differential thermal contraction was used to generate compressive stresses of 2 GPa in thin Al_2O_3 layers sandwiched between thick, tetragonal $Zr(Y)O_2$ layers.[1] In these experiments, bifurcation only occurred when the compressive layers were 75μm thick. Because the thicker layers must be >20 times thicker than the thin compressive layers to avoid substantial tensile stresses in the thicker layers,[4] other routes to generate higher compressive stresses were sought. In the second set of experiments, the tetragonal to monoclinic ZrO_2 phase transformation was used to generate higher compressive stresses.[5] In these experiments monoclinic ZrO_2 powder, without Y_2O_3, was mixed with tetragonal $Zr(Y)O_2$ powder to produce thin, compressive layers, sandwiched between thick tetragonal $Zr(Y)O_2$ layers. At the processing temperature, the two ZrO_2 powders that formed the thin layer interdiffused to produce a transformation strain and a transformation temperature that was dependent on the volume fractions of the two powders. Although bifurcation was observed for layers as thin as 50 μm, the interdiffusion of Y_2O_3 from the thicker layers into the thin layer limited the possibility to determine the thickness necessary to produce bifurcation for a given composition.[5]

The aim of this work is to reduce the layer thickness required for bifurcation, using the tetragonal to monoclinic ZrO_2 phase transformation as a residual stresses

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developer but to prevent Y_2O_3 interdiffustion by mixing ZrO_2 powder with Al_2O_3 powder to form the thin layers and sandwiching these thin layers between thick Al_2O_3 layers.

2. Experimental procedure

The powders used in this study were Al_2O_3 (AKP-30, Sumitomo, Japan) and pure ZrO_2 (TZ-0, Tosho, Japan). Plate specimens (75×75 mm) were prepared by sequential slip casting[5–8] to contain a single, central, thin layer of 50/50 vol% of monoclinic ZrO_2/Al_2O_3 (50MZ/50A) sandwiched between two thick layers (20 mm) of Al_2O_3 (A). Different specimens were prepared to vary the thickness of the thin layer between 5 and 100 µm thick. Aqueous slurries for the thin layers were prepared at pH 11 to contain 0.05, 0.10, 0.20 and 0.30 volume fraction of the mixed, 50MZ/50A powders.

The thick, Al_2O_3 layers were produced using 0.30 volume fraction slurry, and casting times calculated to fix the thickness at 20 mm. Two methods were used to fabricate specimens with different thicknesses of the central thin, 50MZ/50A layer. One method was to fix the solid content of the slurry at 0.30 volume fraction and use casting times of 30, 60 and 210 s. The second method was to maintain constant the casting time of 30 s, but to use slurries containing 0.05, 0.10 and 0.20 volume fraction of solids. The plates were densified at 1550°/2 h in air. The dense plates were diamond ground and cut into bar specimens (47×6×60 mm) for mechanical characterization. Four point bending tests were performed (Instron Universal Testing Machine, 8045) using inner and outer spans of 20 and 40 mm at a crosshead speed of 0.01 mm/min. Fractured specimens were observed by reflected light optical microscopy. Some specimens were diamond polished to observe edge cracks with the SEM.

3. Results and discussion

Table 1 reports the thickness of the central 50MZ/50A layers for all specimens fabricated. As shown in Fig. 1, the ZrO_2 and Al_2O_3 phases in the central layers were uniformly dispersed when they were fabricated with slurries containing 0.20 and 0.30 volume fractions of dispersed solids. The edge crack was clearly observed for layer thicknesses ⩾ 60 µm. Fig. 2 shows the sharp interface between the Al_2O_3 and 50MZ/50A layers; microcracks due to the transformation can be detected neither at the interface nor within the central, 50MZ/50A layer. These sharp interfaces were obtained for slurries with volume fractions of solid greater than 0.1. When slurries with 0.10 and 0.05 of solid volume fraction were used, thiner layers were obtained but the interfaces between the alumina consolidated layer and the 50MZ/50A layers were not as defined as in the more concentrated slurries. Fig. 3 illustrates the very thin central layer produced with the slurry containing 0.05 volume fraction of the mixed 50MZ/50A powders. It appears that the very dilute, dispersed slurry disturbed the adjacent Al_2O_3 consolidated layer increasing the Al_2O_3 ratio in the casting surface and generating a graded

Table 1
Thickness of the central, thin layer for laminates processed with different casting times and from slurries containing different solid contents

Solid content of the slurry (vol%)	30	30	30	20	10	5
Casting time (s)	210	60	30	30	30	30
Middle layer thickness (µm)	160	75	60	25	8	4–5

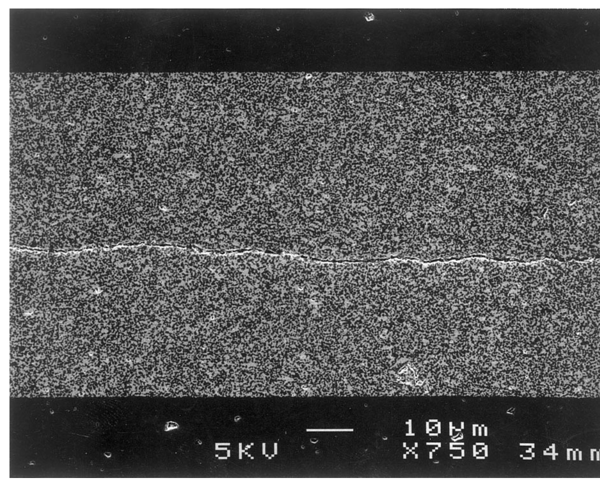

Fig. 1. SEM micrograph of the cross section of the laminate cast using a 30 vol% slurry of 50MZ/50A for 60 s. The final thickness of the layer is 75 µm. It is clearly observed the edge crack developed at the middle of the layer.

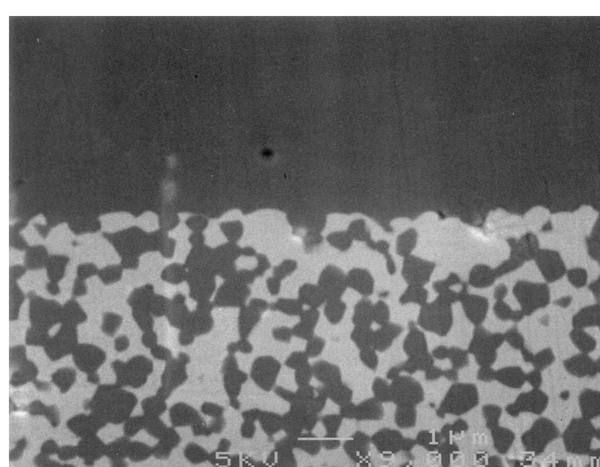

Fig. 2. Close up to the (alumina)–(50MZ/50A) interface. No microcracking was observed at any sample in both the inner thin and the outers alumina thick layers.

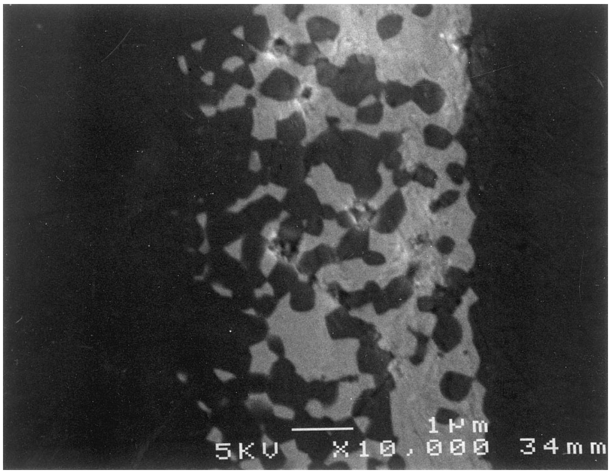

Fig. 3. View of the middle layer in a laminate cast using a 5 vol% slurry of 50MZ/50A for 30 seconds. The final thickness of the layer is 4 µm.

composition at the interface. When the concentrated alumina slurry is used to create the other, sandwiching thick layer no dilution is observed and the interface is sharp again.

For residual stresses calculations, the required data like the elastic modulus or shrinkage of the 50MZ/50A composite are not available, because the expansion of the zirconia degrades the properties of the monolithic compacts. As demostrated elsewhere,[9] this expansion does not degrade the properties in the layered structure, but develops high residual stresses. Using the elastic properties of the two materials,[10] and assuming that: (i) the ZrO_2 completely transformed during cooling with a linear transformation strain of 0.003 developing a diferential strain between the layers of 0.0015, and (ii) no microcracking took place to decrease the elastic modulus; the compressive stress within the 50MZ/50A layers was estimated[4] to be 650–620 MPa, whereas the tensile stresses within the thicker, sandwiching Al_2O_3 layers was estimated to range from 20 to 40 MPa. Table 2 reports the results of mechanical testing (flexural strength: average of three specimens for each condition) and shows that bifurcation was observed for all specimens fabricated with a central 50MZ/50A layer $\geqslant$25 µm. Since the grain size of the alumina outer layers was not controlled, the specimens failed at relatively low values of stress.

Fig. 4 shows the optical micrographs corresponding to the cross section of tested beams with layer thickness of 80 (4.a) and 9 (4.b) µm. As can be appreciated the sample with central layer thickness of 75 microns clearly bifurcates. The extension of bifurcation (estimated as the length of the crack running through the central thin layers before continuing into the alumina thick layers) was observed to be smaller as the layer thickness decreases.

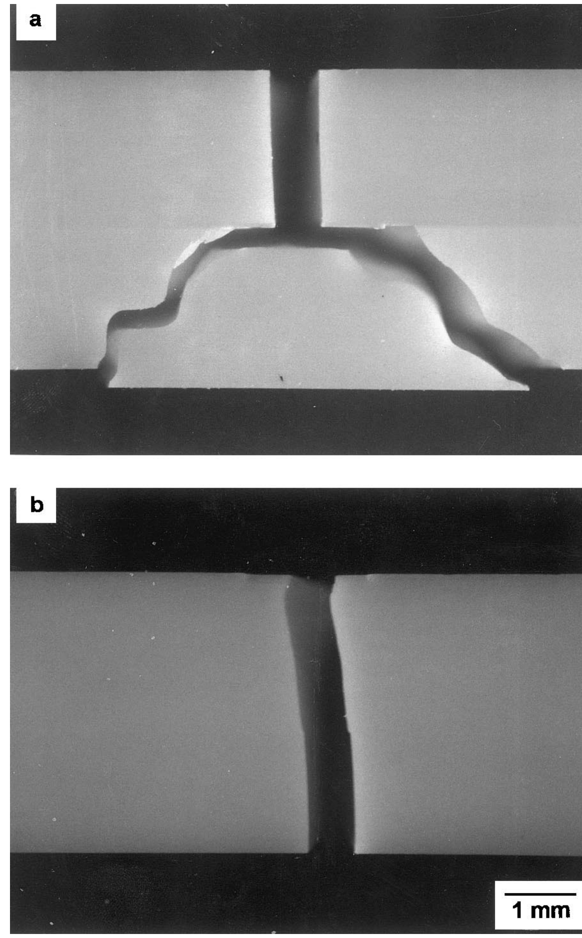

Fig. 4. Cross section of the tested samples with layer thickness of (a) 75 µm (bifurcation) and (b) 9 µm (no bifurcation).

The mechanical and microstructural observations show that very thin layers of a zirconia-containing material can be inserted into an alumina matrix by colloidal routes developing high compresives stresses due to the tetragonal to monoclinic transformation during cooling. Up to a certain thickness, this stresses can vary the fracture patern by a bifurcation mechanism depending on the thickness of the middle layer. In the studied case of 50MZ/50A layers inside a alumina matrix, for layers

Table 2
Flexure strength for specimens containing 50MZ/50A thin layers

Layer thickness (µm)	4–5	9	25	60	80	160
Fexural strength (MPa)	320 ± 20	250 ± 25	260 ± 15[a]	260 ± 15[a]	270 ± 10[a]	260 ± 20[a]

[a] Bifurcated samples.

thicker that 25 μm the bifurcation is observed. It is predictible that higher zirconia content layers can generate bifurcation at lower thickness.

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