

# Residual Stresses in Layered Ceramic Composites

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## Abstract

*Laminar composites containing layers of Y-TZP or Ce-TZP and either Al<sub>2</sub>O<sub>3</sub> or a mixture of alumina and zirconia have been fabricated using a sequential centrifuging technique of water solutions containing suspended particles. Controlled crack growth experiments with notched beams of composites showed the significant effect of barrier layer thickness and composition on crack propagation path during fracture. Distinct crack deflection was observed in alumina layers. In the case of layers made of an oxide mixture, crack deflection was not found independently on layer thickness. The observed changes have been correlated with distribution of residual stresses in barrier layers created during cooling of sintered composites from fabrication temperature. © 1999 Elsevier Science Limited. All rights reserved*

**Keywords:** crack deflection, thermal expansion, ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>.

## 1 Introduction

Ceramic materials are naturally brittle, so much effort has been directed to improve their toughness by various methods. Since the discovery of transformation toughening in ZrO<sub>2</sub> in 1975<sup>1</sup> a variety of toughened ZrO<sub>2</sub>-based materials have been developed. A new way of optimization in ceramic systems strengthened by tetragonal ZrO<sub>2</sub> was found by Marshall<sup>2,3</sup> in multilayered ceramic composites. A significant toughness increase of Ce-TZP matrix with Al<sub>2</sub>O<sub>3</sub> or Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> barrier layers was attributed to the spreading of the transformation zone along the regions adjacent to the layers. On the contrary to Marshall results, reported work

showed that the main mechanism responsible for toughness increase in laminated composites is deflection of crack caused by presence of residual stresses. Correlation between crack deflection and distribution of residual stresses found in barrier layers of composites was the aim of this work.

## 2 Experimental Procedure

Composites of Y-TZP or Ce-TZP with alumina layers with thicknesses of 10 to 70 μm were fabricated by sequential centrifuging (Model Z382, Hermle) of powder suspensions (0.6 μm ZrO<sub>2</sub> + 3.4 mol% Y<sub>2</sub>O<sub>3</sub> from Unitec Ceramics, 0.3 μm Ce-ZrO<sub>2</sub> from Tosoh, 0.29 μm Al<sub>2</sub>O<sub>3</sub> from Sumitomo). Aqueous slurries containing 5 to 10 wt% of subsequent powder were prepared by ultrasonication the powders in deionized water at pH 4. Cast samples were dried, additionally isostatically pressed at 120 MPa and then sintered at 1600°C. The larger shrinkage of Y-ZrO<sub>2</sub> and Ce-ZrO<sub>2</sub> than Al<sub>2</sub>O<sub>3</sub> during sintering caused that in some layered composites the mixed composition of 50 vol% alumina and zirconia was used instead of a pure Al<sub>2</sub>O<sub>3</sub> to minimize this mismatch. The samples after sintering were cut and ground and one surface perpendicular to the layers was polished. The sharp notch in the center of the beams was prepared.

The tests of controlled crack growth were performed using a universal testing machine (Model 1446, Zwick) in three-point bending with 1 μm min<sup>-1</sup> loading speed and 40 mm bearing distance. The crack was initiated and slowly grown up step by step in a controlled way by permanent loading and removing of the load. The path of the crack during fracture was registered by SEM using a microscope (Model Opton DSM 950).

The spatial distribution of residual stresses within the alumina and a mixture of alumina and zirconia layer of composites was measured using

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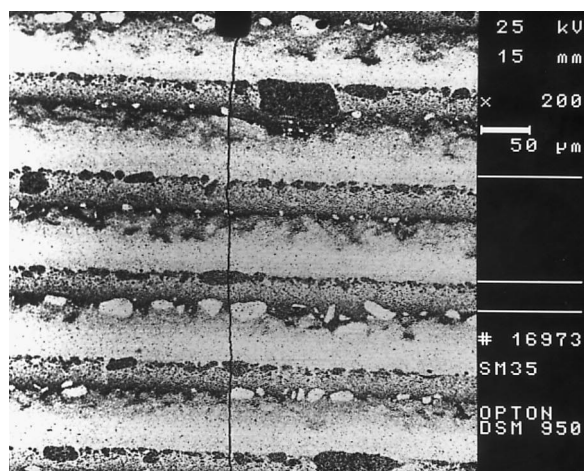
piezospectroscopic technique<sup>4</sup> (spectrometer Dilor X4800).

### 3 Results and Discussion

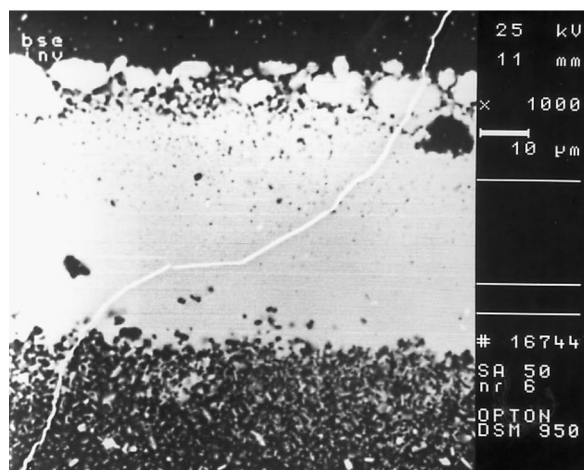
Controlled crack growth experiments indicated significant crack deflection in barrier layers of composites dependent on barrier composition but not on type of zirconia matrix. It occurred that crack deflects only in alumina layers (Figs 1 and 2). In zirconia layers crack deflects back to its original direction perpendicular to the layers. The degree of crack deflection depends on alumina layer thickness (Table 1). As it was shown at Figs 1 and 2 in barrier layers made of an oxide mixture crack propagates perpendicularly to the layers without deflection independently on layer thickness.

Generally crack deflection is related to residual thermal stresses and instantaneous elastic modulus mismatch effects. In the present case the residual biaxial compressive stress was expected in the layer with lower thermal expansion coefficient (alumina),

biaxial, tensile stresses were expected in the layer with the greater thermal expansion (zirconia). Piezospectroscopic measurements showed that the distribution of compressive stresses was different in both types of barrier layers. In the alumina layers compressive stress was not only a function of the layer thickness but also of the position across the layer (Figs 3 and 4). Maximum of compressive stress was observed at the interface (280 MPa) and minimum in the center of alumina layer. The minimum stress was dependent on alumina layer thickness (Table 1). On the contrary, in barrier

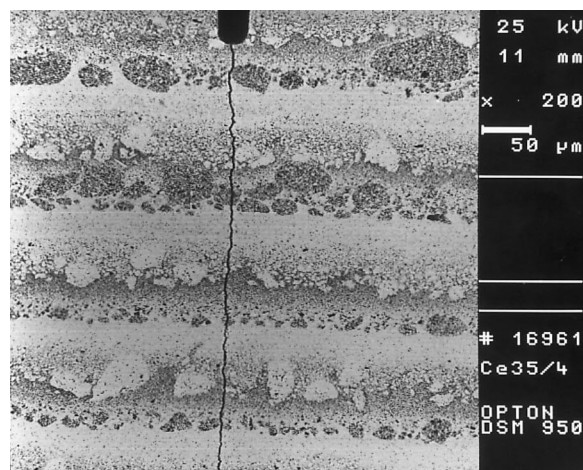


(a)

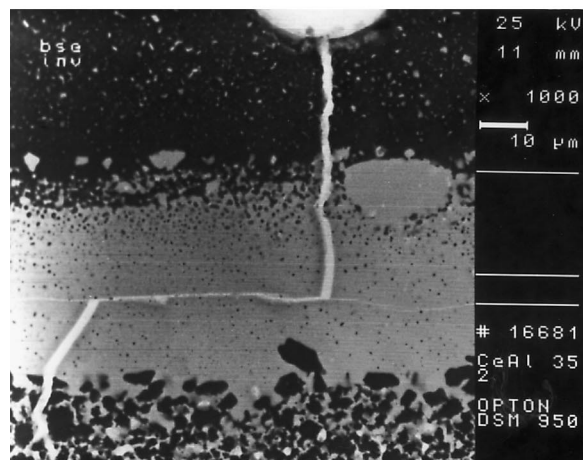


(b)

**Fig. 1.** Crack path in 60  $\mu\text{m}$  thick alumina layer (bottom, inverted image) and in 45  $\mu\text{m}$  thick barrier layers made of an oxide mixture (top, normal image) of Y-TZP/ $\text{Al}_2\text{O}_3$  composite.



(a)



(b)

**Fig. 2.** Crack path during fracture of Ce-TZ/ $\text{Al}_2\text{O}_3$  composite with 50  $\mu\text{m}$  thick barrier layers made of alumina (bottom, inverted image) and an oxide mixture (top, normal image).

**Table 1.** Crack deflection angle and compressive stress in minimum in alumina layers of Y-TZP/ $\text{Al}_2\text{O}_3$  composite as a function of layer thickness

Thickness of alumina layer ( $\mu\text{m}$ )	Crack deflection angle ( $^\circ$ )	Compressive stress in minimum (MPa)
10	0	266.8
25	$22 \pm 5$	229.2
40	$62 \pm 8$	121.9
60	90	91.6

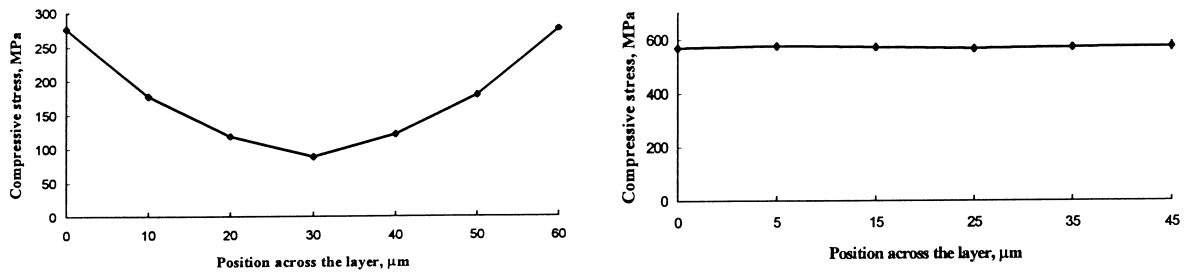


Fig. 3. Compressive stress distribution in barrier layers made of alumina (left) and a mixture of alumina and zirconia (right) of Y-TZP/ $\text{Al}_2\text{O}_3$  composites.

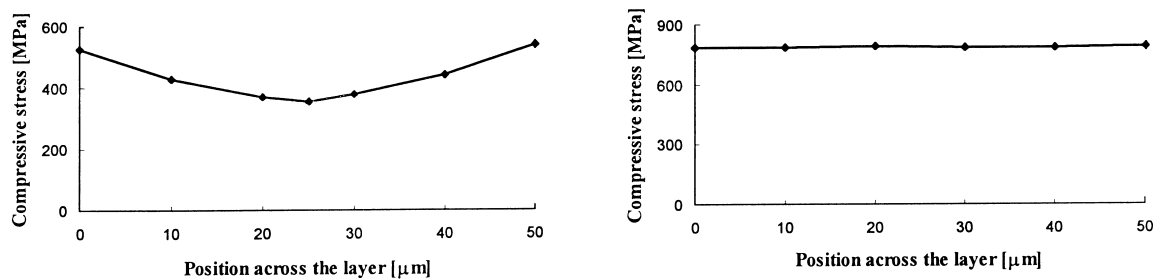


Fig. 4. Compressive stress distribution in barrier layers made of alumina (left) and a mixture of alumina and zirconia (right) of CE-TZP/ $\text{Al}_2\text{O}_3$  composites.

layers made of an oxide mixture the compressive stress was constant on position across the layer (Figs 3 and 4). The present results reveal that compressive stress distribution in barrier layers can be regarded as an important factor responsible for crack deflection in layered composites.

#### 4 Conclusions

The aim of this work was to determine the residual stress effects on the character of crack propagation in layered ceramic composites. During tests of controlled crack growth a distinct crack deflection in alumina layers was observed. The crack deflection angle was proportional to the layer thickness. In

barrier layers made of an oxide mixture crack deflection was not found. Observed changes were related to measurements of residual stress distribution in barrier layers.

#### References

1. Garvie, R. S., Hannink, R. H. J. and Pascoe, R. T., Ceramic steel? *Nature (London)*, 1975, **258**, 703–704.
2. Marshall, D. B., Ratto, J. J. and Lange, F. F., Enhanced fracture toughness in layered microcomposites of Ce-ZrO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. *J. Amer. Ceram. Soc.*, 1991, **74**, 2979–2987.
3. Marshall, D. B., Design of high-toughness laminar zirconia composites. *Ceram. Bull.*, 1992, **78**, 969–973.
4. He, J. and Clarke, D. R., Determination of the piezo-spectroscopic coefficients for chromium-doped sapphire. *J. Am. Ceram. Soc.*, 1995, **78**, 1347–1353.