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# Residual stresses, strength and toughness of laminates with different layer thickness ratios

R. Bermejo a, Y. Torres A.J. Sánchez-Herencia b, C. Baudín b, M. Anglada a, L. Llanes a,\*

 a Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica, ETSEIB, Universidad Politécnica de Cataluña, Avda. Diagonal 647, E-08028 Barcelona, Spain
b Instituto de Cerámica y Vidrio (CSIC), C/Kelsen 5, 28049 Madrid, Spain

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#### Abstract

The effect of residual stresses on the strength, toughness and work of fracture of  $Al_2O_3$ –5 wt.%  $tZrO_2/Al_2O_3$ –30 wt.%  $mZrO_2$  layered ceramics with different thickness ratios has been investigated. The laminates, as well as a monolithic  $Al_2O_3$ –5 wt.%  $tZrO_2$  used as reference material, were fabricated by sequential slip casting. Residual stresses were estimated experimentally using indentation techniques and analytically using a three-dimensional finite element model. Flexural strength was evaluated by means of four-point bending tests on specimens with natural and artificial (indentation) flaws. Experimental findings show the existence of a threshold strength in the laminates whose value depends on the layer thickness ratio. Crack growth resistance behaviour was studied by crack opening displacement-controlled tests and by recourse to a weight function analytical approach. The high compressive stresses in the internal layers yield a pronounced R-curve behaviour in the laminates. Regarding work of fracture, it is found to be enhanced to levels up to about six times that of the reference monolith. The results are discussed in terms of the optimum layered architectural design for structural applications on the basis of the compromise between threshold strength and energy absorption capability associated with crack bifurcation mechanisms occurring at fracture.

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#### 1. Introduction

Design against brittle-like fracture assumes that materials contain defects either within the bulk or at the surface, resulting from processing and/or machining procedures. This is specifically true for ceramic components where intrinsic or extrinsic flaws are the common source of failure due to the stress concentration associated with them. From this perspective, it is well established that the stress concentration at a crack tip depends on crack geometry; hence, the size and type of these defects will condition the mechanical strength of the material. As a result, structural ceramics exhibit a statistically variable brittle fracture which

limits their use for load-bearing applications. In the last three decades, remarkable advances have been achieved to overcome the lack of toughness of structural ceramics. Several processing routes have emerged which do not recall conventional "flaw elimination" approaches, but rather "flaw tolerant" ones based on the operability of energy release mechanisms aiming to improve strength reliability. Among those, doping, fibre and/or particle reinforcement, functional grading and layered architectural design may be highlighted. Particularly, alumina–zirconia [1,2] and mullite–alumina [3] ceramic composites with a layered structure, among others, have been reported to exhibit increased apparent fracture toughness and energy absorption, as well as non-catastrophic failure behaviour.

One of the most used multilayer designs that ensures higher apparent toughness is that which combines layers

<sup>\*</sup> Corresponding author. Tel.: +34 93 4011083; fax: +34 93 4016706. E-mail address: luis.miguel.llanes@upc.edu (L. Llanes).

with different volume changes during cooling from the sintering temperature. Under these conditions, an alternate tensile-compressive residual stress state develops with specific location of the compressive layers, either at the surface or internally, depending on the attempted design approach. based on either mechanical resistance or damage tolerance, respectively. In the former case, the effect of the compressive residual stresses on the nominally applied stress results in a higher, but single-value, apparent fracture toughness together with enhanced strength (the main goal) and some improved reliability [4,5]. On the other hand, in the latter case, the internal compressive layer is microstructurally designed to rather act as stopper of any potential processing flaw at surface layers, independent of original size and location, such that failure tends to take place under conditions of maximum crack growth resistance. As a consequence, strength becomes flawsize independent and reliability is significantly increased. Within this framework, an "extreme" case is the possibility of developing materials exhibiting a "threshold strength", i.e. a stress below which failure would not occur despite the presence of relatively large cracks, as reported for alumina-alumina mullite [6,7] or alumina–alumina zirconia multilayered systems [8]. From this viewpoint, much effort has been expended on the fabrication of laminates with a tailored residual stress profile arising from mismatch of thermal expansion coefficients between layers, selective phase transformation and/or chemical reactions [7,9,10]. In these investigations, zirconia-containing laminar ceramics have been employed to develop compressive stresses in the internal layers by means of the tetragonal to monoclinic phase transformation that takes place in the zirconia phase when cooling down during sintering. The corresponding volume increase associated with such transformation determines the residual stress field within the multilayer. Under certain conditions, these compressive stresses may act as a barrier to crack propagation. In other cases, crack deflection at the interface of dissimilar materials [11–13] and/or crack bifurcation due to the high compressive stresses in the internal layers of the composite [14,15] result in an increase of fracture toughness and energy absorption capability [16]. The search of laminar ceramic composites for structural applications must be focused on "flaw tolerance" materials, where reliability gets significantly enhanced, exhibiting a fairly high resistance to failure.

The purpose of the investigation reported here was to optimize the design of alumina–zirconia layered ceramics to obtain flaw-tolerant materials with pronounced crack growth resistance and work of fracture. In doing so, three multilayered architectures of the same composition but different layer thickness ratios fabricated by slip casting were studied. The residual stress profile was determined both experimental and analytically using the indentation technique and a three-dimensional finite element model, respectively. Four-point bending tests were performed on virgin and indented samples to account for the existence of a threshold strength in the laminates. Although no standard-

ized methods are available to evaluate the fracture toughness in layered composites, a fracture mechanics weight function analysis was effectively used to estimate the crack growth resistance behaviour (R-curve) as a function of position within the layered architectures investigated. Additionally, crack opening displacement (COD)-controlled tests were conducted to determine the work of fracture in monoliths and laminates. Finally, a discussion of the optimum multilayered architectural design is provided in terms of threshold strength, toughness and energy absorption capability associated with energy-dissipating mechanisms operative during initial crack extension up to final catastrophic failure.

#### 2. Experimental

#### 2.1. Materials

The following starting powders were used: (i)  $\alpha$ -alumina (Condea, HPA05, USA) with 0.29 µm average particle size and 8.5 m<sup>2</sup>/g specific surface area (N<sub>2</sub> adsorption; BET method), (ii) Y<sub>2</sub>O<sub>3</sub>-free and Y<sub>2</sub>O<sub>3</sub> (3 wt.%)-stabilized zirconia (TZ-0 and TZ-3YS, Tosoh, Japan) with 0.60 and 0.37 μm average particle size and 14.0 and 6.7 m<sup>2</sup>/g specific surface area, respectively. A slurry composed of Al<sub>2</sub>O<sub>3</sub>/5 vol.% Y<sub>2</sub>O<sub>3</sub>-stabilized ZrO<sub>2</sub> (t-ZrO<sub>2</sub>), referred to as ATZ, was used to form all the thicker layers. The t-ZrO<sub>2</sub> was utilized to control the grain size of the Al<sub>2</sub>O<sub>3</sub> during sintering. In order to form the thin layers a slurry containing Al<sub>2</sub>O<sub>3</sub>/30 vol.% Y<sub>2</sub>O<sub>3</sub>free ZrO<sub>2</sub> (m-ZrO<sub>2</sub>), named AMZ, was employed. Each suspension was re-used every time to form the successive layers of the corresponding composition. The content of non-stabilized zirconia in these layers was selected to promote high residual compressive stresses, as studied in previous works [15,17,18]. Preparation of the batches has already been detailed elsewhere [19]. Wall thickness vs. time curves were experimentally determined on monolithic samples for both slurries and then used to calculate the time for sequential slip casting of three different multilayered systems named A, B and C, with the same composition but different layer thickness ratios [20]. Laminates were composed in all cases of five thick ATZ layers alternated with four thin AMZ layers. Cast specimens were carefully removed from the moulds, dried at room temperature for 48 h, and finally fired at 1550 °C for 2 h using heating and cooling rates of 5 °C/min. Rectangular plates of approximately  $60 \text{ mm} \times 60 \text{ mm} \times 4 \text{ mm}$  were obtained for the three multilayered architectures. The thickness of the layers resulting for each case was measured by optical microscopy on polished samples, and the experimental values, including the corresponding layer thickness ratios, are listed in Table 1. The outer ATZ layers were cast thicker than the inner ones to allow grinding and polishing procedures. Density measurements were carried out for both ATZ monoliths and laminates, yielding values of 99.5% and 99.3%, respectively.

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