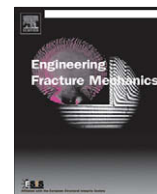




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High failure resistance layered ceramics using crack bifurcation and interface delamination as reinforcement mechanisms

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ABSTRACT

Layered ceramics designed with weak interfaces favour interface delamination, while laminates with strong interfaces show higher strength and enhanced mechanical reliability. In this paper, conditions are reviewed aiming to combine crack bifurcation and interface delamination mechanisms in a unique architecture to design layered ceramics with high failure resistance. Based on a bi-material theoretical approach supported by experiments it is found that interface delamination can be favoured if crack bifurcation occurs in the compressive layers with a low inclination angle. The thickness and stresses of the compressive layers are the key features to optimise the mechanical behaviour in layered ceramics.

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1. Introduction

The interest for the mechanical behaviour of ceramic materials has been always motivated by their possible application as structural components. However, most of the new engineering designs need to withstand tensile stresses which imply potential limitations due to the inherent brittleness of ceramic materials. In addition, it is known that the flaw distribution (size, location, etc.) and size effect in ceramic materials yield a statistical strength distribution (described by the Weibull theory [1]), which conditions the mechanical reliability of ceramic components [2–4].

Despite the outstanding features of colloidal processing in terms of flaw size reduction (*i.e.* increase of strength) [5], the presence of processing and/or machining defects in ceramic materials is in most cases unavoidable. In this regard, trends to design “*flaw tolerant*” materials rather than reducing the size of such defects have been the focus of many researchers in the last decades [6–14]. In particular, layered ceramics have been proposed as an alternative choice for the design of structural ceramics with improved fracture toughness, mechanical strength and reliability. As a result, the brittle fracture of monolithic ceramics has been overcome by introducing layered architectures of a different kind, *i.e.* geometry, composition of layers, residual stresses, interface toughness, etc. The main goal of such layered ceramic designs has been to enhance the fracture energy of the system on the one hand and to increase the strength reliability of the end component on the other hand.

Among the various laminate designs reported in literature, two main design approaches regarding the fracture energy of the layer interfaces must be highlighted. On the one hand, laminates designed with weak interfaces have been reported to yield a significant enhanced fracture energy (failure resistance) through interface delamination [15–20]; the fracture of the first layer would be followed by crack propagation along the interface, the so-called “*graceful failure*”, preventing the material from catastrophic failure. On the other hand, laminates designed with strong interfaces have shown crack growth resistance

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Nomenclature

a	length of a reference crack
a_d	length of a deflecting crack
a_p	length of a penetrating crack
c	dimensionless parameter
d	dimensionless complex function
e	dimensionless complex function
E	elastic modulus
E'	plain strain elastic modulus
G_i	fracture energy of the interface
G_{layer}	fracture energy of the layer
G_d	energy release rate of a deflecting crack
G_p	energy release rate of a penetrating crack
k_I	parameter proportional to the applied load
K_I	stress intensity factor in mode I
t	layer thickness
α	first Dundur's parameter
β	second Dundur's parameter
η_n	non-dimensional (normal residual stress) length parameter
η_t	non-dimensional (tangential residual stress) length parameter
φ	crack bifurcation angle
λ	stress singularity exponent
μ	shear modulus
ν	Poisson's ratio
σ_c	residual stress in the compressive layer
σ_n	normal residual stress
σ_t	tangential residual stress
σ_{xx}	traction ahead of the crack tip

(*R*-curve) behaviour through microstructural design (e.g. grain size, layer composition) [21–25] and/or due to the presence of compressive residual stresses, acting as a barrier to crack propagation [6,9,11,13,26–29]. The increase in fracture energy in these laminates is associated with energy dissipating mechanisms such as crack deflection/bifurcation phenomena. In particular, the utilization of tailored compressive residual stresses (generated during cooling down from sintering) to act as physical barriers to crack propagation has succeeded in many ceramic systems, yielding in some cases a so-called “*threshold strength*”, i.e. a minimum stress level below which the material does not fail [6,7,9,11,13,30–32]. For instance, alumina/zirconia based ceramic composites with a layered structure designed with strong interfaces have been reported to exhibit relatively large apparent fracture toughness, energy absorption capability and, consequently, non-catastrophic failure behaviour [11,21,23,27,29,33–37]. However, the high level of fracture energy provided by laminates designed with weak interfaces has not been achieved in these systems.

The understanding of the conditions under which such energy dissipating mechanisms occur and the influence of the layered architecture on the crack propagation must be assessed in order to improve such ceramic designs. The motivation of this work is to investigate the conditions which may favour the presence of different energy release mechanisms in a unique layered ceramic architecture during crack propagation, considering its architectural design and material properties. Among the different mechanisms available, crack bifurcation and crack deflection along the interface (i.e. interface delamination) are studied in detail based on a crack deflection/penetration criterion for bi-materials as theoretical framework [38] and on experimental results of a reference layered structural (alumina–zirconia) ceramic previously investigated [29,39].

2. Experiments on layered ceramics

2.1. Material of study

A layered ceramic system consisting of alternated layers of alumina with 5 vol.% content of tetragonal zirconia (Al_2O_3 –5 vol.% tZrO_2), named A, and layers of alumina with 30 vol.% content of monoclinic zirconia (Al_2O_3 –30 vol.% mZrO_2), referred to as B, was fabricated by sequential slip casting. The procedure is described elsewhere [40]. Samples were sintered at 1550 °C for 2 h using heating and cooling rates of 5 °C/min. As a result, a symmetrical multilayered system with four thin B layers sandwiched between five thick A layers was obtained (Fig. 1). Due to the differential thermal strain between adjacent layers, associated with the $t \rightarrow m$ zirconia phase transformation in layers B, biaxial residual stresses (parallel to the layer plane) appear within the layers during cooling down from sintering. They are tensile in the A layers and compressive in the B ones [29]. In Table 1, the material properties measured in layers A and B are presented [13,29,40,41].

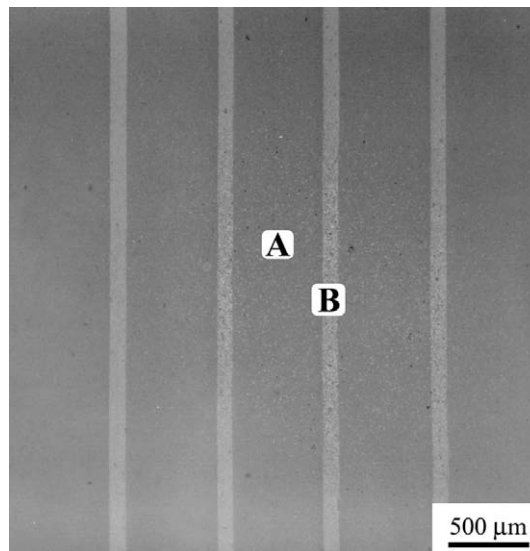


Fig. 1. SEM micrograph of an alumina–zirconia layered architecture designed with residual stresses and strong interfaces.

Table 1

Material properties measured in monolithic specimens corresponding to layers A and B.

Layer	Thickness (μm)	E (GPa)	ν (-)	μ (GPa)	CTE ($\times 10^{-6}$) ($^{\circ}\text{C}^{-1}$) (20–1200 $^{\circ}\text{C}$)	Res. stress (MPa)	σ_f (MPa)	K_{Ic} (MPa $\text{m}^{1/2}$)	G_{layer} (J/m^2)
A	540 ± 10	390 ± 10	0.22	160 ± 4	9.8 ± 0.2	$+100 \pm 5$	482 ± 65	3.2 ± 0.1	26 ± 1
B	95 ± 5	290 ± 15	0.22	119 ± 6	8.0 ± 0.2	-690 ± 8	90 ± 20	2.6 ± 0.1	23 ± 1

2.2. Mechanical behaviour

The mechanical response of this layered ceramic has been investigated elsewhere under different loading scenarios [29,31,32,39]. The high compressive biaxial stress in the thin B layers yields a so-called “threshold strength”, *i.e.* a minimum stress level below which the material does not fail independent of original defect size, such that failure tends to take place under conditions of maximum crack growth resistance [13]. As a consequence, the presence of relative large cracks in the outer layer (A) would not lead to catastrophic failure of the layered structure (the initial crack may arrest at the compressive layer, as seen in Fig. 2), thus increasing the reliability of the system.

The further propagation of the arrested cracks into the next layers under applied stress can be seen in Fig. 3. A typical step-wise fracture can be observed, which is caused by the compressive layers hindering and/or deviating the initial straight crack path. In this regard, crack bifurcation has mainly been found in this kind of laminates, as energy dissipating mecha-

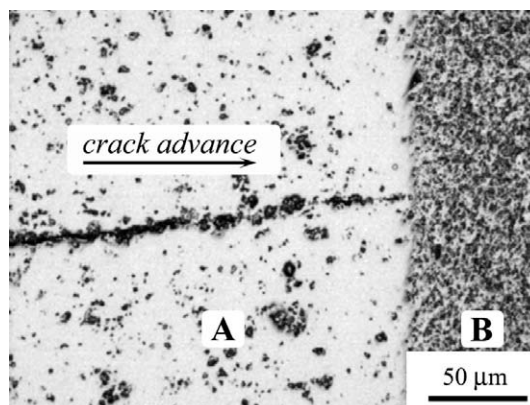


Fig. 2. Optical micrograph of a crack approaching the compressive layer B and arresting at the A/B interface.

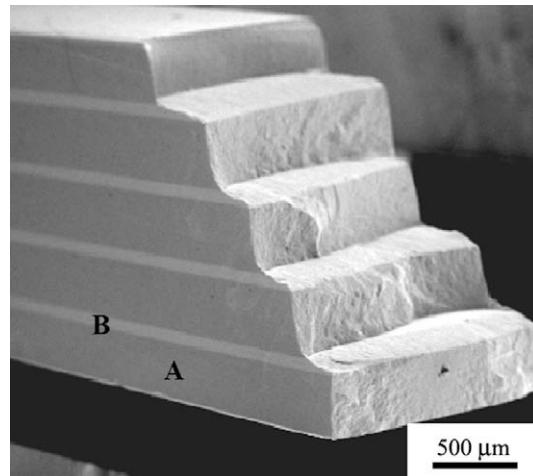


Fig. 3. SEM micrograph showing the step-like fracture of a laminate associated with the compressive layers which hinder the straight crack propagation.

nism. Compared to alumina-based monolithic ceramics the fracture toughness of the multilayer significantly increases [29]. In most cases, bifurcation mechanisms take place right after the crack has penetrated into the thin compressive layer, as seen in Fig. 4. In fact the combination of the magnitude of compressive stresses and of the thickness of the layer conditions the crack bifurcation angle; φ [42,43].

Experimental observations of the crack path in the multilayered ceramics tested under several flexural conditions (e.g. monotonic-, cyclic loading [29,32], thermo-mechanical loading [39], etc.) showed crack penetration (i.e. crack propagating normal to the layers; $\varphi = 90^\circ$) followed by crack bifurcation when the crack propagated from layer A into layer B (from the tensile to the compressive layer). Then the bifurcated crack inside layer B propagated towards the next layer impinging the B/A interface with a new angle, $\varphi \neq 90$. In order to rationalize the conditions for crack propagation in these layered ceramics (i.e. whether the crack penetrates through or deflects along the interface) a fracture mechanics approach proposed by He and Hutchinson (HH) [38] will be discussed in the following section.

3. Modelling of crack penetration or deflection in bi-materials

More than 20 years ago, He and Hutchinson analysed the conditions for a crack to penetrate into or deflect along the interface of two dissimilar materials (having different elastic and/or mechanical properties) [38]. The tendency of a crack approaching the interface between materials B and A with $\varphi = 90^\circ$ either to penetrate through the next layer or to deflect along the interface depends on the relations between the involved fracture energies (of material layer A and B, G_{layer} , or of the interface G_i) and the relevant energy release rates (of penetrating and deflecting cracks, G_p and G_d respectively). Penetration occurs if the ratio G_i/G_{layer} is greater than the ratio G_d/G_p and vice versa. This also depends on combinations of some material parameters associated with their elastic properties, the so-called Dundurs' parameters, α and β [44]:

$$\alpha = [\mu_A(1 - \nu_B) - \mu_B(1 - \nu_A)] / [\mu_A(1 - \nu_B) + \mu_B(1 - \nu_A)] \quad (1a)$$

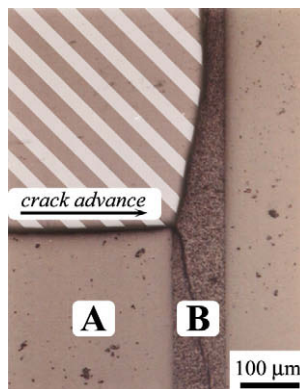


Fig. 4. Crack bifurcation mechanism along the center of the thin compressive layer B.

$$\beta = [\mu_A(1 - 2\nu_B) - \mu_B(1 - 2\nu_A)] / [\mu_A(1 - \nu_B) + \mu_B(1 - \nu_A)] \quad (1b)$$

where μ and ν are the corresponding shear modulus and Poisson's ratio respectively; the indexes A and B refer to the corresponding layers. Given the shear modulus as $\mu = E/2(1 + \nu)$, the first and more important parameter α can be expressed as:

$$\alpha = \frac{E'_A - E'_B}{E'_A + E'_B} \quad (2)$$

where $E' = E/(1 - \nu^2)$ is the plain strain elastic modulus, E the Young's modulus and ν the Poisson's ratio of the corresponding layers A and B. Assuming a bi-material with a reference small crack a (propagating from B to A) with the tip at the interface the traction ahead of the crack in layer A is given by the following equation:

$$\sigma_{xx}(0, y) = k_1(2\pi y)^{-\lambda} \quad (3)$$

where k_1 is proportional to the applied load and the stress singularity exponent λ is a real number that depends on α and β . More details can be found elsewhere [45]. Indeed the singularity exponent $\lambda = 0.5$ if both materials have the same elastic properties, as predicted by conventional linear elastic fracture mechanics.

The crack may advance mainly in two ways: (a) straight, penetrating into layer A (Fig. 5a or b) deflecting along the B/A interface (Fig. 5b).

In case of penetration into layer A, the stress state at the crack tip is pure mode I. The stress intensity factor

$$K_I = c(\alpha, \beta) \cdot k_1 a_p^{(0.5-\lambda)} \quad (4)$$

depends on the parameter k_1 and crack penetration length a_p according to [38] (see Fig. 5a). c is a dimensionless parameter which depends on α and β . Normally it ranges between 0.8 and 1.2 [46]. The corresponding energy release rate is:

$$G_p = \frac{1}{E'_A} K_I^2 = \frac{1}{E'_A} c^2 k_1^2 a_p^{(1-2\lambda)} \quad (5)$$

In case of crack deflection along the interface B/A the traction on the interface directly ahead of the deflected crack tip can be expressed using the complex notation given by Rice [47]:

$$\sigma_{xx}(x, 0) + i\sigma_{xy}(x, 0) = (K_1 + iK_2) \cdot (2\pi r)^{-1/2} r^{i\epsilon} \quad (6)$$

where K_1 and K_2 can be considered to be the conventional mode I and mode II stress intensity factors, $r = x - a_d$, and $\epsilon = (1/2\pi)\ln((1 - \beta)/(1 + \beta))$. The crack deflection length is called a_d (see Fig. 5b). According to He et Hutchinson, dimensional considerations require that

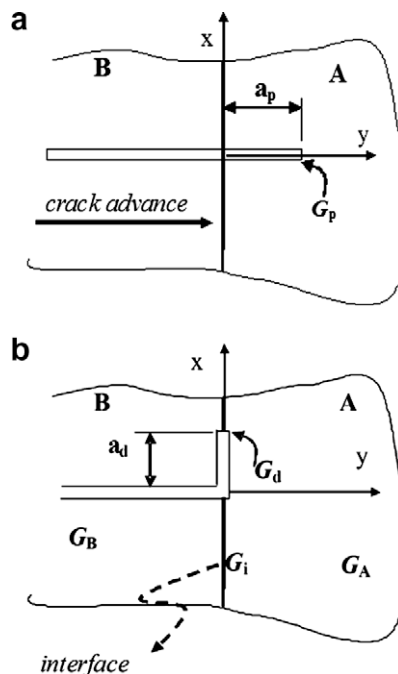


Fig. 5. Scheme of a crack propagating in a bi-material: (a) crack penetration and (b) crack deflection.

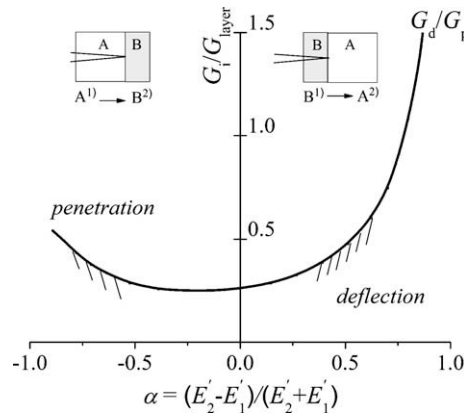


Fig. 6. Crack deflection/penetration criterion for a crack propagating normal to the interface of two dissimilar materials B and A.

$$K_1 + iK_2 = k_1 a^{(0.5-\lambda)} [d(\alpha, \beta) \cdot a_d^{ie} + e(\alpha, \beta) \cdot a_d^{-ie}] \quad (7)$$

where d and e are dimensionless complex functions of α and β defined in [38]. Thus, the energy release rate of the deflected crack results in:

$$G_d = \left[\left(\frac{2}{E'_A} \right) + \left(\frac{2}{E'_B} \right) \right] (K_1^2 + K_2^2) / (4 \cosh^2 \pi \varepsilon) \quad (8)$$

The ratio G_d/G_p is independent of a_d (and a_p) and k_1 and is given by:

$$G_d/G_p = [(1 - \beta^2)/(1 - \alpha)] \cdot [|d|^2 + |e|^2 + 2\text{Re}(d \cdot e)] / c^2 \quad (9)$$

The influence of the parameter β on this ratio is not significant and thus $\beta = 0$ has been assumed for the following analysis. The ratio G_d/G_p is presented in Fig. 6 as function of α on a so-called HH plot. A crack propagating from layer B to layer A would deflect along the interface if $G_i/G_A < G_d/G_p$. Likewise the crack will tend to penetrate when the inequalities are reversed.

This analysis has been extended for the laminates of study to take into account the effect of residual stresses and of the inclining crack angle φ observed in the experiments (e.g. during crack bifurcation) in order to establish guidelines for the design of layered structures with optimised mechanical behaviour.

4. Results and discussion

4.1. Effect of residual stresses on the crack propagation in layered ceramics

Loading conditions and geometry of the system influence the energy release rate. Therefore, internal stresses (which act similar to external stresses) have to be considered. He et al. extended the analysis described above to bi-materials in that respect [46]. In the presence of normal (σ_n) and/or tangential (σ_t) residual stresses two additional non-dimensional length parameters (η_n and η_t) become important. They are defined as [46]:

$$\eta_n = \frac{\sigma_n \cdot a_d^\lambda}{K_I} \quad \text{and} \quad \eta_t = \frac{\sigma_t \cdot a_p^\lambda}{K_I} \quad (10)$$

where K_I is a factor proportional to the applied stress field, as reported in [38]. The stress singularity exponent λ depends on the elastic mismatch of the layers. For the case of laminates, where the elastic mismatch between layers is not too large, it holds: $\lambda \approx 0.5$. In layered ceramics, the η_n parameter (related to the stresses normal to the interface) is usually zero, and the occurrence of interface delamination is dominated by η_t . For the case of a crack propagating towards a layer with compressive stresses, it holds $\eta_t < 0$, what enhances interface delamination. On the other hand, when the elastic mismatch is not so significant crack penetration is more likely to occur.

In order to calculate η_t , the characteristic flaw size (a_p) has to be known. For our laminates we assume that a_p is a typical micro structural feature and take the mean grain size of these materials (i.e. $\sim 1 \mu\text{m}$) as initial defect size¹. For the crack propagating from A to B or from B to A the corresponding λ has been interpolated out of the values given by He et al. in Table 1 in Ref. [46] as function of α , resulting in $\lambda \approx 0.53$ and $\lambda \approx 0.48$ respectively (it can be observed how λ is approximately 0.5 in every case). Finally, K_I has been chosen as the fracture toughness for each layer, as given in Table 1.

¹ This assumption has been made for other authors when trying to estimate the η_t parameter (see for instance Ref. [17]), or for the determination of fracture toughness of ceramics with the SENB-S method (see Ref. [48]).

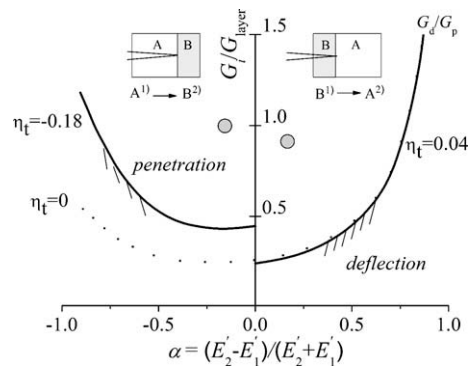


Fig. 7. Crack deflection/penetration criterion for a crack propagating normal to the interface between layers A and B, where the layers have residual stresses. G_i/G_B and G_i/G_A (corresponding to the laminate investigated) are represented as full symbols, remaining in the region of crack penetration.

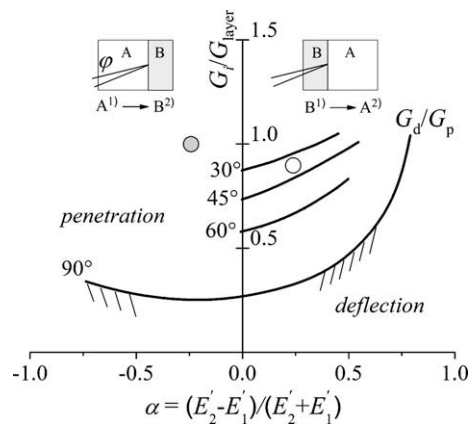


Fig. 8. Crack deflection/penetration criterion for a crack propagating with different angles towards the interface. G_i/G_B is represented as full symbol, lying in the region of crack penetration. G_i/G_A is represented as empty symbol, under the G_d/G_p curve corresponding to 30° , remaining in the region of crack deflection.

In Fig. 7, the η_t curves corresponding to our laminates (with compressive residual stresses in layers B) are represented on a HH plot [46]. Remember that such multilayered architecture consists of thick A layers alternated with thin B layers (see Fig. 1), which have $\approx +100$ MPa and ≈ -690 MPa in-plane residual stresses respectively [29]. For comparison, the case for zero residual stresses, $\eta_t = 0$, is also presented with a dotted-line. It can be observed that, in case the crack propagates from layer A to layer B the compressive residual stresses in layer B yield a negative η_t ($\eta_t = -0.18$). This leads to an upwards shift of the G_d/G_p curve, thus enhancing crack deflection along the interface. On the other hand, for a crack propagating from B to A η_t is almost zero ($\eta_t = 0.04$), hence there is not significant effect on the HH plot. Therefore, the presence of high compressive stresses in layer B might favour crack deflection along the interface only when the crack would propagate from layer A to layer B². Nevertheless, by representing the corresponding G_i/G_B and G_i/G_A values in Fig. 7 (see full symbols), for the corresponding $\alpha = \pm 0.15$ (the interface fracture toughness G_i has been assumed as the toughness of layer B, i.e. $2.6 \text{ MPam}^{1/2}$, based on indentation fracture (IF) experiments), it can be inferred that the tendency for crack deflection is not favoured in layered ceramics with strong interfaces even in presence of relative high residual stresses. Hence, it can be concluded that the effect of the residual stresses does not play any significant role for the crack deflection/penetration conditions, when the crack approaches the interface with an angle of $\varphi = 90^\circ$.

4.2. Influence of the impinging angle for interface delamination

He et al. analysed the influence of the angle on deflection/penetration mechanisms in bi-materials [46]. They demonstrated that the tendency for a crack to deflect along the interface increases for small impinging angles ($\varphi \ll 90^\circ$). As reported

² For the case of laminates with high porosity (i.e. with a pore-like defect size, $2a \approx 10 \mu\text{m}$ [49]), the parameter η_t would result in $\eta_t = -0.4$ and $\eta_t = 0.1$ for a crack propagating from A to B and from B to A respectively, which would favour interface delamination.

above, experimental observations of the crack path in our layered system showed in fact bifurcation effects in the compressive layers (Fig. 4). In such cases, the crack branches (as it enters the compressive layer B) and thus faces the B/A interface with a new angle of propagation $\varphi \ll 90^\circ$. Therefore, in Fig. 6 an “upwards” correction of the curves is required. Now, a tendency for crack deflection along the interface might be feasible.

Fig. 8 sketches the new curves for penetration/deflection of a crack approaching the interface of the laminate of study with different angles, using again the HH plots [38]. Under these conditions, and considering the correct angle of crack propagation, the inequality $G_i/G_A < G_d/G_p$ is easier to be fulfilled, and thus crack deflection along the interface is now more likely to occur. This tendency of a bifurcating crack to deflect along the interface (in this case along the B/A interface) has been in fact experimentally evidenced by the authors in the layered ceramics of study under certain loading conditions (*i.e.* flexural loading at relative high temperatures (*e.g.* 800° C) [39]), as it can be seen in Fig. 9. In such cases, the bifurcation angle ranged approximately from $\varphi = 25^\circ$ to $\varphi = 35^\circ$. In addition, the Young’s moduli of the layers at the testing temperature (*i.e.* 360 GPa for layer A and 220 GPa for layer B [39]) increased the absolute value of the parameter α up to ≈ 0.24 , what also promotes crack deflection along the interface. Now, if we consider the new G_d/G_p curve in Fig. 8 corresponding to an angle of 30° , it holds for our material (empty symbol in Fig. 8): $G_i/G_A < G_d/G_p$, *i.e.* the crack should deflect along the interface (as found experimentally) when approaching layer A. In such cases the failure resistance of the laminate can be significantly increased due to the subsequent action of crack bifurcation and interface delamination.

4.3. Guidelines to design of layered ceramics

The theoretical approach using the HH plot confirmed by experimental observations raises the query whether an optimal design for multilayered architectures should be pursued that uses crack bifurcation followed by interface delamination. In comparison with monolithic ceramics, the effect of both mechanisms would significantly enhance the failure resistance of the system. In addition, the fact that the bifurcating crack is prone to deflect along the interface would prevent the material from catastrophic failure (as for the case of layered ceramics with weak interfaces), thus increasing the mechanical reliability of the component.

In previous work of the authors, it has been shown that crack bifurcation occurs if the product of layer thickness and the square of the compressive stress ($t \cdot \sigma_c^2$) exceeds a critical value [50]. These results have also been supported by finite element analyses [8,51–54]. Thus, an optimal laminate design should consist of compressive layers, which are thin enough to ensure a high threshold strength (*i.e.* the thinner the compressive layer, the higher are the compressive residual stresses, and thus higher strength) but thick enough to induce crack bifurcation [29]. The angle of bifurcation depends on the magnitude of compressive stresses (and therefore again on the layer thickness) [29,55]. If the angle is low enough, interface delamination will also occur. Therefore, an optimal design that favours small crack bifurcation angles should contain high compressive stresses, which can be obtained with thin compressive layers, bearing in mind that the thickness should always remain above the critical thickness for promoting crack bifurcation [50].

In addition, the Dundur parameter α (which depends on the elastic constants of the layers) is also relevant for the delamination behaviour. To promote deflection along the interface α should be as large as possible (see Fig. 8). To give an example based on the material properties reported in Table 1 ($E_A = 390$ MPa and $E_B = 290$ MPa) the coefficient is $\alpha \approx \pm 0.15$. By

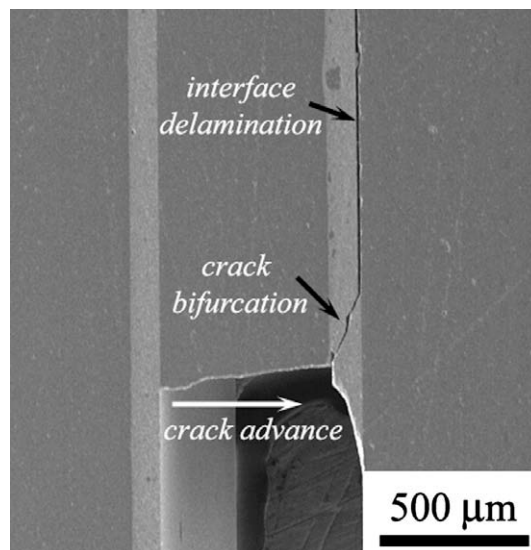


Fig. 9. SEM micrograph of a multilayer tested under flexure at 800 °C, where a bifurcating crack approaches the B/A interface causes interface delamination, while the structure underneath remains intact.

decreasing for instance the stiffness of layer B by 20% (e.g. it may be achieved by increasing the porosity of the layer in approximately 10%)³ the parameter results in $\alpha \approx \pm 0.25$. This would increase the critical ratio of the strain energy release rates G_d/G_p (the curve would shift upwards) by approximately 10% (see Fig. 8).

Summarising, an optimal design that favours crack bifurcation mechanisms followed by interface delamination strongly depends on the magnitude of compressive stresses which is associated with the layered architecture (*i.e.* layer thickness, composition) and elastic properties of the layers. These parameters are intrinsically related and should be taken into account when a high failure resistance is pursued for such layered structures.

Although this analysis was based on experimental observations on alumina–zirconia layered ceramics, it can be used to optimise other laminate systems which also hold such energy release mechanisms.

5. Conclusions

The combination of crack bifurcation and interface delamination in layered ceramics with strong interfaces should be pursued in order to enhance the failure resistance of such architectures. It has been found that an optimal design which favours crack bifurcation mechanisms followed by interface delamination is strongly dependent on: (a) the level of compressive stresses (which depends on the layer thickness ratio and differential strain between layers), (b) the combination of layer thickness and compressive stresses (in the ratio $t \cdot \sigma_c^2$), which provokes crack bifurcation, and (c) the inclined angle of bifurcated cracks (which again depends on the compressive stresses) and the elastic mismatch between the layers, which both will favour interface delamination.

A critical design parameter is the thickness of the compressive layers, which – for a given system – has an influence on almost all critical parameters (compressive stress level, occurrence of bifurcation, inclination angle and delamination).

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³ We caution the reader that higher porosity contents could lower the magnitude of compressive stresses and thus bifurcation mechanisms might not take place [56].

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