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Failure Resistance Optimisation in Layered Ceramics Designed with Strong Interfaces

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Abstract

Layered ceramics designed with weak interfaces favour interface delamination and provide high failure resistance, while laminates with strong interfaces show higher strength and enhanced mechanical reliability. In this paper it is proposed to combine crack bifurcation and interface delamination in a unique layered architecture with strong interfaces to optimise both failure resistance and mechanical reliability.

Theoretical approaches from He and Hutchinson for delamination in bi-materials and from Lange *et al.* for crack bifurcation in multilayers are here employed to define an optimal design. These approaches are supported by own experimental results on an alumina-zirconia multilayer system designed with compressive stresses and strong interfaces. It is shown that a favourable condition for interface delamination occurs for bifurcated cracks if such cracks have a low inclination angle towards the next interface. The occurrence of bifurcation is triggered by the thickness of the compressive layers as well as by the compressive stresses, which also influence the bifurcation angle. Therefore, they are the key features for optimising these multilayered systems.

Keywords: Layered ceramics, failure resistance, crack bifurcation, interface delamination.

I. Introduction

Layered ceramics have been proposed as an alternative choice for the design of structural ceramics with improved fracture toughness and mechanical strength reliability. As a result, the brittle fracture of monolithic ceramics has been overcome by introducing layered architectures of 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 and to increase the strength reliability of the end-component.

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 significant enhanced failure resistance and R-curve behaviour through interface delamination[1-8]; 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 significant crack growth resistance (R-curve) behaviour through microstructural design (*e.g.* grain size, layer composition)[9-12] and/or due to the presence of compressive residual stresses, acting as a barrier to crack propagation[3, 13-21]. The increase in fracture energy in these laminates may be associated with energy-dissipating mechanisms such as crack deflection/bifurcation phenomena. In particular, the utilisation 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[14, 16, 20, 22-25]. 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[9, 15, 16, 19, 26-28]. However, the high failure resistance found in weak interfaces laminates has not been achieved in systems designed with strong interfaces.

The motivation of this work is to identify the conditions which favour the presence of both crack bifurcation and interface delamination mechanisms in a unique layered ceramic architecture during crack propagation. This study is based on a crack deflection/penetration approach for bimaterials as theoretical framework (established by He and Hutchinson[29]) and on experimental results of a reference layered structural (alumina-zirconia) ceramic previously investigated by one of the authors[19, 30]. In this work it is recognised that in laminates designed with strong interfaces crack bifurcation can be followed by interface delamination, thus combining the beneficial effects of both energy release mechanisms. The conditions for the occurrence of this beneficial situation are discussed and the idea

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of laminates which use crack bifurcation followed by interface delamination is established.

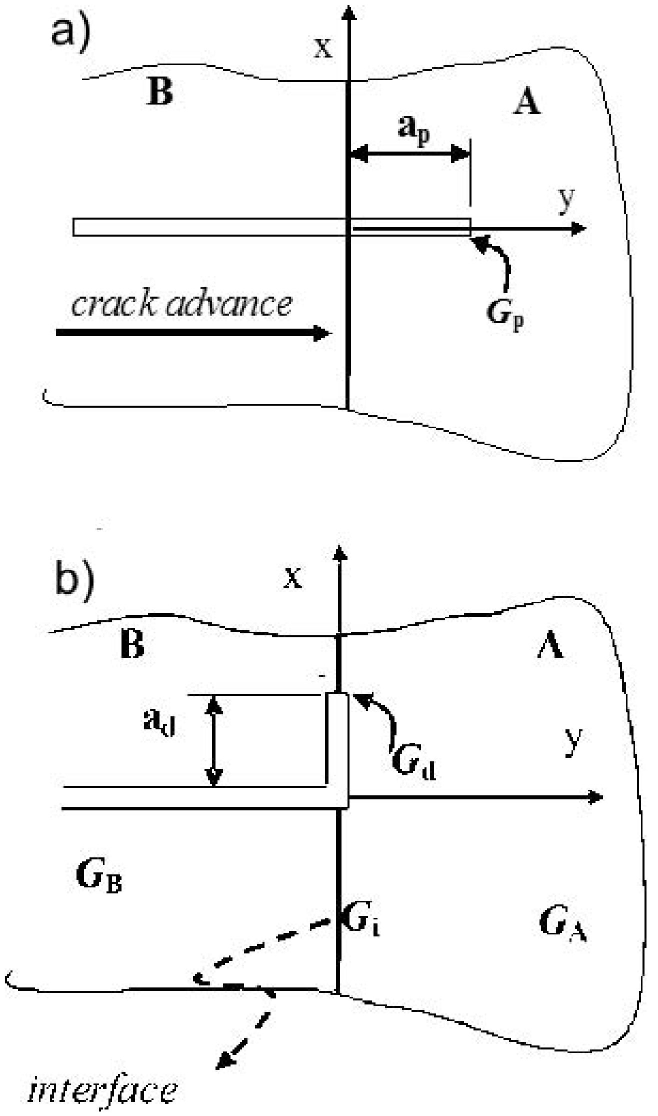

Fig. 1: Scheme of a crack propagating in a bi-material: a) Crack penetration and b) crack deflection.

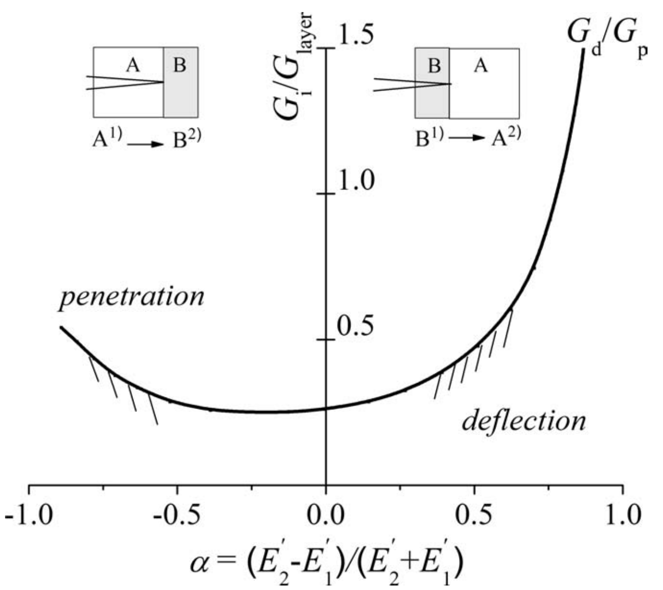

Fig. 2: Crack deflection/penetration criterion for a crack propagating normal to the interface of two dissimilar materials **B** and **A**[29].

II. Theoretical approach for bi-materials

The conditions for a crack to penetrate into or deflect along the interface of two dissimilar materials (having different elastic and/or mechanical properties) were first studied by He and Hutchinson[29]. The tendency of a crack, approaching with an angle j the interface between two materials **B** and **A,** either to penetrate through the next layer (see fig. 1a) or to deflect along the interface (see fig. 1b) depends on the relations between the involved fracture energies (of the material layer **A** and **B**, G_{layer}, and of the interface G_i) and the relevant energy release rates (of deflecting and penetrating cracks, G_d and G_p respectively). This also depends on combinations of the elastic properties of layers A and B, as described by the Dundurs parameters α and β[31]:

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

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

where μ and υ 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 $\mu = E/2(1+\upsilon)$, the most important parameter, α, can be expressed as:

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

where $E' = E/(1-\upsilon^2)$ is the plain strain elastic modulus, *E* the Young's modulus and υ the Poisson's ratio of the corresponding layers **A** and **B**.

In case that the crack penetrates into layer **A** the corresponding energy release rate, G_p, is given by[29]:

$$G_p = \frac{1-\upsilon_A}{2\mu_A} K_I^2 \quad (3)$$

In case of crack deflection along the interface **B/A** the energy release rate of the deflected crack, G_d, results in[29]:

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

where K_1 and K_2 can be considered to be the conventional mode I and mode II stress intensity factors, and $\varepsilon = (1/2\pi)\ln((1-\beta)/(1+\beta))$.

The ratio G_d/G_p is represented in fig. 2 as a function of α on a so-called HH plot. For more details refer to[29]. Hence, 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 if $G_i/G_A > G_d/G_p$.

III. Experiments on layered ceramics

The mechanical behaviour of the alumina-zirconia layered ceramics investigated here have been published elsewhere[19, 20, 25, 32, 33]. 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 in[34]. Samples were sintered at 1550 °C for 2 hours using heating and cooling rates of 5 °C/min. As a result, a symmetrical multilayered system with 4 thin **B** layers sandwiched between 5 thick **A** layers was obtained. In fig. 1a multilayer with a total thickness of *ca.* 3 mm and a layer thickness ratio of $t_A/t_B \approx 5$ is presented. Due to the differential thermal strain between adjacent layers, associated with the t → 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[19]. In Table 1 the magnitude of these residual stresses along with the material properties measured in layers **A** and **B** in previous works are presented[19, 20, 34, 35]. The mechanical response of this layered ceramic has been characterised in detail under different loading scenarios[19, 24, 25, 30]. Some of the experimental observations will be used for this investigation. Special attention will be drawn to experiments where bifurcation and interface delamination phenomena have been observed. The fracture mechanics approach described above will be used to rationalise the conditions which may favour the appearance of both phenomena in a unique multilayer design.

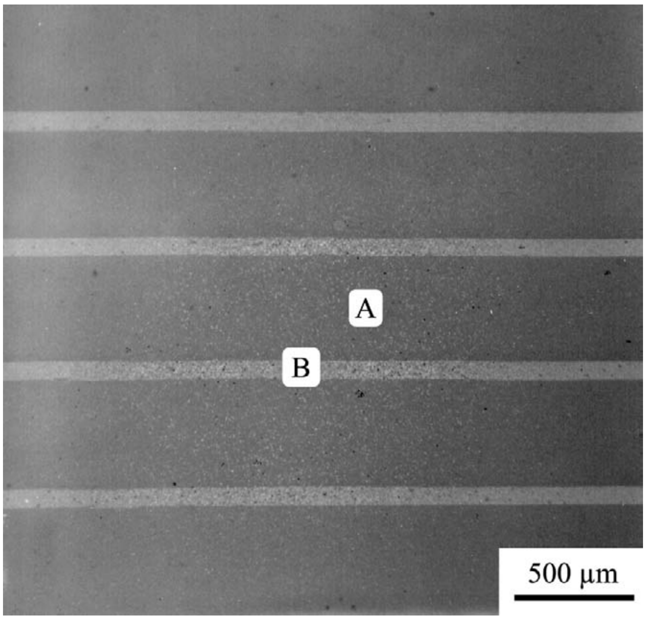

Fig. 3: SEM micrograph of an alumina-zirconia layered architecture designed with residual stresses and strong interfaces.

IV. Results and discussion

The propagation of cracks through the laminate investigated, under applied bending stress, takes place in a tortuous way (step-wise fracture), which differs from the straight crack path mostly found in monolithic ceramics. This is caused by the compressive layers hindering and/or deviating the crack from straight propagation. Crack bifurcation can be observed as the main energy dissipating mechanism acting during fracture, which takes place right after the crack has penetrated into the thin compressive layer, as it can be clearly seen in fig. 4. The angle, j, with which the bifurcating crack further propagates through the compressive layer, depends on two important parameters: i) the magnitude of compressive stresses and ii) the thickness of the layer, as shown in the literature[36, 37].

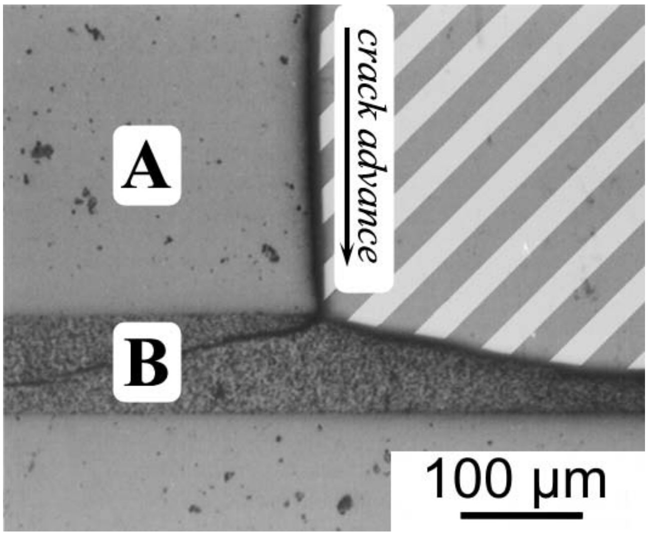

Fig. 4: Crack bifurcation along the centre of the thin compressive layer **B**.

Experimental observations of the crack path in the layered ceramics of study tested under several flexural conditions (*e.g.* monotonic-, cyclic loading[19, 25], thermo-mechanical loading[30], etc.) showed crack penetration (*i.e.* crack propagating normal to the layers; $\varphi = 90°$) followed by crack bifurcation when the crack propagated from layer **A** into layer **B** (from the tensile to compressive layer). Then the bifurcated crack inside layer **B** advanced towards the next layer impinging the **B/A** interface with a new angle, $\varphi \neq 90$. Very interesting, it has been experimentally evidenced that, under certain loading conditions (*i.e.* flexural loading at relative high temperatures (*e.g.* 800° C)[30]), crack bifurcation can be followed by interface delamination, as it can be clearly seen in fig. 5*). In such experiments, the bifurcation angle ranged approximately between $\varphi = 25°$ and $\varphi = 35°$, which raises the query whether the combination of both mechanisms in a solely multilayer design could be achieved.

*) Under flexural loading at elevated temperatures (800 °C), although the compressive stresses at 800 °C decreased down to -250 MPa, bifurcation could be observed[30].

Table 1: Material properties measured in monolithic specimens corresponding to layers **A** and **B**.

Layer	Thickness [µm]	*E* [GPa]	υ [–]	*CTE* (20-1200 °C) [$\times 10^{-6}$ °C^{-1}]	Res. Stress [MPa]	K_{Ic} [MPam$^{1/2}$]	G_c [J/m^2]
A	540 ± 10	390 ± 5	0.22	9.8 ± 0.2	+100 ± 5	3.2 ± 0.1	26 ± 1
B	95 ± 5	290 ± 5	0.22	8.0 ± 0.2*	-690 ± 8	2.6 ± 0.1	23 ± 1

* The volume change associated with the t→m zirconia phase transformation has been taken into account in the *CTE* of layers **B**.

It is known that the tendency of a crack to deflect along the interface increases for small impinging angles (φ)[29]. In fig. 6 the curves for penetration/deflection of a crack approaching the interface of the laminate of study with different angles are shown, using the HH plots[29]. For the angle of crack propagation experimentally observed, the inequality $G_i/G_A < G_d/G_p$ is now easier to be fulfilled compared to cracks propagating perpendicular, and thus crack deflection along the interface is now more likely to occur. In addition the Young's moduli of the layers (*i.e.* 360 GPa for layer **A** and 220 GPa for layer **B** measured at the testing

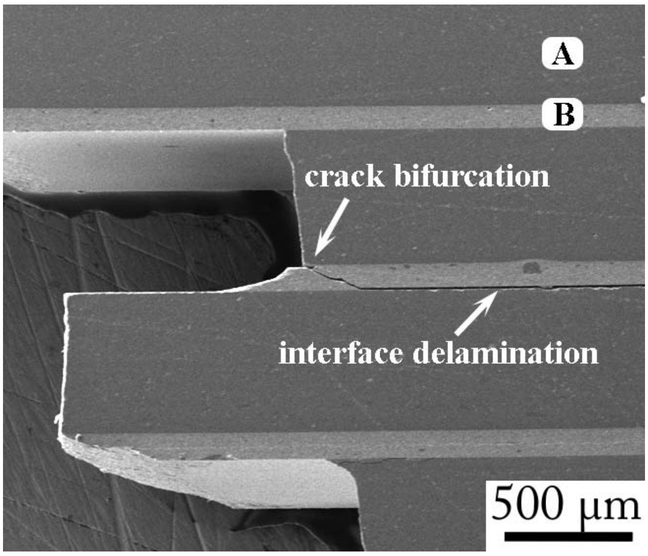

Fig. 5: SEM micrograph of a multilayer tested under flexure at 800 °C. The bifurcating crack approaches the **B**/**A** interface and delaminates along the interface, while the structure underneath remains intact.

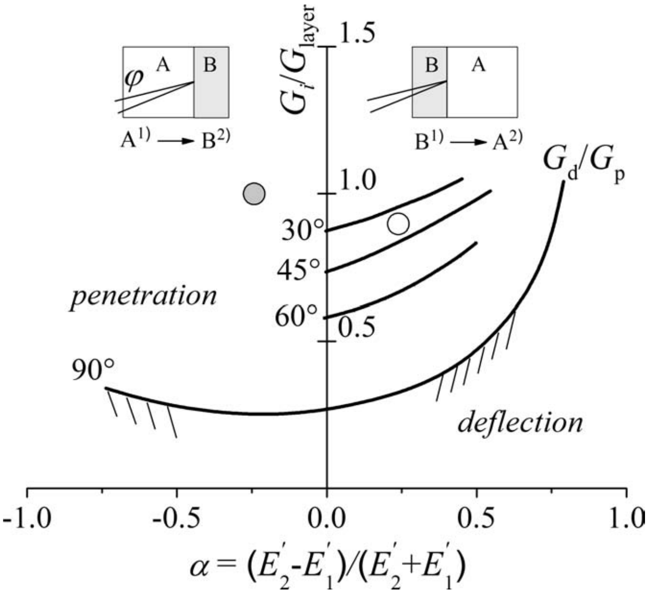

Fig. 6: Crack deflection/penetration criterion for a crack propagating with different angles towards the interface. G_i/G_B is represented as full symbol, lying in the region of crack penetration. G_i/G_A is represented as empty symbol, under the G_d/G_p curve corresponding to 30°, remaining in the region of crack deflection along the interface.

temperature[30]) increased the absolute value of the Dundur's parameter α up to ≈ 0.24, which also promotes interface delamination[38] (as it can be inferred from fig. 3 and fig. 6). For the case analysed, and assuming the interface fracture energy between layers **B** and **A**, G_i, as the fracture energy of layer **B**, *i.e.* 23 J/m², (based on indentation fracture (IF) experiments), the corresponding ratios G_i/G_A and G_i/G_B can be calculated. Now considering the G_d/G_p curve in fig. 6 corresponding to a bifurcating angle of 30°, it holds for our material (empty symbol in fig. 6): $G_i/G_A < G_d/G_p$, *i.e.* the crack will deflect along the interface (as found experimentally) when propagating from layer **B** into layer **A**.

From the fracture resistance point of view, by comparing the load-displacement curves registered during the referred experiments (*i.e.* four-point bending test on indented specimens) with a monolithic taken as reference, it can be observed in the former several step-wise fracture events (fig. 7), which correspond to interface delaminations at several **B**/**A** interfaces, as it can be appreciated in fig. 5. Attempting to quantify the work of fracture, γ_{WOF}, the area under the load-displacement curve of the referred figure was divided by twice the cross-section of the specimen according to[39], giving as a result $\gamma_{WOF} = 16 \pm 1$ J/m² for the **A** monolith and $\gamma_{WOF} = 52 \pm 1$ J/m² for the multilayer*). Although far from the values obtained in weak interface composites, this result evidences the higher failure resistance of the multilayer structure designed with strong interfaces compared to the alumina-based monolith. Moreover 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 reliability of the system. In such cases the mechanical reliability of the laminate can be significantly increased due to the combined action of crack bifurcation and interface delamination.

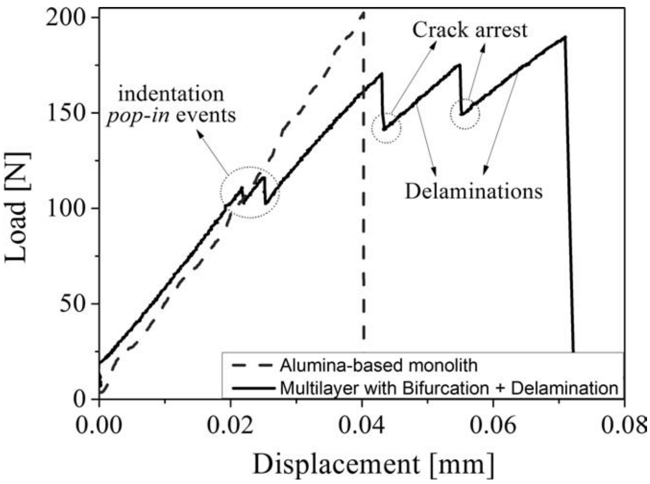

Fig. 7: Load-displacement curves of an indented monolithic and multilayer specimen under four-point bending. The crack propagation in the multilayer shows a step-wise fracture owing to the combination of bifurcation and interface delamination mechanisms, compared to the brittle and catastrophic failure of the alumina-based monolith.

In previous works it has been shown that crack bifurcation in laminates with strong interfaces occurs if the prod-

*) The $_{WOF}$-values were calculated from specimens indented in the same way. They should be only taken as relative comparison.

uct of layer thickness (t) and the square of the compressive stress (σ) exceeds a critical value according to[40-42]:

$$t \cdot \sigma_c^2 > \frac{E \cdot G_c}{0.34 \cdot (1 - \nu^2)}. \qquad (5)$$

With E, ν and G_c being respectively the Young's modulus, Poisson's ratio and fracture energy of the compressive layer. For our case, the right hand term of (5) based on the material properties of Table 1 results in $\approx 2.1 \times 10^7$ µm·MPa^2. For the layered architecture of study (see Table 1), the first term results in $\approx 4.5 \times 10^7$ µm·MPa^2 which fulfils (5). Thus, an optimal laminate design should consist of compressive layers, which are thin enough to ensure high compressive residual stresses but thick enough to induce crack bifurcation[19]. Moreover, the bifurcated cracks should have a small inclination angle φ with respect to the **B**/**A** interface, which decreases with the magnitude of the compressive stresses[37]. If the angle is low enough, interface delamination will be favoured. In addition, the first Dundurs parameter α (which depends on the elastic constants of the layers) is also relevant for the delamination behaviour. To promote deflection along the interface this parameter should be as large as possible, as it can be inferred from figs. 3 and 6. However, it has been demonstrated that the stiffer the approaching layer is, the easier the crack kinks out of the interface[38]. This is a kind of trade-off for interface cracking vs. kinking, which should be taken into account to optimise the layered architecture. In our case, this feature can be observed in fig. 5, where the bifurcating crack in layer **B** (E_B=*290 GPa*) which deflects along the **B**/**A** interface kinks out of the interface and penetrates into the **A** layer (E_A=*390 GPa*).

Summarising, the presence of interface delamination is associated with the angle with which the crack bifurcates when entering the compressive layers and with the elastic mismatch between adjacent layers. This angle can be tailored by inducing high compressive stresses in the multilayer design, the thickness of such compressive layers being the key parameter to be optimised. For the case of alumina-zirconia multilayer ceramics it has been found that crack bifurcation angles below 30° would lead to interface delamination, thus increasing the failure resistance of the laminate and the reliability of the system.

V. Conclusions

The combination of crack bifurcation followed by interface delamination is an effective way to increase toughness and reliability of layered ceramics designed with strong interfaces. This situation is favoured by the following conditions: (i) thin compressive layers to ensure high compressive residual stresses, but thick enough to induce crack bifurcation, (ii) a small inclination angle of the bifurcating crack which approaches the tensile layer, tailored again by the compressive stresses and (iii) high elastic mismatch between layers which favours interface delamination. Indeed, compressive stresses depend on the layer thickness as well as on the elastic mismatch and, therefore, changing one parameter has influence on the others. From the practical design point of view, the thickness of the compressive layers is the key design parameter to be optimised, but its value will depend on the multilayer system considered.

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