

## Fracture of layered ceramics

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**Abstract.** Layered ceramics are foreseen as possible substitutes for monolithic ceramics due to their attractive mechanical properties in terms of strength reliability and toughness. The different loading conditions to which ceramic materials may be subjected in service encourage the design of tailored layered structures as function of their application. The use of residual stresses generated during cooling due to the different thermal strain of adjacent layers has been the keystone for the improvement of the fracture response of many layered ceramic systems, e.g. alumina-zirconia, alumina-mullite, silicon nitride-titanium nitride, etc.

In this work, the fracture features of layered ceramics are addressed analysing two multilayered structures, based on the alumina-zirconia system, designed with tailored compressive residual stresses either in the external or internal layers. Contact strength and indentation strength tests have been performed to investigate the response of both designs to crack propagation. The experimental findings show a different response in terms of strength and crack growth resistance of both designs. While layered structures with compressive stresses at the surface provide a better response against contact damage compared to monoliths, a flaw tolerant design in terms of strength and an improved toughness through energy release mechanisms is achieved with internal compressive stresses. The use of layered architectures for automotive or biomedical applications as substitutes for alumina-based ceramics should be regarded in the near future, where reliable ceramic designs are needed.

### Introduction

The wide range of advanced ceramic components now available to meet the performance of complex engineering systems faces the designer with difficult choices for selecting the appropriate material for every particular application. This selection is not only based on the intrinsic properties of the material but also on its processing capabilities and the cost efficiency of the manufacturing process. The pressure for reaching a high quality product demands a high mechanical reliability of the material during service, which in most of the cases is conditioned by the processing steps and handling of the ceramic part when mounting onto the end component.

The interest for the mechanical behaviour of ceramic materials has been always motivated by their possible application as structural components, especially in the cases where properties such as high hardness, chemical stability, low density and high strength, among others, have been sought. In fact, ceramics have been used for many decades as structural elements, but almost always under effective compressive loading conditions. However, most of the new engineering designs need to withstand tensile stresses which imply potential limitations due to the inherent brittleness of ceramic materials. The brittle-like fracture of ceramics is a consequence of the material defects located either within the bulk or at the surface, resulting from the processing and/or machining procedures [1, 2]. This is an important issue 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 the crack geometry; hence, the size and type of these defects will determine the mechanical strength of the material [3]. As a result, structural ceramics exhibit a statistically variable brittle fracture which limits their use for load-bearing applications [4, 5].

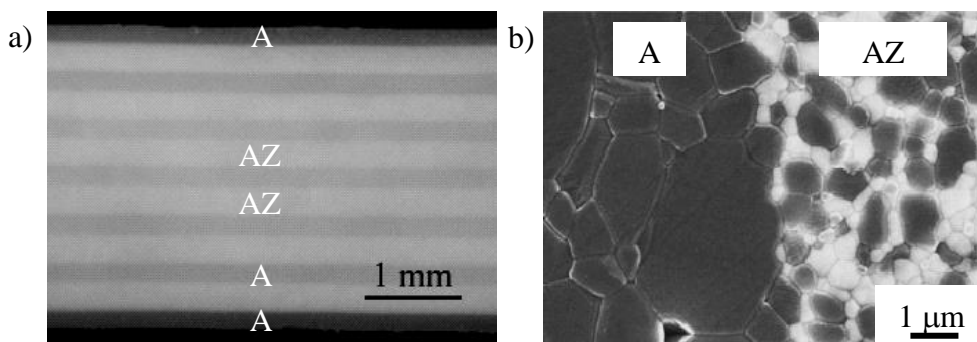
In recent years, as a direct consequence of remarkable progress in terms of microstructural design and advanced processing [6-8], toughness and strength reliability of structural ceramics have been increasingly enhanced by recourse to crack shielding resulting from microstructure-related mechanisms [9-16]. Particular attention has been paid to fibre and layered reinforced ceramics, where the better mechanical performance is associated with the second phase or layer addition as well as with the arrangement of the fibres or the layer assemblage [12, 17, 18]. As an extension of this laminar ceramic/fibre-reinforced concept, multilayered architectural designs have also been attempted in many ways aiming to improve both the resistance to crack propagation and the mechanical reliability of ceramic components [16, 19-27]. This approach has been demonstrated to be less cost effective than the one based on fibre structures and more accurate in terms of tailoring mechanical requirements. Within this context, a commonly used multilayered structural design is that associated with the presence of compressive residual stresses developed in the laminate during cooling from sintering, as related to differences in elastic or thermal properties (Young's modulus, thermal expansion coefficient, etc.) between the layers, as well as to phase transformations and/or chemical reactions within them [13, 21, 28]. The specific location of the compressive layers, either at the surface or internal, is associated with the attempted design approach, based on either mechanical resistance or damage tolerance respectively. In the former case, the effect of the compressive residual stresses results in a higher, but single-value, apparent fracture toughness together with enhanced strength (the main goal) and some improved reliability [20, 29, 30]. On the other hand, in the latter case, the internal compressive layers are microstructurally designed to rather act as stopper to any potential processing and/or machining flaw at or near the surface layers, independent of the original defect size (threshold strength), such that failure tends to take place under conditions of maximum crack growth resistance [22, 25, 26, 31]. In particular, ceramic composites with a layered structure such as alumina/zirconia have been reported to exhibit relatively large apparent fracture toughness, energy absorption capability and, consequently, non-catastrophic failure behaviour [13, 19, 21, 26, 32-36]. The selection of multilayered systems with tailored compressive stresses either at the surface or in the bulk should be based on the end application, conditioned by the loading scenarios where the material will work [37].

In this paper the fracture response of two alumina/zirconia layered architectures is addressed under different loading conditions, in order to establish an optimal material selection according to their end application. The two layered architectures here characterised are similar in composition, but differ in the location of the compressive residual stresses (either in the external or in the internal layers). Experimental and analytical case studies have been performed, covering the main loading situations which may occur during service. In this regard, the mechanical behaviour of the multilayered material designed with compressive residual stresses at the surface (envisaged for contact applications) has been assessed using contact loading and analysed based on a contact mechanics framework. On the other hand, the mechanical response for the multilayer material with internal compressive residual stresses (aimed to increase strength reliability) has been investigated under thermo-mechanical loading conditions based on a linear elastic fracture mechanics approach.

## Materials of study

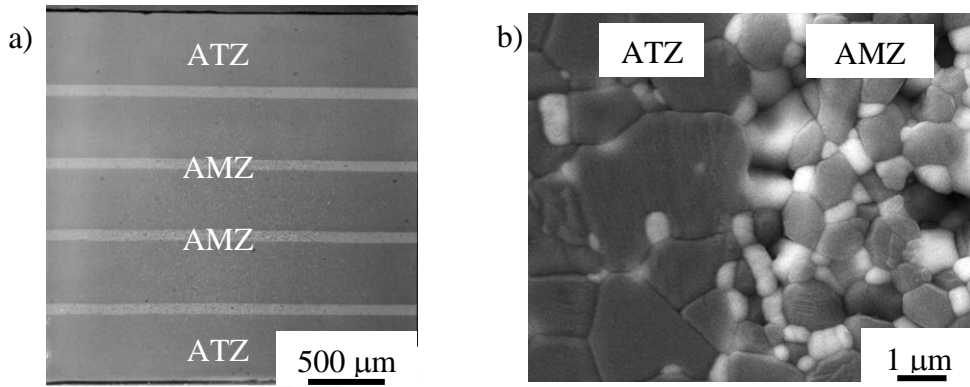
**Layered composites with external compressive layers (ECS-laminates).** The materials were manufactured at the Institute of Science and Technology for Ceramics (ISTEC), Faenza, Italy. Starting powders were high purity (99.7%) alumina (Alcoa A16-SG, Alcoa Aluminum Co., New York, USA) with a mean particle size ( $d_{50}$ ) of 0.3  $\mu\text{m}$ , and tetragonal zirconia polycrystals (TZ3Y-S, Tosho Corp., Japan) containing 94.7% of  $\text{ZrO}_2$  and 3 mol% of  $\text{Y}_2\text{O}_3$  (usually referred to as 3Y-TZP)

with  $d_{50} = 0.3 \mu\text{m}$ . Sheets of pure alumina (hereinafter designated as A) as well as of the composite alumina-zirconia (hereinafter designated as AZ) in the volume ratio 60/40 were prepared by tape casting. Details on the processing can be found elsewhere [38]. The thicknesses of the green tapes were selected in order to obtain, after sintering, layers of about  $200 \mu\text{m}$  (A) and  $250 \mu\text{m}$  (AZ). Several samples were obtained by alternately piling one layer of alumina and one layer of alumina-zirconia (this structure is hereinafter designated as A/AZ). The structures were designed in order to have an alumina layer in both outer surfaces, containing layers with a thickness ratio of about 1/1.3 (A/AZ), with a total thickness of about 3.0 mm. Due to the higher thermal expansion coefficient of the AZ composite, the *external* alumina layers undergo residual *compressive stresses* during cooling down from sintering (*ECS-laminate*). As a reference material (i.e. nominally stress free), pure monolithic alumina (MA) was also tape cast. Fig. 1 shows an optical micrograph of a cross-section of an ECS-laminate and a SEM micrograph of the interface between two layers of alumina and alumina-zirconia; it can be observed that the interface is well bonded.



**Figure 1.** a) Multi-layered structure of alumina-zirconia (AZ) layers sandwiched between alumina (A) layers, b) detail of the strong interface between adjacent layers.

**Layered composites with internal compressive layers (ICS-laminates).** The materials were manufactured at the Institute of Glass and Ceramic (ICV-CSIC), Madrid, Spain. Starting powders were submicron-sized alumina (HPA 0.5, Condea, USA) with a mean particle size ( $d_{50}$ ) of  $0.3 \mu\text{m}$ , tetragonal zirconia polycrystals (Y-TZP) with 3 mol% of  $\text{Y}_2\text{O}_3$  (TZ-3YS, Tosoh, Japan) with  $d_{50} = 0.4 \mu\text{m}$ , and pure zirconia (TZ-0, Tosoh, Japan) with  $d_{50} = 0.3 \mu\text{m}$ . Suspensions were slip cast in a plaster of Paris mould with only one filtrating surface in order to obtain 7 cm x 7 cm plates. Monoliths of  $\text{Al}_2\text{O}_3$  with 5 vol% Y-TZP (labelled as ATZ) and  $\text{Al}_2\text{O}_3$  with 30 vol% of TZ-0 (referred to as AMZ) were also prepared. Sequential slip casting [39, 40] was used to fabricate laminates composed of 5 thick layers of ATZ alternated with 4 thin layers of AMZ. The thickness of the layers was controlled from the measurement of the wall thickness after different casting times for both ATZ and AMZ suspensions [27]. Laminates with thickness ratios between 1/5 and 1/10 (AMZ/ATZ) were obtained. The expansile tetragonal  $\rightarrow$  monoclinic zirconia phase transformation within the AMZ layers yields a significant thermal strain mismatch between adjacent layers after cooling down from sintering. As a result, residual *compressive stresses* develop in the *internal* AMZ layers (*ICS-laminate*). As a reference material (i.e. nominally stress free), monolithic ATZ samples were also produced for comparison purposes. Fig. 2 shows a SEM micrograph of a cross-section of an ICS-laminate and the interface between ATZ and AMZ layers; the well bonded interface can be observed.



**Figure 2.** a) Multi-layered structure of AMZ layers sandwiched between ATZ layers, b) detail of the strong interface between adjacent layers.

### Residual stresses

Residual stresses in ceramic laminates can be due to different factors, either intrinsic, like epitaxial growth, variations of density or volume, densification or oxidation at the surface, etc.; or extrinsic such as thermal or thermoplastic strains developed during cooling. The most common approach is that associated with the differences in the coefficient of thermal expansion (CTE) between adjacent layers. It is considered that, during sintering, stresses are negligible due to the accommodation of strain mismatch by mass transport mechanisms. However, as the temperature decreases ( $\Delta T$ ), the differences in the CTE ( $\alpha_i$ ) should promote a differential strain between layers. In addition to this strain source, other strain differences should be considered as those due to phase transformations ( $\Delta\varepsilon_t$ ) [13, 41] or reactions ( $\Delta\varepsilon_r$ ) [42] inside one particular layer. Hence, the final strain difference between two given layers “a” and “b” after cooling may be expressed as

$$\Delta\varepsilon = (\alpha_a - \alpha_b)\Delta T + \Delta\varepsilon_t + \Delta\varepsilon_r. \quad (1)$$

In ceramic laminates with strong interfaces the differences in  $\alpha$  [22, 43] as well as the zirconia phase transformation [13, 44] have been commonly used to develop residual stresses. Regarding the latter, as zirconia cools down from the sintering temperature, it transforms from the tetragonal to the monoclinic phase with a volume expansion of about 4 vol%. The magnitude of the transformation can be controlled by adding small amounts of stabilizers like  $Y_2O_3$  or CaO [41] or by varying the amount of zirconia included inside the composite [23]. The zirconia expansion inside an alumina matrix has been used as a stress developer to change the fracture behaviour of ceramics [23, 32, 44, 45]. For a multilayer system composed of  $n$  layers of composition “a” and thickness  $t_a$  and  $(n-1)$  layers of composition “b” and thickness  $t_b$ , the residual stress at each layer can be estimated (within the bulk material far from the free edges [46]), based on equilibrium force considerations by [47]:

$$\sigma_a = -\frac{\Delta\varepsilon \cdot E_a'}{1 + \frac{E_a' \cdot n \cdot t_a}{E_b' \cdot (n-1) \cdot t_b}} \quad (2a) \quad ; \quad \sigma_b = \frac{\Delta\varepsilon \cdot E_b'}{1 + \frac{E_b' \cdot (n-1) \cdot t_b}{E_a' \cdot n \cdot t_a}} \quad (2b)$$

where  $E_i' = E/(1-\nu_i)$ , being  $E_i$  the Young’s modulus and  $\nu_i$  the Poisson ratio of a given layer. For the case of  $t_b \ll t_a$ , then  $\sigma_a \rightarrow 0$ , i.e. if thin layers are inserted between thick ones, the stresses inside the latter are negligible. This allows the fabrication of laminar ceramics with thin layers subjected to high internal compressive stresses, combined with thick layers exhibiting low tensile residual stresses with a minor effect on the final strength of the material [22, 32, 41, 43]. It has been shown that compressive stresses are usually beneficial for the mechanical performance of a given component as they oppose crack growth [48, 49] and/or may develop a threshold strength (high reliability) [22]. On the other hand, tensile stresses should be subtracted from the strength of the



material, and if they exceed a critical value tunnelling cracks may appear and consequently the mechanical response may be degraded [50]. For these reasons thin compressive layers are desirable, as they will create an additional reinforcement as well as diminish the residual tensile stress associated. Moreover, the thickness of the layers are referred to other observations related to the residual stresses, such as edge crack and crack bifurcation, for which a critical thickness “ $t_c$ ” has to be achieved [32, 35, 41, 43]. In order to satisfy the particular design requirements the number, thickness and composition of the layers should be controlled.

In the layered materials investigated, residual stresses develop during cooling down from sintering owed to the different thermal strain between adjacent layers. In the case of the ECS-laminates this is associated with the different thermal expansion coefficients [51], while in the case of the ICS-laminates is mainly caused by the  $t \rightarrow m$  zirconia phase transformation [52]. According to Eq. 2, the magnitude of these residual stresses can be calculated taking into account the thickness and number of layers, their elastic properties and the thermal strain mismatch. Table 1 compares the residual stresses in the layers of both ECS- and ICS-laminates for a total thickness of about 3 mm.

Layer	$E$ [GPa]	$\nu$ [-]	$CTE$ [ $10^{-6} K^{-1}$ ] ( $20^\circ - 1200^\circ C$ )	$t$ [ $\mu m$ ]	$\sigma_{res}$ [MPa]
A	391	0.24	8.64	200	-160
AZ	305	0.26	9.24	250	+130
ATZ	390	0.22	9.82	500	+120
AMZ	280	0.22	8.02	100	-620

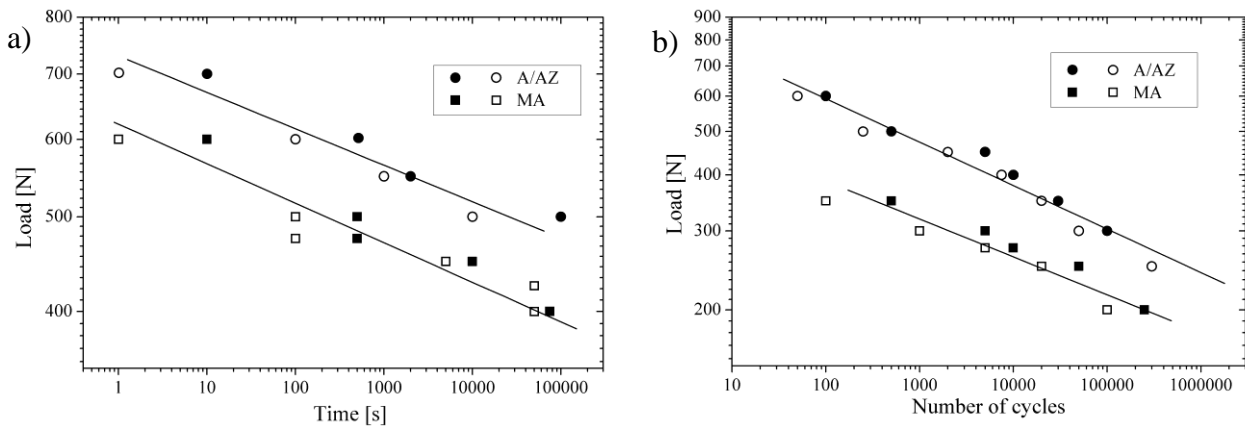
**Table 1.** Residual stress estimation in the bulk of the corresponding layers of ECS- and ICS-laminates according to Eq. 2. Physical properties have been determined elsewhere [51, 52].

## Mechanical behaviour

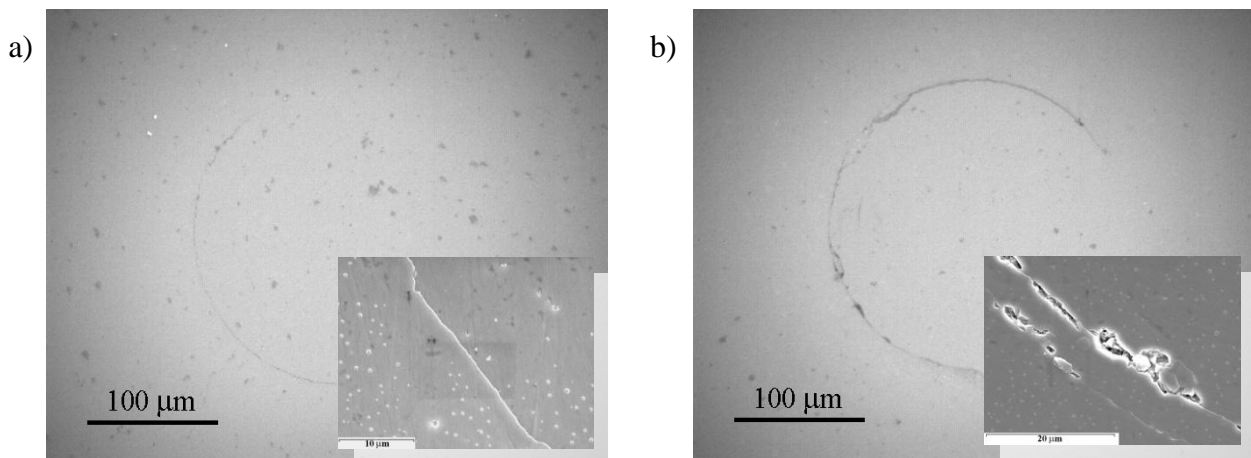
**Contact response of ECS-laminates.** One of the main applications for these materials is that related to surface properties; for this reason the response to contact loading is especially important to characterise their mechanical properties and to assist in the design of advanced ceramic composites. Hertzian indentation techniques provide a powerful tool to study such a type of loading, which is otherwise difficult to assess with the traditional mechanical testing methodologies.

Contact damage in brittle materials appears mainly as surface ring-cracks, which can develop into a characteristic cone-crack [53]. This is detrimental for the functionality of the material and can lead to the failure of the component. Moreover, tough ceramics often present another type of damage, the so-called quasi-plasticity, generated as subsurface microcracking and which is caused by inelastic deformation. These mechanisms of damage are analysed below.

**Surface ring-crack appearance and cone crack growth.** Static and cyclic loading were applied on the material surface (subjected to compressive residual stress) for a given time or number of cycles [34, 54]. The damage criterion was the appearance of a ring-crack on the surface, observed after removal of the testing sample from the test jig. Results in terms of crack appearance range are presented in Fig. 3, and compared with the monolithic alumina used as reference material. It can be seen that the laminated materials present a higher strength under both types of loading, whose amount is approximately the value of the surface residual stress. The degradation slopes are comparable. Therefore, the micromechanisms of damage in all the materials are equivalent and the compressive residual stress decreases the apparent stress intensity factor. It can also be inferred that all materials under cyclic loading (Fig. 3) present damage much earlier than under static loading. If the same degradation mechanisms were operating under cyclic and static loading, the experimental results of both cyclic and static loading would be equal after conversion of the number of cycles into effective time. Therefore a real cyclic fatigue effect exists; the degradation observed is not only due to the stress corrosion cracking. Such a fatigue effect can be clearly seen in Fig. 4, where the Hertzian crack produced under subcritical static loading (Fig. 4a) and cyclical loading (Fig. 4b) are shown. More damage is observed in Fig. 4b, associated with the fretting during cyclic loading.



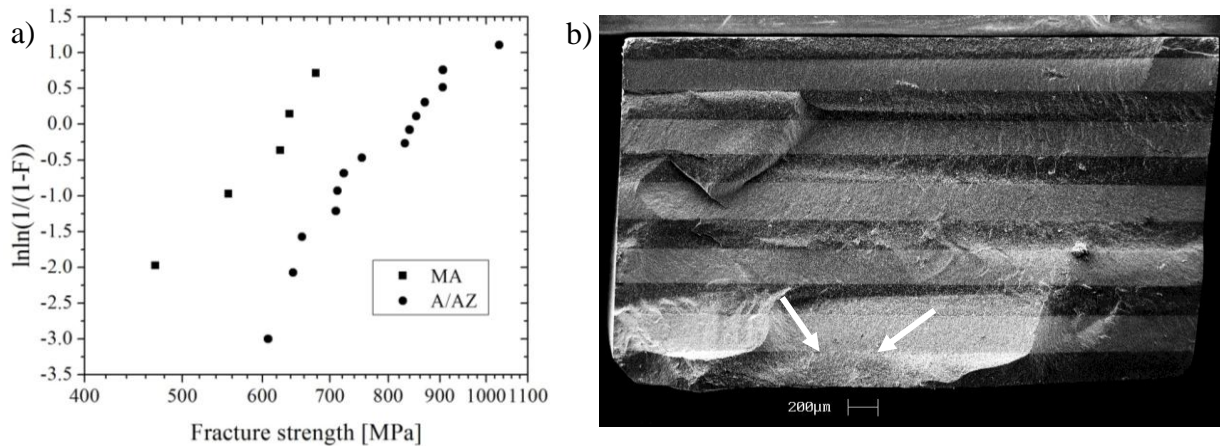
**Figure 3.** Indentation load vs. number of cycles or time under a) static and b) cyclic fatigue tests. Empty and filled points indicate no apparent damage and well-developed ring crack, respectively.



**Figure 4.** Optical micrographs of typical ring cracks of A/AZ material produced under: (a) static and (b) cyclic loading. Regions of grain bridging are visible at the ring-crack in (b).

The cone-crack generated by Hertzian indentation on brittle materials is an effective representation of damage produced in service by blunt loading. The geometrical characteristics of such cone crack can affect the strength and reliability of the component, since such crack can behave as the critical defect from which fracture starts. On the other hand, the geometry of such cone-cracks (i.e. length and angle) has been found to be influenced by the presence of residual stresses. The crack propagation on ECS-laminates has been studied experimentally and by means of finite element simulations elsewhere [55]. It has been stated that in presence of compressive stress, the cone angle is reduced; therefore the strength degradation under remote loading is less.

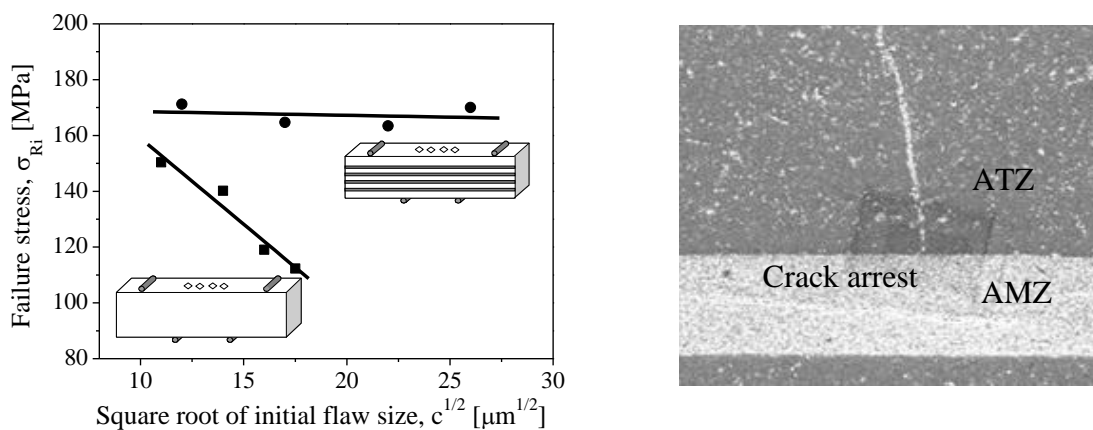
**Contact strength.** The ultimate consequence of contact loading is the failure of the material. Such a test has been performed by means of compression tests using opposed rollers [56, 57]. On monolithic materials, fracture starts from the high tensile stress at the surface, which can be expressed as a function of the equivalent strength as  $\sigma_{\max} = 0.49 \cdot \sigma_{\text{eq}} = 0.49 (F/S)$ , where  $F$  is the applied load and  $S$  the specimen section [53]. As can be inferred from Fig. 5a, the strength of the laminate is increased with respect to the monolith strength by a quantity equivalent to the compressive residual stresses [52]. A typical fracture surface of an ECS-laminate is shown in Fig. 5b, where the fracture starts from the surface. Nevertheless, FEM studies have revealed that, due to the tensile residual stresses associated with the contact loading, the maximal stress during application of load may be located in the internal tensile layers (near to the surface), leading to failure of the structure from internal flaws [58].



**Figure 5.** a) Contact strength of monolithic alumina (MA) and ECS-laminates (A/AZ) and b) typical fractography of a tested ECS-laminate, where the fracture starts from the surface.

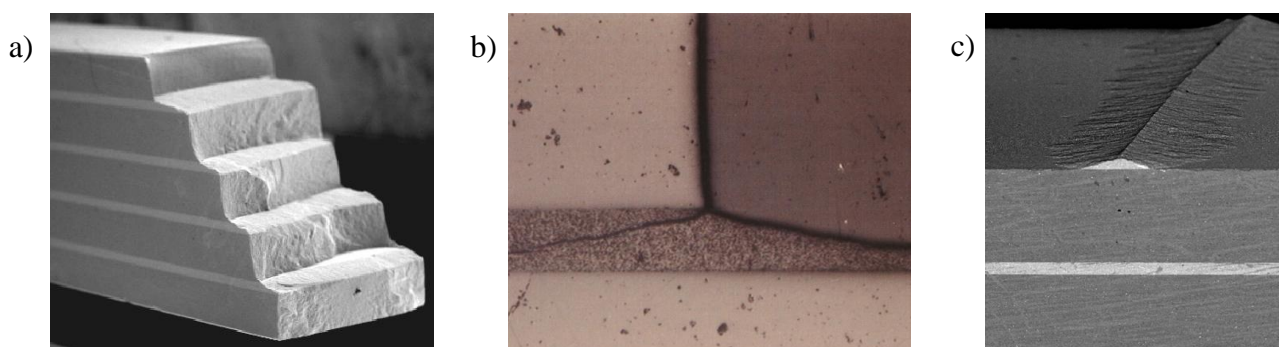
### Fracture response of ICS-laminates

**Monotonic loading.** In order to evaluate the fracture behaviour in the laminates investigated, indentation-strength tests have been performed. ATZ monoliths have been also tested for comparative purposes. The failure stress ( $\sigma_{Ri}$ ) resulting from four-point bending tests was calculated using the elastic beam theory [59], taking into account (for the layered structures) the different elastic properties of the corresponding layers [60]. In all cases, fractographic analysis were made to confirm failure initiation from the indentation sites and not from either interface defects or other surface flaws [31]. As a result, an almost constant level of failure stress (so-called “*threshold stress*”) was found for the ICS-laminates, regardless of the indentation flaw size, which is associated with the internal compressive layers arresting the initial crack propagation (Fig. 6).



**Figure 6.** Plot of measured 4-point bending failure stress vs. square root of crack length in ATZ monoliths (■) and laminates (●) specimens containing several groups of Vickers indentations at the surface. A threshold strength is found for the ICS-laminates based on the crack arrest capability provided by the internal compressive layers.

The main fracture features of these ICS-laminates can be observed in Fig. 7. A typical step-wise fracture can be observed, which is associated with the role of the compressive layers in hindering and/or deviating the initial straight crack path. Crack bifurcation causes energy dissipation, which increases the fracture energy of the material up to five times that of alumina-based monoliths [35]. As can be inferred from the referred figure, this is a 3-dimensional feature which leads to the so called mountain-like fracture, particular of this kind of laminates.

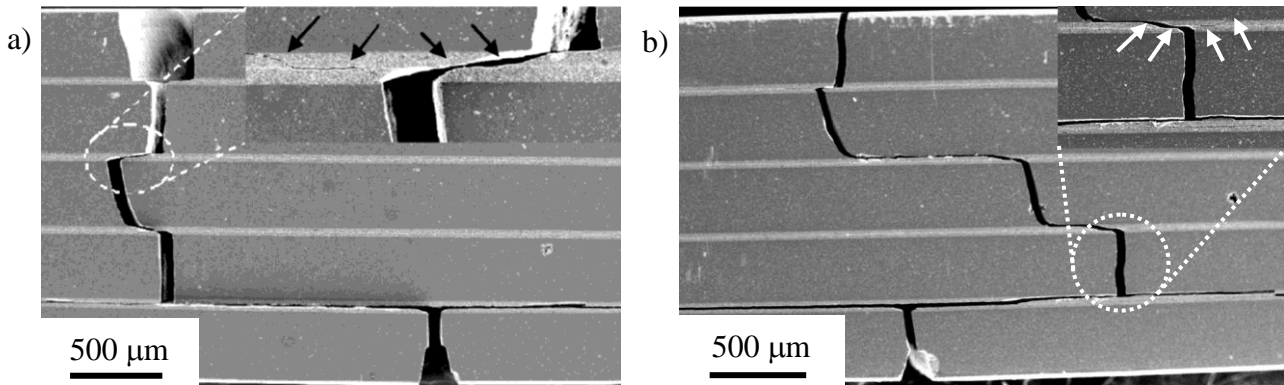


**Figure 7.** Fracture features of ICS-laminates: a) Step-like fracture associated with the compressive layers, which hinder the straight crack propagation, b) Crack bifurcation as the crack enters the compressive layer and c) Typical mountain-like feature showing the 3-dimensional crack bifurcation mechanism.

**Static and cyclic loading.** The mechanical behaviour of layered ceramics has resulted in significant progress on the understanding of the relationships between layer architecture (i.e. number, thickness and composition of the layers as well as interface strength) and critical design requirements such as toughness and strength reliability. However, fracture toughness improvements through material development often does not directly translate into similar beneficial effects on the mechanical response of a given material under service conditions different from monotonic loading (e.g. Refs. [61, 62]). This is particularly true for advanced ceramics where premature failure under sustained or variable stresses, i.e. under static or cyclic fatigue, is associated with the susceptibility of operative toughening mechanisms to be degraded under such loading conditions [63]. Taking into consideration that components made of multilayered ceramics may be employed under constant and cyclic loading, knowledge about their fatigue behaviour is clearly required for efficient usage of them as structural materials. Previous research on the fatigue response of layered ceramics is quite scarce. In this regard, the high cycle fatigue resistance of mullite-alumina composites has been reported to be improved by inducing a functionally graded residual stress distribution through layered design [64]. For the case of alumina-zirconia layered ceramics, microstructural design based on multilayered architectures where internal layers are the ones exhibiting residual compressive stresses has been as effective under monotonic as it is under cyclic loading. Within this context, although alumina-zirconia multilayered ceramics do exhibit subcritical crack growth under both sustained and cyclic loading, their fatigue behavior is relatively superior to that evidenced by other toughened ceramics in terms of lower static and cyclic fatigue sensitivity, as well as negligible mechanical fatigue effects [65].

Furthermore, the fatigue crack growth threshold of the ICS-laminates is found to be about 70 % of the corresponding apparent fracture toughness, independent of the loading condition considered, i.e. sustained or cyclic loading. Such an outstanding fatigue response, relative to other alumina- and zirconia-based ceramics, is ascribed to the fact that toughening of laminates at the threshold stage is given by compressive residual stresses “discretely” existing at the thin internal layers and directly acting at the crack tip. These layered ceramics exhibit low susceptibility to mechanical fatigue not only at the early stage of crack extension beyond the first interface but also during the subsequent stable crack growth. Under these conditions, crack bifurcation and interface delamination emerge as complementary toughening mechanisms whose effectiveness is unaffected by the application of constant or variable stresses (Fig. 8).



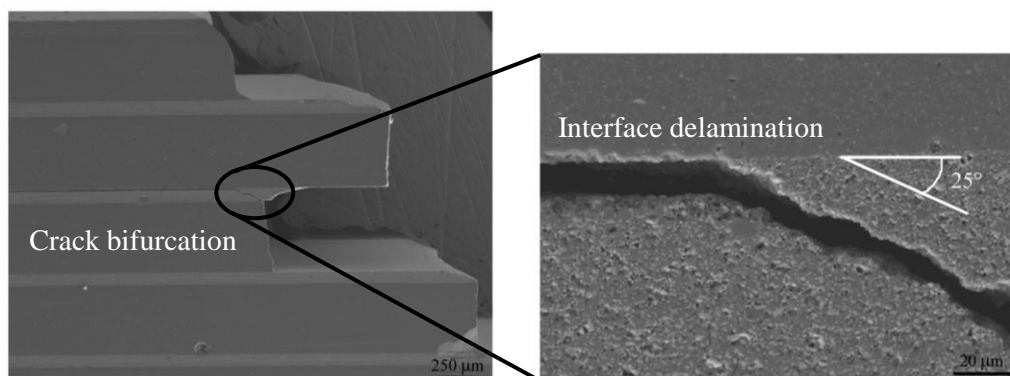


**Figure 8.** SEM micrographs of the step-wise fracture of ICS-laminates under a) static and b) cyclic loading conditions. The crack (at the bottom) initiates at the notch tip. Crack bifurcation and interface delamination occur as toughening mechanisms during fracture.

### Influence of temperature

**Thermo-mechanical loading.** The temperature effect can be of extreme importance for the performance of laminates designed on the basis of residual stresses when the temperature of use approaches that of stress relaxation. In this sense, the high temperature mechanical response has been characterised and compared to that of monolithic materials, with the same composition as the layers. Indentation-strength tests have been performed under four-point bending at different temperatures to analyse the crack growth resistance of the layered material as function of the residual stresses associated with the testing temperature.

Experimental findings showed that improvement in mechanical properties at high temperatures in comparison to the alumina-based reference material is essentially related to the maintenance of the compressive stresses developed during sintering in the internal thin layers of the laminate structure, acting as an effective barrier to crack propagation [66]. In such cases, a steep R-curve behaviour is found, leading to a higher reliability in terms of structural design compared to the brittle behaviour of monolithic ceramics. Additionally, step-wise fracture can be observed under these conditions, owed to crack bifurcation mechanisms taking place in the thin compressive layers. In some cases, i.e. 800 °C during heating, bifurcation within the first compressive layer is followed by interface delamination in the next interface due to the change in the elastic properties of the layers with the temperature as well as the impinging angle of the bifurcated crack (Fig. 9) [67].

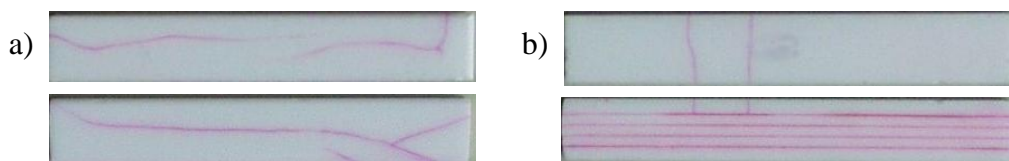


**Figure 9.** SEM micrograph showing interface delamination at the AMZ/ATZ interfaces after bifurcation occurred in ICS-laminates tested at 800 °C on heating. In this case, the bifurcation angle along with the change in elastic properties at the testing temperature helps the crack run along the interface [67].

Special attention must be paid when the material has been subjected to temperatures where the m→t zirconia phase transformation has taken place, i.e. between 1150 °C and 725 °C. In these cases, the

residual stress state reverses yielding a slightly higher failure stress due to the compression in the outer layer, although brittle fracture follows as for the reference alumina-based material. This multilayered design based on the flaw tolerant capability provided by the compressive residual stresses in the internal layers might be used as a substitute for alumina-based monolithic ceramics for high temperature applications, being always aware of the temperature hysteretic conditions under which the material may operate.

**Thermal shock.** It is well established that changing the temperature in a specimen causes thermal strains, and that if these strains are constrained, thermal stresses occur. Since most brittle ceramic materials are susceptible to catastrophic failure under conditions of high heat transfer and/or rapid environmental temperature variations, the thermal shock resistance is one of the most important thermal properties of structural ceramics. In the last two decades, remarkable advances have been achieved to increase their thermal shock resistance, by either limiting the extension of flaws once cracking has initiated or making it more difficult for defects within a body to start growing. Within this framework, improving thermal shock resistance by preventing the onset of crack growth is a difficult task, especially when the thermal shock is severe, as the crack driving forces can be many orders of magnitude greater than the resistance to fracture of conventional ceramic materials. Therefore, it is more usual to try to limit the extent of crack growth into the specimen. This requires either that as much energy as possible is dissipated in growing the crack [68], or that the crack driving force available to each individual crack is reduced [69]. The latter may be achieved by increasing the initial size of the defects in the material, so that cracking begins as soon as possible after the thermal shock has taken place, minimizing the build-up of elastic energy in the body. Although such methods can be very effective, they often lead to degradation of properties such as corrosion resistance, which can be of primary importance where materials are required for handling liquid metals or gases. An alternative approach is the use of layered ceramic structures containing crack-deflecting interfaces, showing enhanced thermal shock resistance in both laboratory and component tests [70]. A particular case is that of zirconia-containing laminates, where the thermal shock response is positively influenced by the presence of compressive layers, in terms of crack deflecting interlayers [71]. This consideration has been extended to ICS-laminates with strong interfaces, aiming to achieve the crack arrest feature present at room temperature. In doing so, thermal shock tests have been conducted both in ATZ monoliths and in ICS-laminates at several quenching temperatures in water to investigate the crack initiation site for both materials [72]. An example is shown in Fig. 10 for a thermal shock test at  $\Delta T = 225^\circ\text{C}$ . In the case of the ATZ material the cracks arose preferentially at the centre of the specimens' surface. On the other hand, in the laminate bars, despite the biaxial residual stresses generated during sintering, the cracks initiated only at the long edges of the specimens. Once initiated, these cracks became through-the-thickness cracks and arrested at the interface of the first thin compressive layer, analogue to the case of monotonic loading reported above. This suggests the presence of a threshold temperature difference,  $\Delta T_{th}$ , below which thermal shock cracks are limited to the extension of the outermost ATZ layer thickness while the rest of the structure remains intact after the thermal loading. This should be definitely foreseen as a reliable design for thermal shock applications.



**Figure 10.** Top and side view of thermal shock cracks after quenching at  $225^\circ\text{C}$  for a) ATZ monolithic and b) ICS-laminate. In the ICS-laminate, cracks propagate up to the compressive layer where they get arrested.

## Summary

The fracture response of two kinds of layered ceramics designed with external or internal compressive residual stresses has been investigated from the viewpoint of their potential application. Laminates with compressive stresses in the external layers have shown an improved material resistance against contact damage mechanisms such as ring cracking and cone crack propagation under both monotonic and cyclic loading. The response to remote loading is conditioned by the cone crack geometry as well as by other factors deriving from the laminated structure, such as the presence of residual stress itself and the load redistribution due to the elastic mismatch between layers. Similarly, the contact strength is improved in the composite materials as a consequence of the residual stresses. Nevertheless, the risk of high stresses in the lower tensile layers has been highlighted for both types of loading, which might affect the reliability of such design. In this regard, laminates with compressive stresses in the internal layers offer an outstanding potential in terms of mechanical reliability (*flaw tolerance*), yielding a minimum failure stress or “*threshold strength*”, under both monotonic and cyclic loading as well as under thermal shock conditions, regardless of the critical processing or machining flaw size. The energy release mechanisms acting during fracture, such as crack deflection or crack bifurcation, associated with the high residual compressive stress level in the internal layers, increase significantly the fracture energy of the material, compared to the brittle fracture of monolithic alumina-based ceramics. An optimal design should comprise a maximum crack growth resistance along with a relatively high threshold strength.

While layered structures with compressive stresses at the surface provide a better response against contact damage, a more reliable design in terms of strength and crack growth resistance is achieved with layered architectures with internal compressive stresses. The use of layered structures for automotive or biomedical applications as substitutes for alumina-based ceramics should be seriously considered, always bearing in mind the loading scenario in service.

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**Fractography of Advanced Ceramics III**

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