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Loading Configuration Effects on the Strength Reliability of Alumina-Zirconia Multilayered Ceramics

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Abstract

The loading configuration effects on the strength reliability of alumina/zirconia multilayered systems, designed with internal compressive stresses, have been investigated. The existence of a threshold strength behaviour, as previously assessed under longitudinal flexure, has been evaluated by indentation-strength tests under transverse flexure. Additionally, the influence of flaw interactions on the strength reliability under both loading configurations has also been studied. Experimental findings show a higher strength level under transverse flexure, as far as the failure-controlling flaw is located between two compressive layers. It is found however that multiple artificial and natural flaws may interact affecting the strength of the specimen under transverse loading. As a consequence, although higher strength values result from this configuration, a more reliable design is rather attained when the loading axis is applied normal to the layer plane, a loading mode where similar effects are negligible.

Keywords: A. Layered structures, B. Strength, C. Damage tolerance, C. Transverse cracking, Loading configuration.

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1. Introduction

Ceramic materials exhibit a relatively wide strength distribution owed to the quite variable characteristics of the flaws induced during their processing and/or machining, i.e. nature, geometry and size, together with their relative orientation with respect to the applied loads. This broad range of strength values and their inherent brittleness have limited the use of ceramics in structural applications. However, as a direct consequence of remarkable progress in terms of microstructural design and advanced processing [1-3], toughness and reliability of structural ceramics have been increasingly enhanced by recourse to crack shielding resulting from microstructure-related mechanisms [4-11]. Particular attention has been paid to fibre and layered reinforced ceramics, where the better mechanical performance is associated with not only the second phase or layer addition, but also the arrangement of the fibres or the layer assemblage with respect to the loading axis, as well as the sliding resistance of the fibre-matrix interface or the interlayer fracture energy, respectively [7, 12, 13]. According to these studies, when the crack front is oriented parallel to the aligned fibres, the crack propagates catastrophically through the composite at the initial cracking stress, i.e. there is not any crack shielding from the fibres arranged normal to the loading direction. On the other hand, when the fibres are oriented perpendicular to the crack front, cracks originated at the outer ceramic layer get arrested at the ceramic-fibre interfaces, providing a higher resistance to crack propagation.

As an extension of this lamina r 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 [11, 14-17]. This approach has 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 [11, 18, 19], 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 [8, 15, 20]. 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 [8, 14, 15, 21, 22].

In addition to the architectural design, loading conditions (Fig. 1) may strongly affect the mechanical response of a layered structure [14]. Although it is well-documented that multilayered ceramics exhibit an enhanced fracture behaviour, independent of the specific loading configuration under consideration, i.e. with loading axis either parallel (transverse flexure) [11] or perpendicular (longitudinal flexure) [17, 23, 24] to the layer plane, studies where the mechanical strength of such multilayered systems are directly compared as a function of loading orientation are quite scarce. In this regard, a recent work from R.J. Moon *et al.* [25] on alumina-zirconia multilayered composites has shown that relative layer orientation with respect to the crack-tip front exerts a pronounced influence on the crack growth resistance behaviour of the material, thus pointing out the need for further investigation from this perspective if these materials are to be properly implemented in structural applications.

Furthermore, although a single crack within a thick tensile layer can be arrested by thin compressive layers, there is a possibility that other cracks and/or flaws may coexist close to each other, either in the same or in adjacent layers, varying in certain cases the critical failure scenario for the material. Within this context, the effective stress intensity

factor for interacting cracks has been the topic of research of several studies following both analytical [26-28] and experimental [29] approaches. In general, it has been found that two closely spaced cracks can interact to increase the stresses at the corresponding crack tips, thus affecting the fracture response of the component in the zone where the cracks are located.

In a previous work, the authors have shown that a single crack, located at the outer tensile layer of an alumina-zirconia laminated system can be arrested by the first internal thin compressive layer when extending under longitudinal flexure [24]. As a result of such crack – layer architecture interaction, these laminates exhibit an almost constant failure stress regardless of the size of the pre-existing cracks (artificially introduced by recourse to indentation techniques), i.e. a threshold stress below which the probability of failure is nearly zero. As it is shown in figure 4 from Ref. [24], this is completely different from the failure behaviour experienced by monolithic alumina-based brittle ceramics, where the flexural strength continuously decreases with increasing indentation load, i.e. flaw size. Because this interesting finding (i.e. the existence of a threshold stress) has been discerned only under longitudinal flexure (Fig. 1a), an immediate query is raised whether such a mechanical response would also apply under conditions where the load axis is set parallel to the layers, i.e. in transverse flexure (Fig. 1b).

The purpose of this work is to investigate the loading configuration effects on the strength reliability of an optimally designed alumina/zirconia multilayered system [22]. In doing so, the existence of a threshold strength behaviour under conditions of transverse flexure is assessed. Additionally, the influence of flaw interactions on the strength reliability under both loading configurations is also addressed.

2. Experimental Procedure

2.1 Material of study

Starting powders were submicron-sized alumina (HPA 0.5, Condea, USA) with a mean particle size (d_{50}) of 0.3 μm , tetragonal zirconia polycrystals (Y-TZP) with 3 mol% of Y_2O_3 (TZ-3YS, Tosoh, Japan) with $d_{50} = 0.4 \mu\text{m}$, and pure zirconia (TZ-0, Tosoh, Japan) with $d_{50} = 0.3 \mu\text{m}$. Slurries were prepared to a solid loading of 36.5 vol% by mixing starting powders with DI water, containing a 0.8wt% of a commercial acrylic based polyelectrolyte (Duramax D-3021, Rohm&Haas, USA) used for stabilisation. Sequential slip casting [30, 31] 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. A slurry composed of Al_2O_3 /5 vol% Y_2O_3 -stabilised ZrO_2 (t- ZrO_2), referred to as ATZ, was used to form the thicker layers. The t- ZrO_2 was employed to control the grain size of the Al_2O_3 during densification. In order to form the thin layers a slurry containing Al_2O_3 /30 vol% ZrO_2 (m- ZrO_2), named as AMZ, was utilised. Details of the colloidal processing procedure may be found elsewhere [32]. The laminate plates, sintered at 1550 $^\circ\text{C}$ for 2 hours in air, consist of a periodic, alternating stack of five thick ATZ layers, i.e. $540 \pm 10 \mu\text{m}$, and four thin AMZ layers, i.e. $95 \pm 5 \mu\text{m}$. Several bar-shaped specimens (3.8 mm x 3.2 mm x 25 mm) were cut from the plates and the top and lateral surfaces of each sample polished to a 3 μm finish with a diamond abrasive. A homogeneous layer thickness was obtained for both ATZ and AMZ layers, resulting in a symmetrical multilayered architecture. As a result of the non-stabilised zirconia content in the thin AMZ layers, a significant thermal mismatch between layers was generated owed to the t \rightarrow m zirconia phase transformation occurring when cooling down during sintering. This martensitic transformation is accompanied by

an increase in volume which modifies the cooling shrinkage behaviour of the laminate, developing in this case compressive stresses inside the thin layers and tensile stresses in the thicker ones [18]. The magnitude of these residual stresses has been calculated for this architecture in a previous work [24], resulting in -690 MPa for the thin AMZ layers and 100 MPa for the ATZ ones. The measured thermal mismatch between adjacent layers resulted in $\Delta\epsilon = 0.00212$, and the corresponding Young's modulus of the ATZ and AMZ compacts were 390 GPa and 280 GPa, respectively [32].

2.2 Indentation-strength tests

In order to evaluate the possible threshold stress under transverse flexure, pre-cracks were introduced in the central thick layer of the laminate using a Vickers indenter, as illustrated in Figure 2a. All pre-cracks were induced using a Vickers indenter and applied loads within a range between 30 N and 200 N. Additionally, attempting to assess crack interaction effects under both loading orientations, indentations of 100 N were placed with an offset separation x ranging from 0 mm (coplanar-cracks) to 3 mm, either at the internal ATZ layers (Fig. 2a) or at the top surface (Fig. 2b). The relative distance between cracks for both cases was measured by means of an optical microscope.

Four-point bending tests were performed under both configurations with a fully articulated test jig with outer and inner spans of 20 mm and 10 mm respectively. Tests were carried out under load control using a servo-hydraulic testing machine (Model 8511, Instron, Ltd.) with a load cell of 10 KN, at a testing rate of 100 N/s. The failure stress for the indented specimens, σ_{Ri} , was determined through the following expression:

$$s_{Ri} = \frac{3F(s_o - s_i)}{2Bh^2} \quad (1)$$

where F is the applied load at fracture, s_o and s_i are the outer and inner spans respectively, B is the specimen width and h is the specimen height. Fractured surfaces were inspected by scanning electron microscopy (JEOL JMS 6400, Germany) to determine the source of failure for each tested specimen.

3. Results and discussion

3.1 Strength on specimens containing a single crack

A constant strength value was encountered for the case of laminates indented at the top surface and loaded perpendicular to the layer plane (longitudinal flexure), as found in a previous work [24]. In these cases, it was observed how all the initial indentation cracks propagated up to the first compressive layer, where they got arrested before catastrophic failure occurred, yielding an effective “threshold stress” level (170 ± 8 MPa) below which the probability of failure is nearly zero. For the laminates indented at the central thick layer and subjected to transverse flexural loading, cracks emanating from the indentation extended towards the two thin compressive layers and got arrested (Fig. 3), before further propagating through the rest of the laminate. As a result, a constant strength of 401 ± 10 MPa was obtained, regardless of the size of the initial indentation crack, as it is presented in Figure 4. Nevertheless, in some cases, and although artificial cracks had been introduced in the central ATZ layer of the tested specimens, fracture was found to initiate from natural flaws located in the outer most ATZ layer (Fig. 5), yielding failure stress levels of 265 ± 25 MPa. From this point of view, a threshold stress under transverse configuration may be defined as far as the failure-controlling

flaw is located between two compressive layers. In these cases, the initial growth experienced by such defect upon loading is inhibited by two thin compressive AMZ layers, which act as a physical barrier to crack propagation, as it was shown in Figure 3. In order to rationalize this behaviour, a linear elastic fracture mechanics analysis, as carried out by Rao *et al* [16] on an alumina/alumina-mullite system, was implemented in this work. Following such ideas, for the case of a well-developed crack of size $2a$ extended sidewise into two surrounding compressive layers, the maximum stress needed to cause the crack to break through the compressive layers occurs as $2a = 2t_2 + t_1$ (being t_1 and t_2 the thickness of the ATZ and AMZ layers respectively), and the subsequent catastrophic failure takes place when the stress intensity factor at the crack tip K overcomes the toughness of the thin compressive layers. Hence, the largest strength (s_{max}) for further crack propagation is associated with the toughness (K_{Ic}) and the magnitude of the compressive stresses (s_c) of such thin layers according to [16, 33]:

$$s_{max} = \frac{K_{Ic}}{\sqrt{p \frac{t_1}{2} \left(1 + \frac{2t_2}{t_1}\right)}} + s_c \left[1 - \left(1 + \frac{t_2}{t_1}\right) \frac{2}{p} \sin^{-1} \left(\frac{1}{1 + \frac{2t_2}{t_1}} \right) \right] \quad (2)$$

Substituting into Eq. (2) the value of the residual compressive stress s_c , the average values of the thick (t_1) and thin (t_2) layers, and the value of K_{Ic} for the AMZ arrester layers, i.e. $2.6 \pm 0.1 \text{ MPam}^{1/2}$ (evaluated in a previous work [24]), the maximum predicted stress s_{max} results in $336 \pm 9 \text{ MPa}$. This stress level is lower than the measured strength of $401 \pm 10 \text{ MPa}$. Such discrepancy comes from the fact that Eq. (2) is derived for arrested cracks that fully extend through the compressive layers in a straight path, whereas the crack is experimentally observed to bifurcate (Fig. 3). Such bifurcation

process implies energy consumption associated with the deviation of the crack from mode I of fracture, which also requires a higher stress level for further crack propagation, as it was demonstrated in a previous work under longitudinal flexure [22].

We caution the reader about the fact that different elastic modulus between the two layers may also affect the absolute threshold stress values predicted by Eq. (2), as assessed by finite element studies taking into account the elastic anisotropy of the layered architecture [34, 35]. However, since the elastic ratio between tensile and compressive layers is not very significant in this case, i.e. $E_1/E_2 \sim 1.4$, the maximum stress value considering the material anisotropy should not differ much from that predicted by Eq. (2).

For the particular case of transverse flexure configuration when the fracture initiating defect is embedded within the outer ATZ layer, failure stress levels are much lower than the corresponding threshold stress predicted by Eq. (2). This fact can be explained by considering that under this layered architectural design only one compressive layer may act as a crack arrester for these propagating critical defects. Hence, although larger artificial flaws are introduced in the central layer, the stress intensity factor associated with the outer natural flaws governs the material fracture, yielding a flaw-size dependence mechanical strength. In order to avoid such crack location effect under such a configuration, a similar stress state in both inner and outer thick layers must be guaranteed, a situation that may be achieved if the laminate is designed with outer compressive layers, as if the composite were to be used in contact-related applications. This design concept may be suggested as an option for preserving relative constant strength values; and thus, for enhancing reliability under transverse loading conditions.

3.2 Strength on specimens containing several defects

Failure stress values were determined for multi-cracked specimens following a similar testing procedure as for the single -cracked samples. All the experimental results under both testing configurations were expressed in terms of stress using Eq. (1) and are shown, in a combined format, in Figure 6.

Under longitudinal flexure testing it was observed that, although the strength of laminates was slightly affected by crack interactions if they were relatively close to each other, i.e. 0.5 ± 0.2 mm apart (Fig. 6a), a constant failure stress value similar to the case of a single-crack was achieved regardless of the crack offset separation. As a matter of fact, for the particular case of co-planar cracks, i.e. with zero offset separation distance, they were found to coalesce at a certain load and behave as one single crack, as it can be inferred from Figure 7. In such case, failure stress fell within the scatter of the threshold stress value encountered for single-cracked specimens. Regarding crack growth mechanisms as related to the laminated architecture, a fracture surface exhibiting a similar step-wise profile as for the case of single-cracked specimens was also appreciated (Fig. 7).

Concerning transverse flexure testing, although a trend qualitatively similar to the one described above on the influence of flaw interaction was also discerned, in agreement with the results reported by H. Moon *et al.* [29] for an alumina-mullite laminar composite, two particular observations should be highlighted. First, relative differences in strength are somehow more pronounced under loading parallel to the layer plane than under longitudinal flexure testing (maximum decrease of about 15% in the former as compared to values close to 9% in the latter). Second, nearby flaws (located at different ATZ layers) do have an effect on the strength for multi-cracked specimens tested under transverse flexure (Fig. 6b). Attempting to understand the

described different crack interaction effects as a function of loading mode, an extensive and detailed inspection of the fracture surfaces of the broken indented specimens was conducted. Different from the case of the specimens tested under longitudinal flexure, interaction of the indentation cracks with intrinsic defects, such as pores or agglomerates, was often evidenced at fracture when the offset separation distance resulted to be less than ~ 0.6 mm. In general, interacting natural flaws introduced during the processing step were encountered within one of the adjacent ATZ layers near the indentation crack; and in all the cases, both the intrinsic defect and the indentation crack experienced an initial growth up to the corresponding ATZ/AMZ interfaces where they got arrested (as evidenced in Figure 8) owed to the high compressive stresses in the thin AMZ layers. Then, by increasing the applied load, the critical stress intensity factor of the AMZ compressive layer was finally overcome, and both defects coalesced and propagated unstably up to catastrophic failure at a stress level below the one resulted for the single-crack specimen, as reported in Figure 6b. Hence, it is suggested that the relatively higher prominence of multiple cracking effects on failure stress under transverse flexure is directly related to the effective capability of the existing defects to interact with each other at regions close to the specimen surface, subjected to nominally maximum tensile stresses. Such interacting capability is expected to be enhanced under loading parallel to the layer plane as a direct consequence of the operation of the natural energy absorption mechanisms for these laminated ceramics, i.e. crack bifurcation and deflection within the thin AMZ layers. This is not the case under loading perpendicular to the layer plane, because similar crack interactions could only occur in the interior of the specimen, i.e. at locations where the levels of the nominally applied stress are effectively lower than at the specimen surface.

4. Conclusions

Loading configuration effects on the strength of an alumina/zirconia layered system designed with internal compressive residual stresses have been investigated. The main results of the present study can be summarised as follows:

The existence of a threshold stress behaviour for the studied multilayer system, as previously assessed under longitudinal flexure (170 ± 8 MPa), has been also found under transverse flexure as far as the failure-controlling flaw is located between two compressive layers, leading to higher strength values (401 ± 10 MPa). However, for the particular case when the fracture initiating defect is only constrained by one compressive layer, i.e. flaw embedded within the outer thick layer, failure stress levels are lower than the expected threshold stress, yielding a flaw-size dependence of the mechanical strength. In order to avoid such crack location effect under such a configuration, a similar stress state in both inner and outer thick layers must be guaranteed, a situation that may be achieved if the laminate is designed with outer compressive thin layers, as for contact-related applications.

Flaw interaction effects on the strength reliability of such multilayered architecture have been found to be markedly dependent on the loading configuration under consideration. For the transverse flexure case, although higher threshold stress levels may be achieved, there exists a higher strength scatter which is associated with the interaction of cracks and natural defects embedded in adjacent layers. By the contrary, flaw interaction effects are found to be neglected under longitudinal flexure; and thus, from this perspective such a factor should not affect the high reliability obtained under this loading configuration.

As a consequence, the choice of a particular laminate design system for structural applications should comprise a prior knowledge of the loading conditions to which the

material is to be subjected during service, together with a fine control of the processing procedure in order to reduce the density of potentially -interactive flaws within the bulk component.

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List of figures

Figure 1. Scheme of a testing configuration where the loading axis is applied a) perpendicular (longitudinal flexure) and b) parallel (transverse flexure) to the layer plane.

Figure 2. Testing configurations imposed on single and multiple indented specimens (x being the offset separation between cracks) corresponding to applied loading axis a) parallel and b) normal to the layer plane.

Figure 3. Optical micrograph illustrating the crack arrest at the ATZ/AMZ interfaces of a layered specimen under loading parallel to the layer plane. Crack bifurcation within the compressive layers can be also observed during the fracture process.

Figure 4. Plot of the measured 4-point bending failure stress vs. indentation load in the laminate tested under transverse flexure. Specimens that failed through indentations located in the central ATZ layer show an almost constant value, whereas the ones failing owed to the natural flaws present a lower value with a higher scatter.

Figure 5. SEM micrograph showing the source of failure (*natural flaw*) encountered at the outer most ATZ layer “f”, despite the indentation crack “i” located in the central layer. A detail of the failure-controlling natural flaw is also presented.

Figure 6. Failure stress values calculated under four-point bending with loading axis a) perpendicular and b) parallel to the layer plane. In the former, flaw interaction effects are almost negligible and thus, from this perspective, such a factor should not affect the high strength reliability assessed under this loading configuration. In the later, natural flaws are observed to interact with indentation cracks under certain offset separation distances, reducing the strength levels encountered for single-cracked specimens.

Figure 7. Step-wise fracture of a laminate with co-planar indentations located at the outer ATZ layer. A detail of the penny-like indentation cracks is also presented.

Figure 8. Fracture site of two specimens where the indentation crack “I” interacts with a natural flaw “F”, located in the adjacent layer. As a consequence, the corresponding failure stress level is lower than that of specimens with a single crack in the central layer.

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Fig 1:

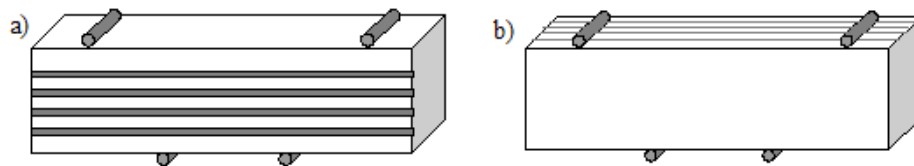


Fig 2:

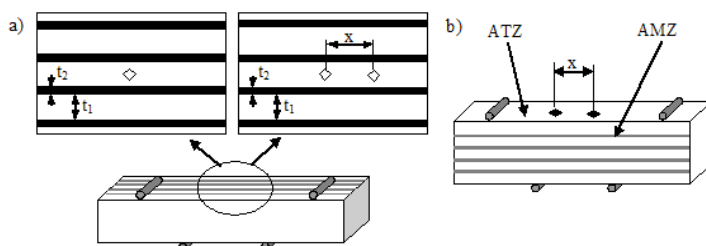


Fig 3:

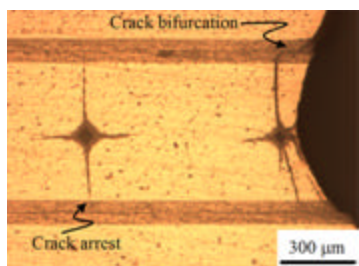


Fig 4:

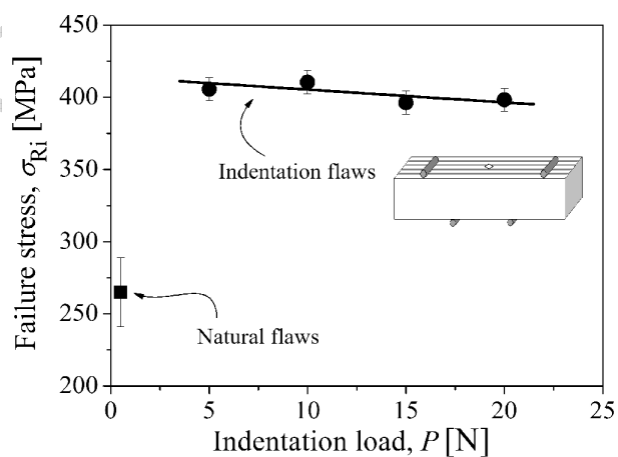


Fig 5:

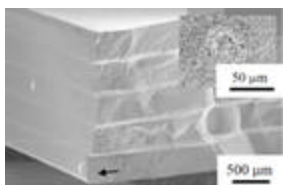


Fig 6:

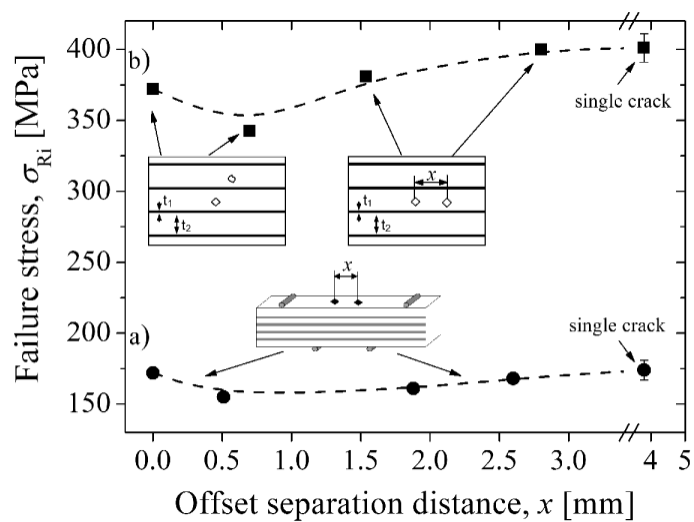


Fig 7:

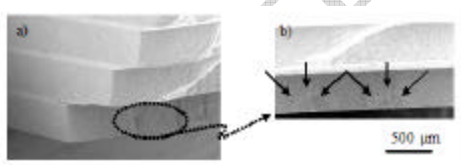


Fig 8:

