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Fracture behaviour of alumina and zirconia thin layered laminate

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Abstract

Laminates with strong bonds between thin layers were examined in this work to explore the influence of developed internal stresses on the fracture behaviour. A set of laminates having different level of internal stresses were prepared. Alumina and zirconia were the model materials for evenly alternating layers. The electrophoretic deposition technique was used for manufacturing of the laminates. The basic mechanical properties as elastic modulus and flexural strength were determined for all prepared materials. The crack propagation changes due to effect of internal stresses, elastic mismatch and surface effects were investigated using modified single edge notched beam technique. An extensive fractographical analysis of fracture surfaces was undertaken using laser confocal microscopy. The changes of the crack direction when crack propagates through alternating layers under different angels were described. Further, the effect of the internal stresses level within individual layers was reported.

Keywords: Ceramics; Laminates; Crack propagation; Internal stresses; Fracture behaviour

1. Introduction

Layered materials are used in industry for a long time. The engineering laminates consisting of layers of ceramic materials are used rather in very special applications.[1–4] Generally the critical point in employing of ceramic materials is their low reliability. Proper design of such ceramic based laminates is one way how to enhance this key parameter and keep other advantages of ceramics. The layered structures as a special type of composite structures offer high variability in tailoring of properties.[5–8] This contribution is dealing with layered ceramics composites having strong interface bond enabling to control the internal stresses. The internal stresses are developed during sintering process due to differences in thermal expansion coefficients of constituents and can be theoretically estimated.[8–11] The presence of internal stresses can enhance wear, strength and moreover fracture toughness properties if the compressive stresses are present in the outer layer and/or reliability if they are developed in the second layer.[7,12,13] The influence of the internal stresses on fracture behaviour of some model layered materials was described experimentally and also numerically.[8,14–22] From these studies it is obvious that it is possible to increase the material characteristic called apparent fracture toughness when the crack propagates through the laminated structure. Most of these experiments and numerical simulations concentrated effort to the crack which propagates in the perpendicular direction to the layers. This contribution tries to describe these effects connected to the level of internal stresses on the fracture process when crack is not perpendicular to the layer interface.

2. Experimental

A set of laminated and monolithic materials was prepared using the electrophoretic deposition technique. The deposition process uses isopropanol suspension of ceramic powder (Al_2O_3 or ZrO_2) in the presence of monochloracetic acid as a surfactant. The main reason of application of monochloracetic acid together with isopropanol is their easy purification from water. Comprehensive description of laminates preparation can be found elsewhere.[23] Parameters of the prepared materials having around hundred alternating individual layers are summarised in Table 1 together with calculated theoretical internal stresses. All laminates had evenly alternating alumina and zirconia layers with the same layer thickness for one component within one laminate, i.e. the specific laminate had symmetric and periodically layered structure. The only difference between laminates was the thickness ratio of layers as is indicated in the table where is shown the nominal and true (measured after

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Table 1
Prepared materials and calculated theoretical internal stresses (sign minus = compressive; plus = tensile).

Material	Layer thickness Al_2O_3/ZrO_2 [μm]	Thickness ratio nominal/true	Internal stresses α-Al_2O_3 [MPa]	t-ZrO_2 [MPa]
A	–	–	0	–
A2/Z1	52.5/25.5	0.667/0.673	−176	+362
A1/Z1	52.5/53.0	0.500/0.498	−295	+292
A1/Z2	26.0/60.0	0.333/0.302	−455	+197
Z	–	–	–	0

sintering) thickness ratio. The discrepancy between nominal and true thickness ratios is relatively low and it was given by irregularities in the kinetics of the electrophoretic deposition. Material was supplied in the plate form of nominal dimensions 25 mm × 50 mm × 4 mm. The precise saw Isomet 5000 (Buhler) was used for specimen sectioning. A razor blade together with a fine diamond paste was applied to prepare sharp V notch. Elastic properties represented by the elastic modulus were determined at first non-destructively using a frequency resonance technique by Grindosonic equipment according to the standard (EN 843-2). The Archimedes method (EN 623-2) was employed to determine density of each specimen essential for elastic modulus calculations. The three and four point bend set-up (span of 10 and/or 10/5 mm, respectively) for testing of specimen having nominal cross-section of 2 mm× 1.5 mm and length 12 mm was used. Three point bending was used for the flexural strength determination (based on EN 843-1) with the cross-head speed of 50μm/min. The surface deformation pattern of the specimen was measured by non-contact ESPI method. This gathered information allowed calculation of the deformation development. The force–deflection curves were constructed and elastic modulus was calculated by the standard (EN 843-2). A modified SEVNB specimens where initial sharp notch was declined to the layer normal (nominally of 12°) were employed for fracture process observation.[24] To allow a crack propagation not affected by the effect of the external loading conditions a four point configuration was used where even stress distribution between inner rollers occur. The cross head speed was set to 20 μm/min. Electromechanical testing system 8862 (Instron) equipped with ESPI Q300 (DantecDynamics) for non-contact 3D surface deformation measurement was employed. Fracture surfaces were reconstructed by laser confocal microscope Lext OLS3100 (Olympus).

3. Results and discussion

Behaviour of prepared laminated structures was influenced by the presence of internal stresses developed within the individual layers. The theoretical calculation of such stresses can be done on the basis of volume fraction of components, i.e. independently on layers thickness and its order when the number of layers is high enough.[8] The dependence for the given component system can be calculated and is displayed in Fig. 1. In the diagram are two curves (dashed blue line for ZrO_2 and dotted red line for Al_2O_3) each indicating the level of internal

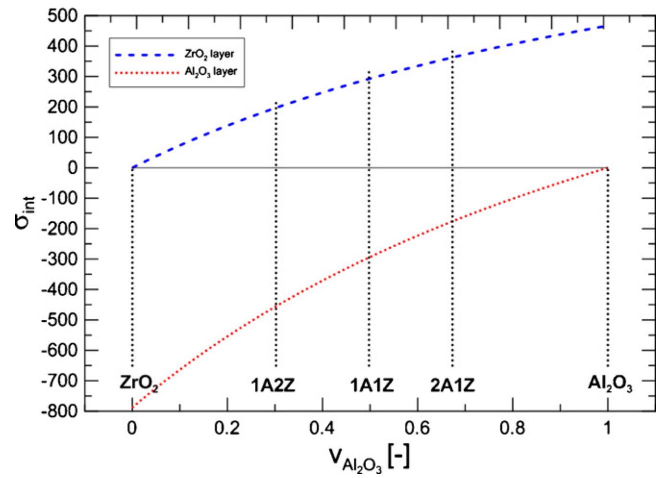

Fig. 1. Dependence of internal stresses in the layers on volume fraction of Al_2O_3.

stresses in the layer. There are marked compositions of materials used in this study with the composition from pure ZrO_2 to pure Al_2O_3. Such diagram can be used also for the design of laminated structure based on knowledge about loading conditions. Another important characteristic playing role in the fracture behaviour of the material is modulus of elasticity because the sharp change of elastic properties cause deflection of the propagating crack. The dependence of elastic modulus on the volume fraction of Al_2O_3 component is shown in Fig. 2. There are summarised data obtained from three different methods. Blue triangles represents results from the frequency resonance method, red diamonds were obtained from the loading curve of the flexural strength test and black circles are values calculated from the mixing rule. There is visible higher scatter of experimentally obtained values from the flexural strength test. However, it is possible to declare a good agreement between both experimental and calculated data using the mixing rule. The different situation appear when flexural strength and fracture toughness are evaluated. Fig. 3 shows dependence of flexural strength and Fig. 4 dependence of fracture toughness. It is obvious that both the flexural strength and the fracture toughness keep the level

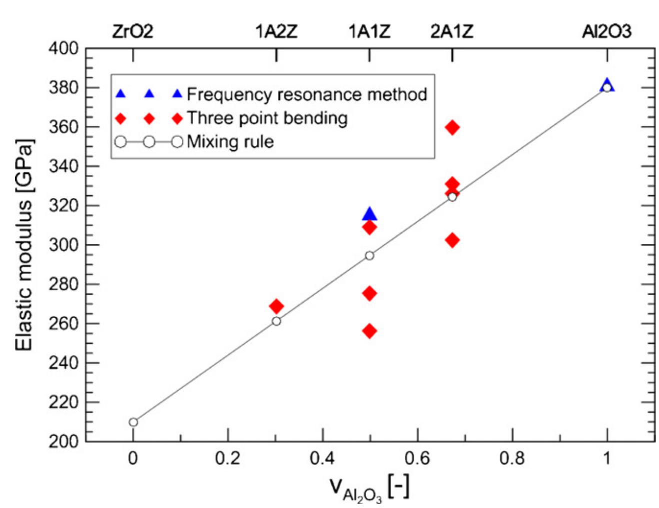

Fig. 2. Dependence of elastic modulus on volume fraction of Al_2O_3.

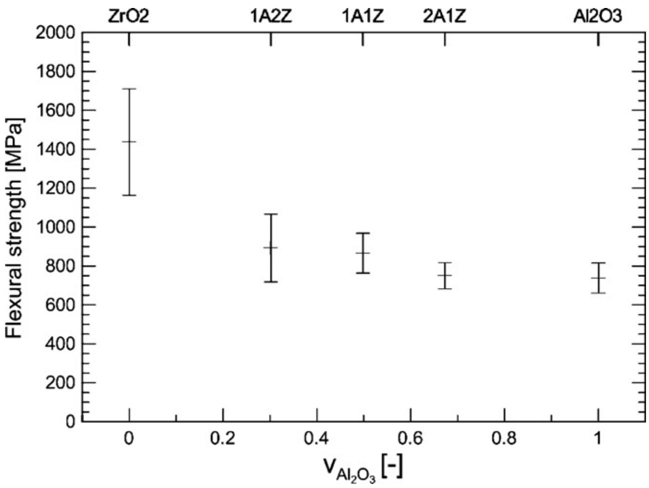

Fig. 3. Dependence of flexural strength on volume fraction of Al_2O_3.

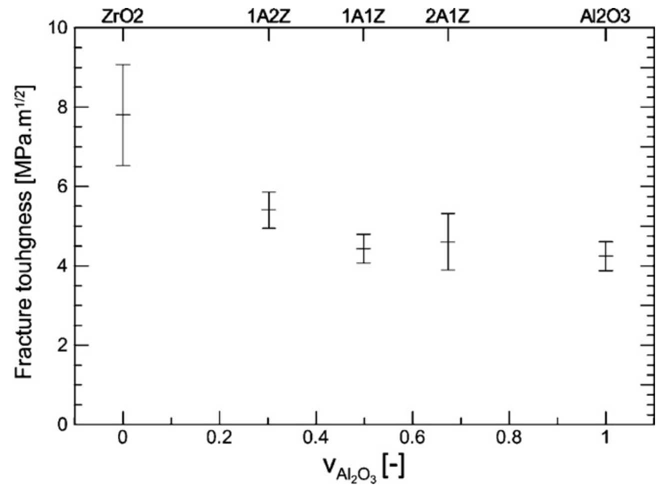

Fig. 4. Dependence of fracture toughness on volume fraction of Al_2O_3.

of properties of the weaker component (in this case of Al_2O_3) and only small improvement is detectable with increasing level of compressive stresses. This effect is caused by high number of layers in the specimen cross-section that imply low layer thickness when symmetrical and periodic layer structure is used. The "weaker" layer is therefore always facing stresses on the level which is very close to the level of maximal stress applied. Additionally, the properties of the layer containing tensile internal stresses (in this case "stronger" layer of ZrO_2) are affected by the superposition of internal tensile stresses with the stress field created by the loading. Therefore, the flexural strength of the laminate is close to the flexural strength of the weaker layer in spite of the amount of volume fraction of "stronger" material in the laminate. All mentioned effects are predictable and can be estimated using numerical simulation of designed materials. However there are some fracture effects which are usually omitted. One is the crack trajectory which can be in some cases predicted but when internal stresses are present and crack is not propagating perpendicularly to the layers the prediction becomes more complicated. The previous experimental works[22–24]

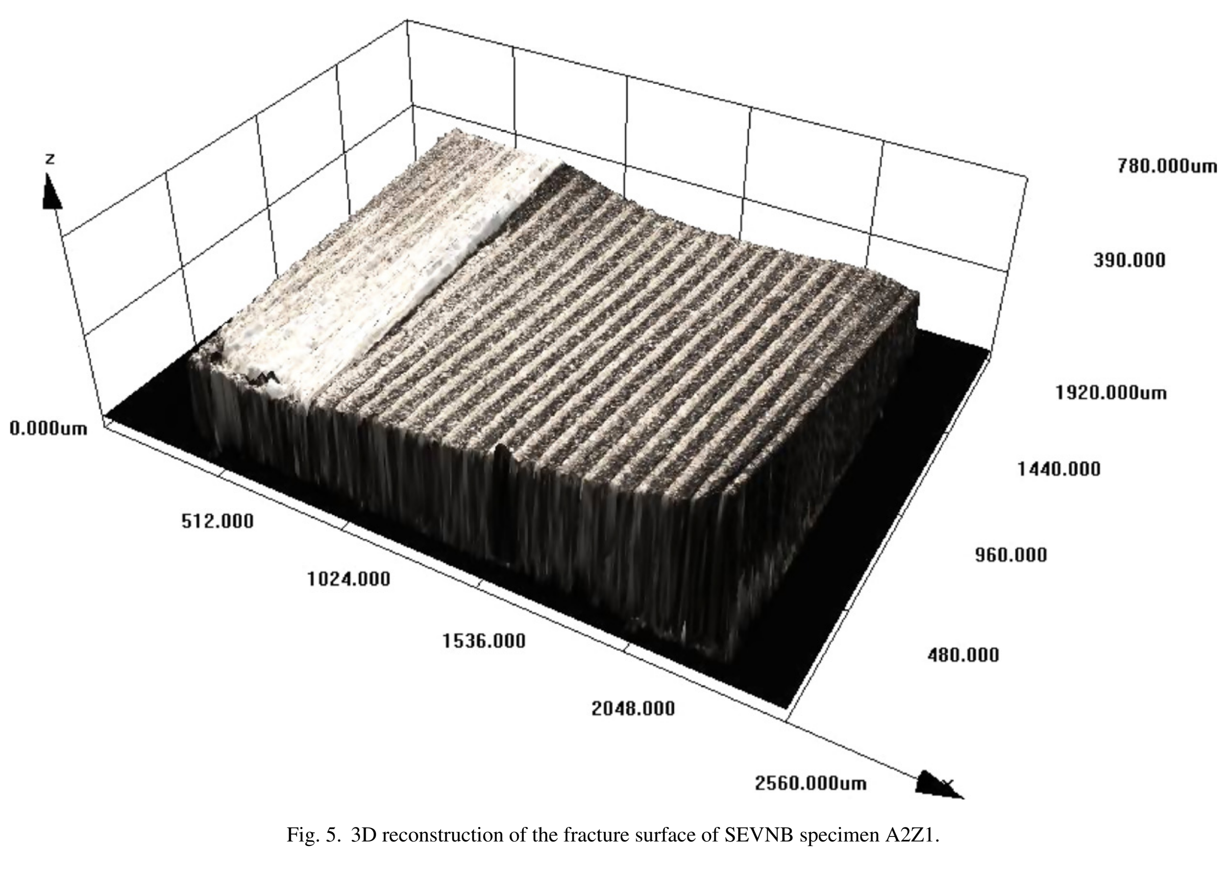

Fig. 5. 3D reconstruction of the fracture surface of SEVNB specimen A2Z1.

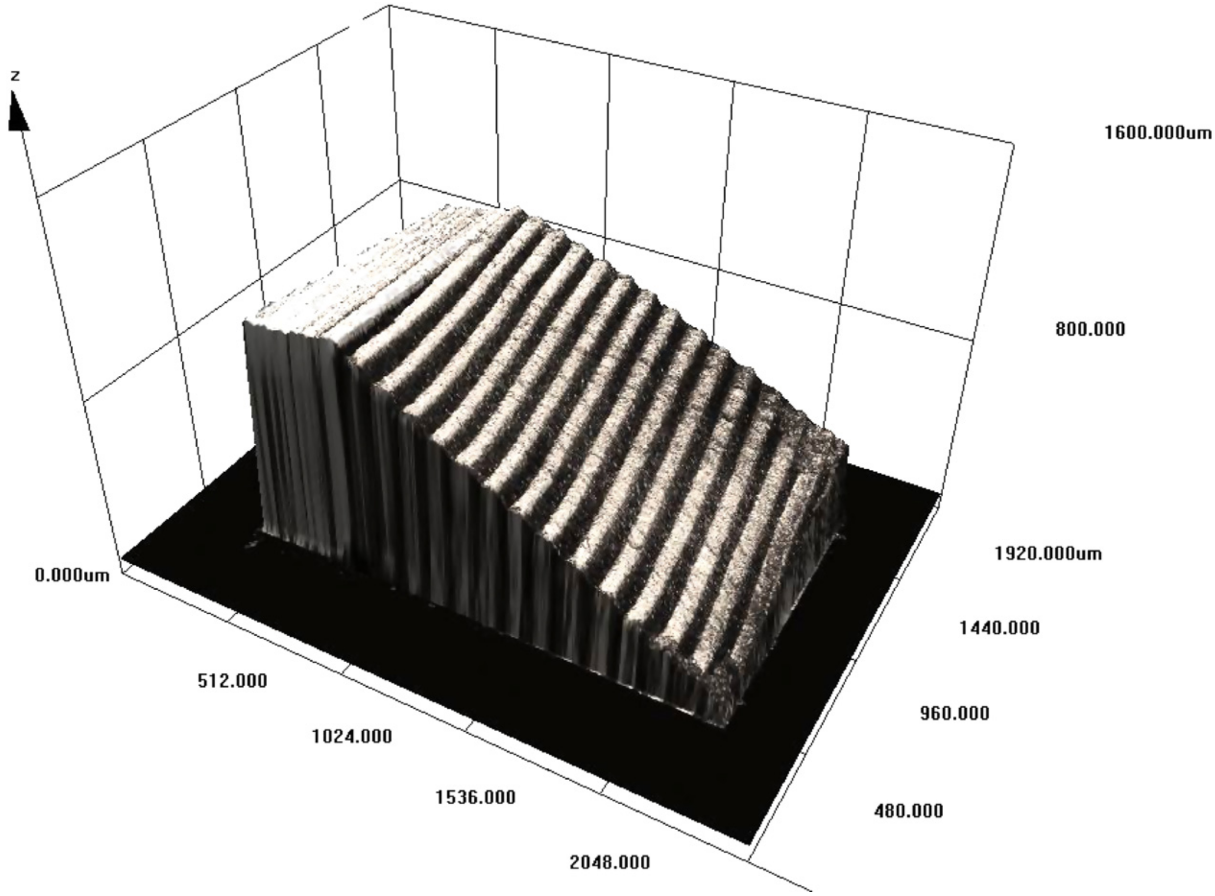

Fig. 6. 3D reconstruction of the fracture surface of SEVNB specimen A1Z2.

conducted on surface cracks shows that there is an effect of elastic modulus of individual layers and effect of the crack entering angle on the crack propagation through the interface of layers. Figs. 5 and 6 show 3D reconstruction of fracture surfaces obtained from the flexural tests of modified SEVNB specimens with different internal stress level in the layers. The significant influence of the internal stresses on the crack trajectory is evident. The fracture surface of A1Z2 specimen (estimated compressive stresses on the level 455 MPa) displayed in Fig. 6 has significantly higher macroscopic crack deflection than the fracture surface of A2Z1 specimen (estimated compressive stresses on the level 176 MPa) showed in Fig. 5. The difference of crack deflection is amplified by impact of tensile stresses which are nearly twice higher for A2Z1 laminate. The compressive stresses play an important role because deflect a crack to the direction parallel with layer interface on the contrary tensile stresses helps to open crack and deflect crack to the layer normal direction. The higher level of compressive stresses is the higher crack deflection was observed. Additionally, it was also observed that deflection of the crack is different along the crack front. On the places situated close to the specimen free surfaces are the effects smaller comparing to the central part of the specimen. This difference is probably given by changed stress conditions on the specimen edges. Therefore the deflection was observed higher for the central part of the crack front for given laminated material (given internal stresses) and depends on the level of internal stresses.

4. Conclusions

Laminates with the strong bonds between evenly alternating layers were prepared by electrophoretic deposition and fracture behaviour was investigated with respect to the level of internal stresses. The positive effect of internal compressive stresses on the crack trajectory was identified. This effect weakens in the area close to the specimen edges (free surfaces). Very thin layers used in this laminates lead to non-linear dependence of both flexural strength and fracture toughness and do not respect the mixing rule of components, i.e. the experimentally measured values are lower then calculated ones. The elastic properties of laminates fully respect the mixing rule.

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