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Microstructure-based modelling and experimental investigation of crack propagation in glass–alumina functionally graded materials

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Abstract

The aim of the present work was the determination of the fracture mechanisms in glass–alumina functionally graded materials (FGMs). The investigation was performed by means of a combined approach based on microscale computational simulations, which provided for an accurate modelling of the actual FGM microstructure, and experimental analysis. The numerical results proved that microstructural defects, such as pores, deeply influenced the damage evolution. On the contrary, the minimization of the mismatch in the coefficients of thermal expansion of the ingredient materials allowed to obtain low thermal residual stresses, which did not relevantly affect the crack propagation. In order to support the numerical model, microindentation tests were performed on the cross-section of FGM specimens and the experimentally observed crack paths were compared to the computationally predicted ones.

Keywords: Microstructure-final; Fracture; Al_2O_3; Glass; Functionally graded materials; Crack propagation

1. Introduction

Functionally graded materials (FGMs) are multi-phase systems, whose composition and/or microstructure are not uniform in space, but vary following a pre-determined profile.[1,2] The gradual compositional change yields a spatial gradient of materials properties and functions, which distinguishes functionally graded materials from traditional composites. If properly designed, the introduction of a smooth variation may significantly reduce the large-scale stress singularities which usually arise at sharp interfaces between heterogeneous materials.[3] Functionally graded materials are currently used in several applications, especially if the thermo-mechanical loading conditions are not uniform in space, since the gradient of microstructure and related properties can be tailored to the service requirements.[4] Therefore, they are used as thermal barrier coatings for nuclear reactors, turbine blades and internal combustion engines, but they are also applied in machine tools, earth moving equipments and military armour.[5] Functionally graded materials are also widely employed in microelectronic devices, aerospace vehicles and bioactive implants.[1] Most of all, FGMs are successfully used as protective coatings in order to face problems of contact damage. For example, in glass–alumina functionally graded materials the gradual variation in Young's modulus may prevent the formation of Hertzian cone cracks, due to stress redistribution.[6] The resistance to sliding-contact may be optimised as well.[7]

In order to carry out a proper microstructural design, great attention has been recently devoted to the FGM microstructure–property relationship. In graded materials the definition of this relation is more complex than in traditional composite materials, since the global property values cannot be related to a single microstructural parameter (e.g., reinforcement volume fraction in not graded composites). As a consequence, many researchers have been looking for the connection between the FGM overall behaviour and a function describing the compositional variation over the whole component.[8] Nevertheless, the FGM microstructure is usually discrete and stochastic, since distinct domains of heterogeneous composition can be identified at the microscale. This means that, even if the compositional gradient is the same at the macroscale, the domains of the constituent phases may have variable placements at the microscale. Dao et al.[9] proposed a first computational micromechanics model to evaluate the effect of discreteness and randomness on thermal residual stresses at the grain size level. On average, the micromechanics approach

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predicted the same macroscopic stresses given by a continuous model, which assumed that the materials properties changed in a continuous way. Yet the micromechanics model highlighted relevant residual stress concentrations at the grain size level, which could initiate local damages and eventually lead to large-scale failures. This investigation, however, used square "building blocks" in order to generate the discrete microstructures. In other words, the model geometry described the microstructural grains with square elements.[9] However, in this kind of approach, the model geometry neglected the actual microstructural features, such as real grain shape or local defects, and pores were not considered.

The target of the present work was to analyse the effect of the actual FGM microstructure taking into consideration all its features. In particular, this research was focused on a glass–alumina system. In fact, though in the past functionally graded materials typically included at least one metal phase,[10,11] currently ceramic–ceramic and glass–ceramics systems are the object of concentrated researches due to their interesting potentialities. In the present study, a $CaO–ZrO_2–SiO_2$ glass was used, since the glasses belonging to this ternary system usually show interesting mechanical properties, such as a high Young's modulus and a relatively good fracture toughness (K_{IC}).[12–14] Moreover, the absence of Al_2O_3 in the glass composition was helpful to characterize the resulting functionally graded materials, since it was easier to distinguish the glass domains and the alumina grains, for example, by scanning electron microscope–X-ray energy dispersion spectroscopy (SEM–EDS) analysis. Finally, in order to reduce the mismatch in thermal properties, the glass composition was carefully formulated thus achieving a coefficient of thermal expansion similar to that of the alumina.

The microstructure-dependent crack growth in such glass–alumina functionally graded materials was investigated via a microscale computational modelling. The simulations were carried out by using OOF,[15,16] an innovative finite element code, which is able to read microstructural images and materials properties as input data. The numerical model, which can be built on the basis of such data allows to evaluate the effect of the constituent phases and pores. The crack propagation was simulated by coupling the finite element method with the Griffith theory.[15] The effect of the thermal residual stresses induced by the fabricating process was also considered and the results were compared with the ideal case of a stress-free system.

2. Experimental

The preparation and experimental characterization of the ingredient materials – glass and alumina – and the functionally graded materials were accurately described in a previous work.[13] The relevant properties of the ingredient materials are summarized in Table 1. The functionally graded materials were obtained by means of glass percolation into the alumina substrate, which was induced by a proper thermal treatment. The samples were heated up to 1600 °C and left at this temperature for 4 h. After this treatment the FGM specimens were taken out from the furnace at 1000 °C and cooled down in the air in order to avoid the crystallization of new phases.[13,14]

Table 1
Ingredient materials properties[13]

Ingredient materials	Young modulus (GPa)	Poisson ratio	Thermal expansion coefficient (°C^{-1})	Fracture toughness (MPa m$^{1/2}$)
Glass	96	0.27	8.65×10^{-6}	0.90
Alumina	358	0.20	8.18×10^{-6}	2.60

A functionally graded material was cut and the cross-section was observed by using a SEM. The microscopy observation, coupled with a local chemical analysis, revealed that the glass infiltrated the alumina reaching a depth of at least 800 μm. The molar percentages of Al_2O_3 and SiO_2 – the latter being considered as a marker of the glass – were measured as a function of depth, but the trend was not strictly monotonic. This result, however, is reasonable, since the FGM microstructure is discrete and random, while the EDS chemical analysis is a punctual measurement technique. The microstructural investigation, moreover, revealed the presence of defects, such as pores, which had not been completely filled by the glass, as illustrated in Fig. 1.

In order to point out the microstructural details, a FGM slice was chemically etched with a 4% hydrofluoric acid for 10 s at room temperature. This chemical etching removed the glass, but did not attack the alumina grains. The cross-section was observed under the SEM as well, several digital images were acquired and then joined via an image-elaboration software. This operation allowed to obtain a picture of the entire profile, reproducing a 100 μm wide, 800 μm deep cross-section area.[17]

Finally, a FGM sample was suitably cut, embedded in unsaturated polyester resin and carefully polished. Then Vickers microindentations (REMET HX-1000) were carried out on to the FGM cross-section by applying a load of 1000 g_f for 15 s. The cross-section was observed by using a SEM, devoting particular attention to the crack paths engendered by the microindentations. The effect of the microstructural details on the crack propagation was analysed and the possible toughening mechanisms were evaluated.

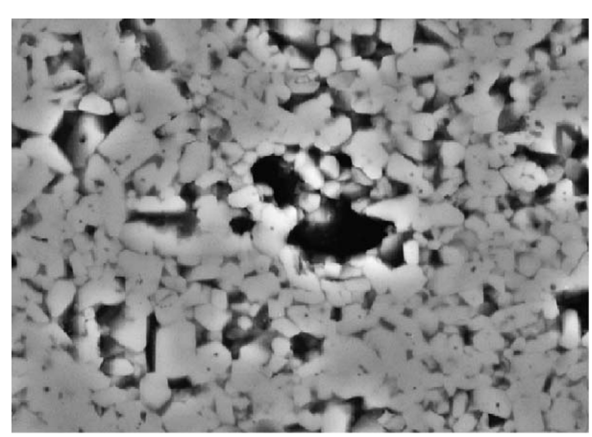

Fig. 1. Detail of a BSD-SEM image of the FGM cross-section (not chemically etched); the image refers to a microstructural defect, that is a pore which was not filled by the glass.

3. Microscale modelling

The microscale modelling was performed by using OOF,[15] an image-based finite element code. As a matter of fact, this software is able to directly acquire microstructural images, such as SEM micrographs, and create 2D finite element meshes.

Since this computational tool is able to operate at the microscale, it is possible to correlate materials properties to microstructure. As a consequence, OOF has been widely used in the past in order to investigate heterogeneous systems such as composite materials. Hsueh et al., for example, studied the stress transfer mechanism in platelet-reinforced composite materials.[18] The same authors also investigated thermal barrier coatings, devoting particular attention to the residual stress distribution and the effect of superficial irregularities.[19,20] Zimmermann et al. analyzed the thermal residual stresses which may arise in ceramic materials because of their thermal expansion anisotropy.[21] A second work was dedicated to the damage propagation in brittle polycrystalline materials.[22] Zimmermann et al. used OOF in order to model the fracture mechanism in textured anisotropic ceramic systems.[23] Vedula et al. modelled the residual stress distribution and spontaneous crack propagation, which may be caused in a polycrystalline alumina by a temperature decrease.[24] Saigal et al. analyzed the microcrack propagation in iron titanate[25] and the distribution of internal stresses in aluminum–silicon alloys.[26] Cannillo and Carter applied OOF to the evaluation of the reliability of composite and heterogeneous systems.[27] Cannillo et al. simulated the elastic behaviour and damage evolution in porous glasses.[28] Moreover, Cannillo et al. investigated glass matrix composite materials, reinforced by means of alumina platelets[29–31] and molybdenum particles.[32] Wang et al.[33] applied OOF to SEM micrographs in the intent of investigating the effect of pores and interfaces on the effective properties of plasma-sprayed zirconia coatings, such as elastic modulus and thermal conductivity. In particular, this microscale model was compared with an alternative computational approach, based on an experimental measurement of porosity and a subsequent artificial graphical re-building of the microstructure. Finally, in a previous work,[34] OOF was used in order to analyse the thermal residual stresses in glass–alumina functionally graded materials.

The simulations carried out in this research focused on fracture mechanisms in functionally graded materials, which is a crucial theme in order to evaluate their reliability. With this aim, the SEM picture of the complete FGM profile was read by the pre-processor, the constituent phases were identified and the respective materials properties were assigned in accordance with Table 1. Then the microstructural image was mapped onto a 2D finite element mesh. Since the SEM observations confirmed the presence of some residual porosity in the FGM cross-section, pores were considered as a third constituent phase, along with the alumina and the glass. The mesh was created with triangular elements, having about 92,000 elements. A detail of a microstructure-based grid is represented in Fig. 2.

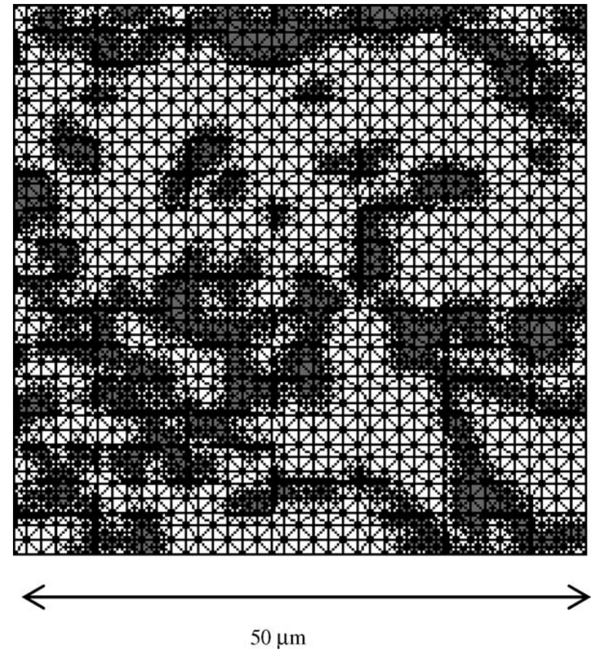

Fig. 2. Finite element mesh (detail located just beneath the surface).

The crack propagation was computationally predicted by coupling the finite element method, applied at the microscale, with the Griffith theory. In OOF, according to the Griffith criterion, a crack propagates if the total surface energy required for the creation of the new surfaces can be supplied by the elastic deformation energy stored in the body.[15]

In the simulation, the sample is loaded and the energy balance is computed. If the Griffith condition is satisfied, the stiffness matrix of the cracked element is reduced according to a specific parameter, which is usually assumed equal to 0.01. Such procedure allows to track the damage evolution up to the complete failure of the system, which is assumed when a crack propagates from one boundary of the mesh to the opposite one. In the present study, a unidirectional loading was applied parallel to the infiltration direction and crack patterns were determined.

Moreover, since the alumina and the glass have similar but not identical coefficients of thermal expansion, the thermal residual stresses that may arise during fabrication were considered. As a matter of fact, below the glass transition temperature the glass viscosity is so high that the thermal stresses cannot be relaxed: in particular, the critical step of the fabricating process was the final cooling down, between the glass transition temperature, which was measured to be 790 °C,[13] and room temperature. The resulting value and distribution of the thermal residual stresses were thoroughly studied in a previous work.[34] In order to account for the effect of thermal residual stresses on crack propagation, in a second simulation a drop of temperature of −770 °C was set to simulate cooling, then the thermal stresses were calculated and the fracture mechanisms were analysed by loading the system as in the previous case. Fracture paths obtained both considering and neglecting the thermal residual stresses were compared in order to evaluate the effect of the thermal history on the FGM behaviour.

4. Results and discussion

The effect of microstructural details on crack propagation in glass–alumina FGMs was investigated. In fact, the computational simulations were performed on the microstructure-based column-like image of the FGM cross-section obtained as described in the previous paragraph. The model, therefore, implicitly accounts for the real graded distribution of the constituent phases, since the image clearly shows that the glass volume fraction decreases as a function of depth.[13,17] In a previous contribution,[17] an analogous column-like picture was used to evaluate the compositional profile, which resulted to be parabolic. Moreover, the SEM acquired photographs also include microstructural defects, such as pores, which may deeply influence the crack propagation.

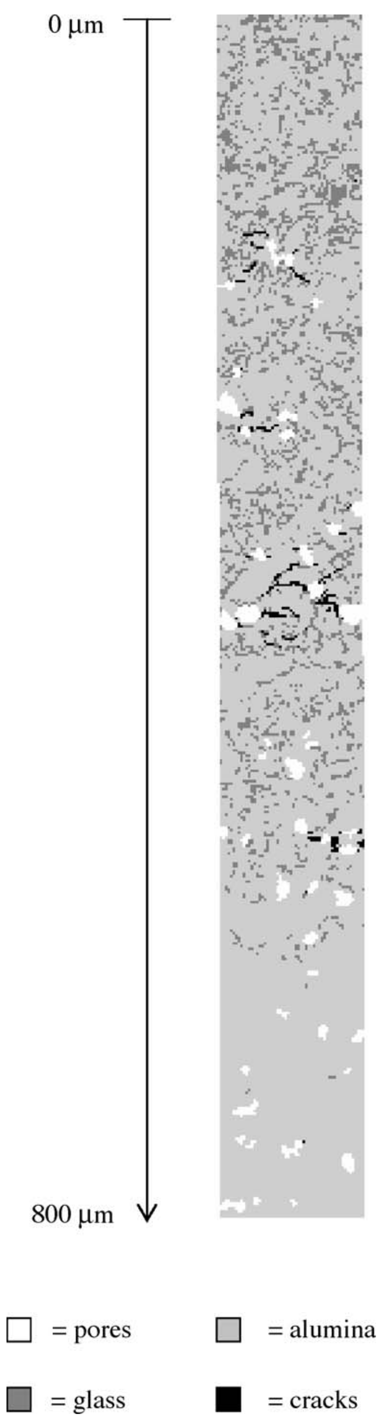

Fig. 3. Damage evolution in the FGM cross-section (strain = 0.4%).

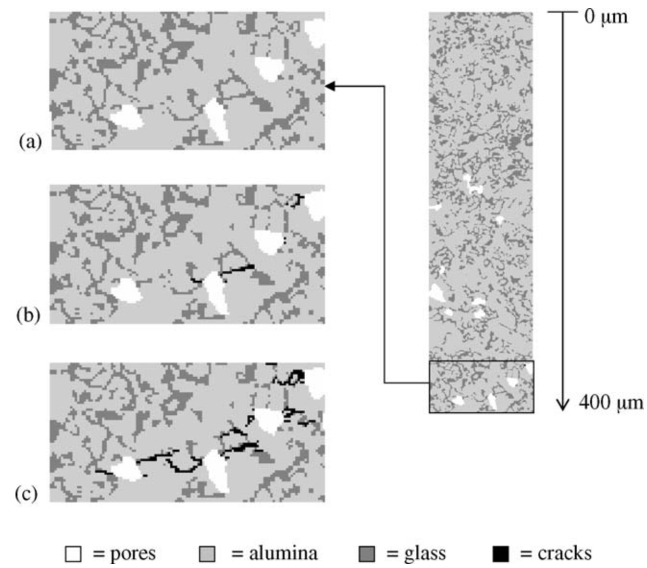

Fig. 4. The simulations performed reveal the influence of microstructural details (a) on the fracture mechanisms. In fact cracks are likely to initiate ahead of pores (b, strain = 0.4%) and then propagate throughout the glass domains(c, strain = 0.6%).

An example of a typical crack path is shown in Fig. 3.

The simulations performed suggest that pores act as crack-triggers, as the first elements to fail are preferentially located next to pores, especially the larger ones; as a matter of fact, pores are the weakest link in the microstructure.[35] Then cracks are likely to propagate through the glass phase, as depicted in Fig. 4. In fact, the glass is weaker than the alumina, which in turn has a higher value of the fracture toughness (K_{IC}) as reported in Table 1, and therefore, it is reasonable that cracks preferentially propagate through the glass phase. Accordingly, the simulations show that the first cracks in the FGM cross-section develop in the area at about 400 μm of depth, where the glass phase is still abundant but the pores are not completely filled. It is only when the applied load is increased that cracks propagate also in the superficial area, which is characterized by the highest glass volume fraction but is devoid of pores, and in the deeper area, which is still defective but really rich in alumina.

Though the glass toughness is lower than the alumina one, the glass penetration into the alumina substrate results into a toughening mechanism, since it establishes a complex crack mechanism by modifying the damage evolution of the pure alumina. In particular, the glass phase plays a double role: since the glass is mechanically weaker than the alumina, cracks preferentially move throughout the glass domains, thus following a tortuous path; on the other hand, the glass partially fills the alumina pores, which are likely to initiate cracks. By the way, some simulations carried out on the pure alumina cross-section show that the superficial area (up to about 300 μm of depth), whose pores are not filled by the glass, is more severely damaged than the correspondent area of the FGM cross-section (under the same applied strain).

In conclusion, the simulations confirm that the FGM microstructure morphology deeply influences the damage evolution: the crack propagation is governed by the constituent phase distribution and gradient.

As already mentioned, in order to evaluate the effect of thermal residual stresses on the crack propagation, the simulation was repeated by setting a temperature decrease equal to −770 °C before mechanical loading. The results, however, do not significantly differ from those previously found. A comparison of the damage evolution in the examined structures, with and without thermal residual stresses, is shown in Fig. 5. A quantitative analysis of the predicted data is presented in Fig. 6; the graphs were constructed by plotting the fraction of fractured elements, which is a damage parameter, as a function of the applied strain. The simulation proves that, since the thermal residual stresses are very low in value, they can be neglected due to their reduced effect on fracture propagation. Actually, the glass composition used in this work was carefully designed in order to reduce the mismatch in thermal properties of the ingredient materials, thus minimizing the residual stresses.[34]

Thus, it can be remarked that, according to the numerical simulations, the fracture mechanisms in the investigated functionally graded material are marginally influenced by the thermal residual stresses, which are really low in value thanks to the optimisation of the ingredient materials. On the contrary, fabrication-induced defects, such as residual pores, govern the damage evolution, acting as crack-triggers. Moreover, the evolution of the crack paths is influenced by the interplay of the constituent phases. In particular, the glass is a preferential propagation site, since it is less tough than the alumina, but it plays a beneficial role by filling some pores and inducing a complex crack mechanism.

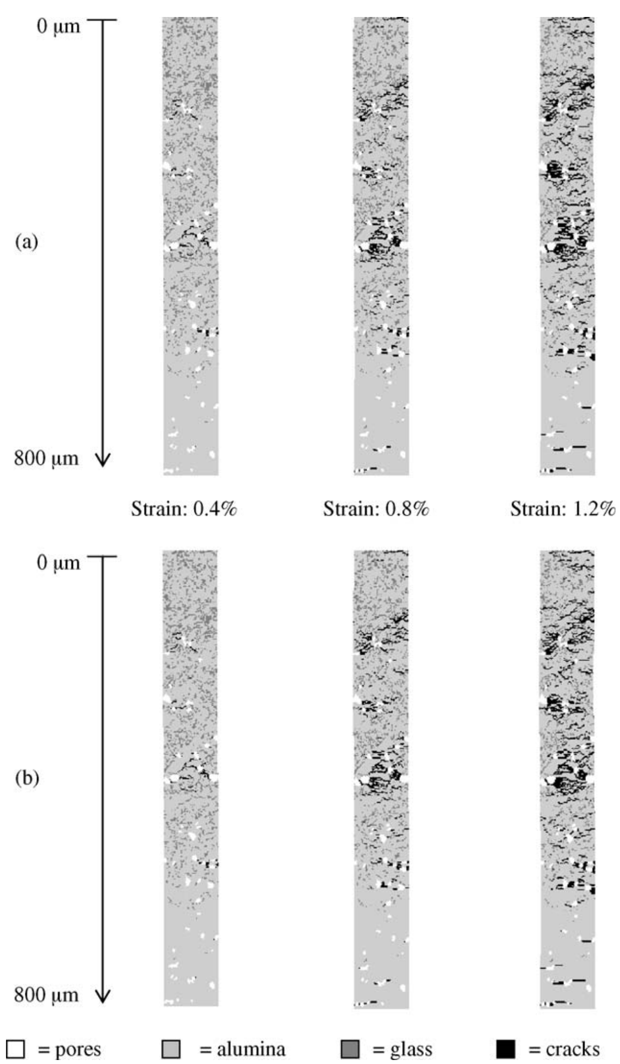

Fig. 5. The damage evolution in the FGM cross-section is not deeply influenced by the thermal residual stresses, which are really low in value due to the minimized mismatch in thermal properties of the alumina and the glass: (a) crack propagation with thermal residual stresses and (b) crack propagation without thermal residual stresses.

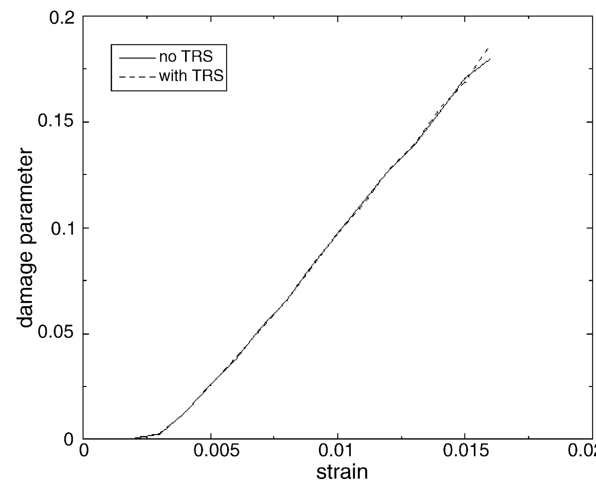

Fig. 6. The damage parameter as a function of the applied strain was calculated with (dashed line) and without (solid line) thermal residual stresses (TRS). The comparison between the graphs show that the residual stresses, which are low in value, do not affect the crack propagation.

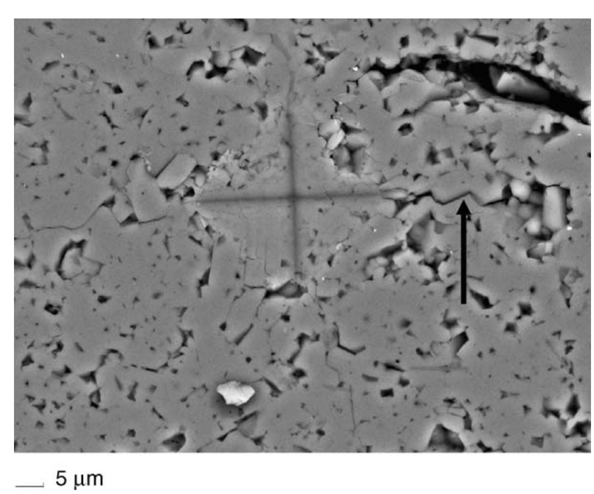

Fig. 7. The microindentations of the FGM cross-section (load of 1000 g_f applied for 15 s) were investigated devoting particular attention to the crack propagation. As indicated by the arrow, the cracks induced by the microindentations usually propagate towards pores.

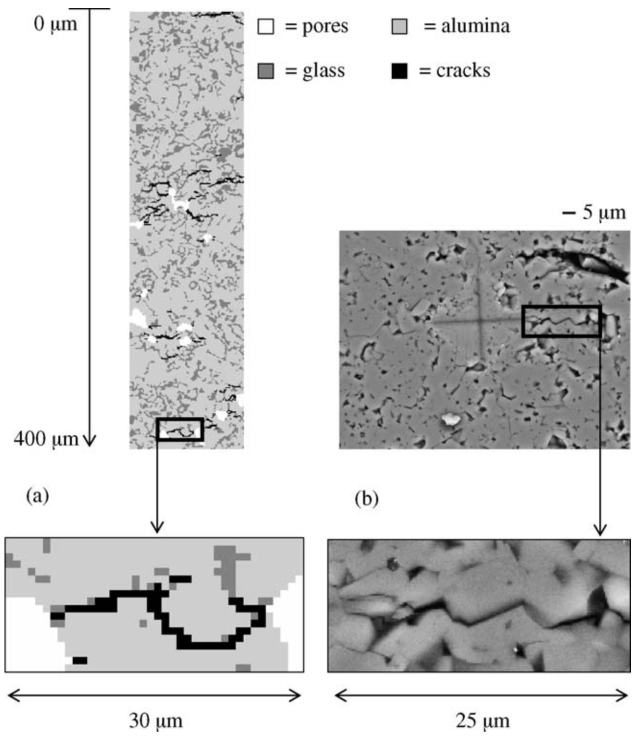

Fig. 8. Comparison between the damage evolution predicted via the computational modelling (a) (strain = 0.6%) and the experimental results (b). Cracks preferentially start from and propagate towards pores.

The experimental evidence provided by the microindentation tests supports such computational results. An example of the cracks propagating from the indents on the functionally graded material cross-section is shown in Fig. 7. A direct comparison between a typical experimental crack path and a simulated one is represented in Fig. 8. Of course, the loading conditions are different in the numerical model and in the experimental approach. Nevertheless, as predicted by the microscale simulations, the cracks induced on the FGM cross-section preferentially evolve starting from pores and moving towards pores. Moreover, the experimentally observed crack paths are likely to develop along the alumina grain boundaries, where the glass is usually located,[36] as predicted by the numerical model. The good qualitative agreement between simulations and experimental observations confirms the reliability of the approach.

5. Conclusions

This work aimed at investigating the damage evolution in glass–alumina functionally graded materials by means of microscale simulations supported by experimental analysis.

The FGM specimens, obtained via glass percolation into an alumina substrate, were observed by means of a scanning electron microscope. The microstructural investigation of the FGM cross-section, besides confirming the glass penetration, clearly revealed the presence of residual pores, which were not filled by the glass. The images acquired during SEM observation were directly mapped onto 2D finite element meshes, thus providing for an accurate modelling of the actual system microstructure. Then the crack propagation was simulated by coupling the finite element method with the Griffith theory. The numerical tests demonstrated that pores act as crack-triggers, since the first failures occur ahead of pores. Furthermore, cracks preferentially propagate through the glass phase, which is mechanically weaker than the alumina. The computational predictions suggest that the damage evolution in the present system is not significantly influenced by thermal residual stresses; this result, however, depends on the minimization of the thermal stresses, which was achieved via a proper glass formulation. On the contrary, the fracture mechanism is deeply influenced by the distribution of the ingredient materials and by the presence of microstructural defects such as pores.

Though the microscale model is two-dimensional, the numerical simulations agree well with the experimental evidence. As a matter of fact, the FGM cross-section microindentations cause cracks, which propagate from pore to pore, thus validating the computational simulations.

To conclude, a faithful simulation of crack propagation should account for real microstructural details, such as pores and phase distribution, which may deeply influence the system's overall behaviour. The computational approach applied in this work fully satisfies this requirement and ensures a good reliability, thus providing a useful tool to model functionally graded materials and complex multiphase systems.

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