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# Fracture toughness of a layered-gradient ceramic composite $ZrC-Al_2O_3$

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Fracture toughness and propagation of the main crack in ceramic composite $ZrC-Al_2O_3$ with a layered-gradient structure have been studied. Outer layers of the studied material consisted of ZrC and $Al_2O_3$, while intermediate layers were composites of these two materials with different ratios of components. The study has shown that fracture toughness of the layered-gradient composite with crack initiation in the ZrC layer is higher than that in the case of crack initiation in the $Al_2O_3$ layer. This is due to the influence of tensile and compressive elastic residual stresses along the crack path, whose magnitude is determined by the difference in thermal expansion coefficients on different sides of the phase interface crossed by the crack.

**Keywords:** layered-gradient composites, fracture toughness, zirconium carbide, aluminum oxide.

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Due to their high melting point, as well as high hardness and strength, ceramic composites are of particular interest as materials for manufacturing component parts of machines and mechanisms operating under thermomechanical loads and intense abrasive impact [1]. The use of refractory ceramic composites in high-energy setups will significantly increase their power because the permissible operating temperature of ceramic products is higher than that of metal ones [2].

It is known that the main disadvantage of ceramic materials is low fracture toughness causing low tolerance to the crack initiation and development, which often leads to a catastrophic loss of strength [3]. Available methods for increasing the fracture toughness, which imply introducing additional phases and inclusions, are often of a compromise character, and fracture toughness increases due to reduction of other mechanical properties, e. g. hardness, elastic modulus, and wear resistance [4]. Fabrication of layered and layered-gradient composites is one of the efficient approaches to increasing the fracture toughness of ceramic composites, since low fracture toughness significantly restricts the area of ceramic materials application [5]. Study [6] has shown that fracture toughness of layered composite $Al_2O_3-ZrO_2$ reaches $11.7\,\mathrm{MPa}\cdot\mathrm{m}^{1/2}$, which significantly exceeds those of individual components due to deflection and bifurcation of cracks getting the fields of residual compressive stresses caused by the difference in thermal expansion coefficients of the composite layers.

Another advantage of layered ceramic composites is the possibility of creating a material with different properties of surfaces (layers), for instance, obtaining a composite based on oxygen-free and oxide ceramics such as ZrC and $Al_2O_3$, which are interesting as materials for complex electrical-engineering solutions in high-energy setups. Zirconium carbide possesses high thermal stability and is an electrical conductor [7], while aluminum oxide is one of the most commonly used dielectrics. At the same time, fabrication of layered composites $ZrC-Al_2O_3$ is hindered by a significant difference in thermal expansion coefficients of the components, which inevitably induces the composite delamination at the phase interfaces. However, as shown in [8], layered composite of the $(ZrB_2-SiC)-ZrO_2$ system whose components also have different thermal expansion coefficients may be obtained by forming intermediate layers consisting of the initial components with a stepwise decrease in the content of $ZrB_2-SiC$ and increase in $ZrO_2$.

The goal of this study was to determine the fracture toughness and crack propagation trajectory in the layered-gradient composite of the $ZrC-Al_2O_3$ system.

In this work, we have examined a composite with a layered-gradient structure whose layers consisted of ZrC and $Al_2O_3$ in different ratios. Average particle size of the initial ZrC and $Al_2O_3$ powders was $5.7 \pm 4.3$ and $7.3 \pm 6.1\,\mu$m, respectively. The powder mixtures were subjected to high-energy mechanical activation in a planetary mixer for 1 min in argon atmosphere. After the mechanical activation, the average powder-mixture particle size was $1.54 \pm 0.7\,\mu$m. The method for composite fabrication consisted of uniaxial pressing of the first layer in a graphite mold under the load of 2 kN, after which the next layer was poured onto the press, and operations were repeated until the required number of layers was obtained. Further consolidation of the composite was performed by sintering under pressure.

The composite microstructure was examined by scanning electron microscopy (SEM). The composite phase composition was determined by X-ray diffraction under the $\mathrm{Cu}K_\alpha$ radiation). The crystal lattice microdistortions were estimated by the method of graphical construction of the Hall–Williamson dependence [9]. To assess the magnitude of second-order stresses in individual composite layers, there

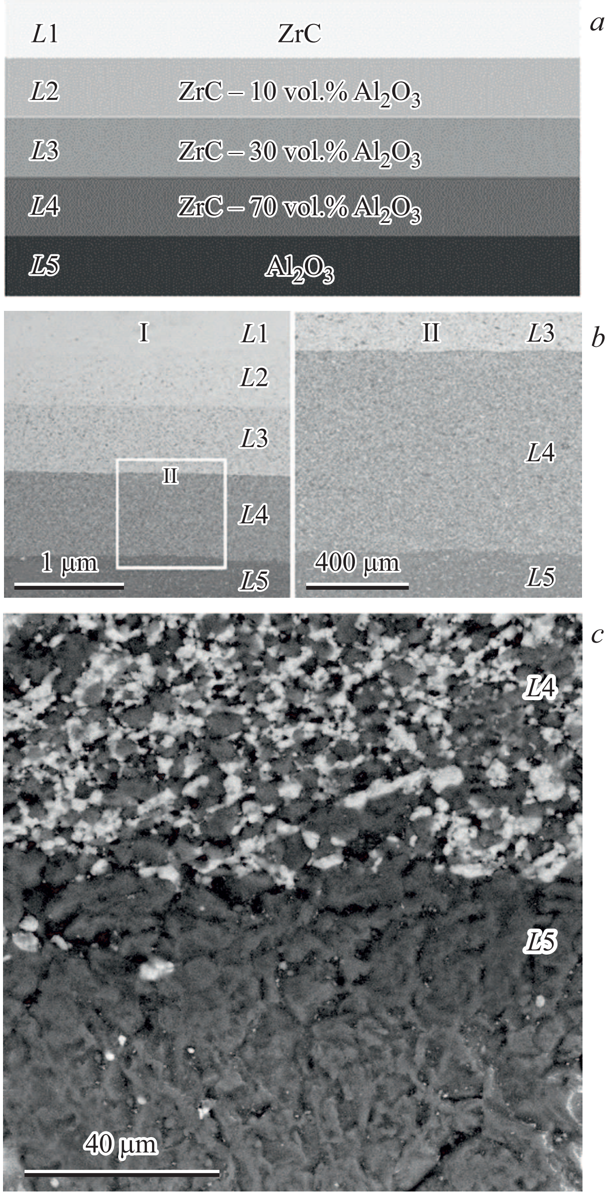

**Figure 1.** *a* — structural diagram of the layered-gradient composite material; *b* — SEM image of the structure of the composite under study; *c* — SEM image of the *L*4–*L*5 layer interface.

was used X-ray imaging of surfaces of each layer parallel section by gradually removing materials with a diamond cutter. The second-order residual stresses were calculated as a product of the crystal lattice microdistortions and Young's modulus of the corresponding phase.

Experimental determination of fracture toughness was performed in accordance with standard ASTM C1421 (A2.4.2) using beam-shaped samples whose outer layers were half as thick as samples as a whole. To compare the crack propagation trajectories, two series of tests were conducted. In the first case, an *U*-shaped notch was made in the ZrC layer (*L*1); in another case, the notch was made in the $Al_2O_3$ layer (*L*5). Radius of the circle inscribed in the notch apex was about 50 $\mu$m.

In this paper, term „layered-gradient composite" is applied because of using layers of dissimilar ceramic phases mixed in different proportions and consolidated in a certain sequence. The structure of the composite under study is characterized by the phase composition variation with distance from one surface to the opposite one; on a macroscale, this allows such composites to be referred to as gradient composites. Fig. 1 shows the structural diagram and SEM images of the obtained composite and layer interface. Proper adjusting of the compositions of intermediate layers (*L*2–*L*4) allowed avoiding delamination of the layered-gradient composite material, which could be caused by the difference in thermal expansion coefficients of the composite components.

Analysis of the X-ray diffraction patterns of the studied composite individual layers showed that the phase composition is represented predominantly by ZrC and $\alpha$-$Al_2O_3$, as well as by low-intensity reflections and $ZrO_2$ monoclinic modification (Fig. 2). The authors of [10] also noted formation of monoclinic phase $ZrO_2$ during interaction between ZrC and $Al_2O_3$.

The Table presents calculations of second-order stresses.

Introduction of 10 vol.% of $Al_2O_3$ into the ZrC matrix makes second-order stresses in ZrC slightly increasing; however, stresses in ZrC relax with further increase in the $Al_2O_3$ content in the layer. On the other hand, it is clear that adding 30 vol.% of ZrC to the $Al_2O_3$ matrix also increases the second-order stresses in the latter; thereat, further increase in the ZrC content is also accompanied by an increase in stresses acting in $Al_2O_3$. Based on the known thermal expansion coefficients $((6.7 \pm 0.2) \cdot 10^{-6}$ and $(7.2 \pm 0.2) \cdot 10^{-6}\,K^{-1}$ for ZrC and $Al_2O_3$, respectively [11,12]), we may conclude that compressive stresses are formed in ZrC, while tensile stresses arise in $Al_2O_3$. The authors of [13] have proposed

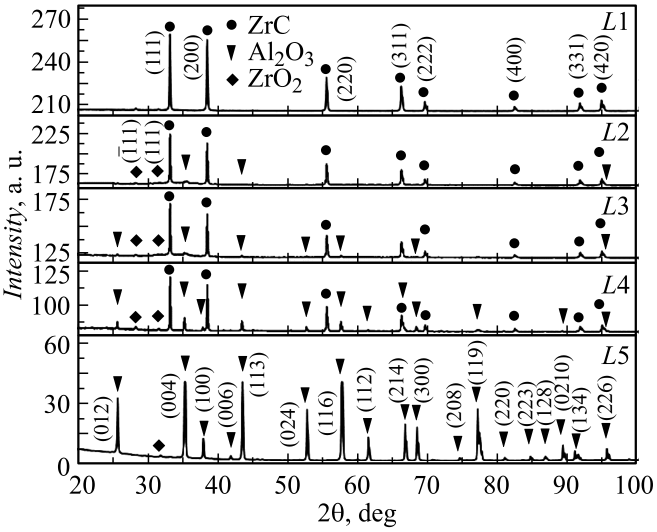

**Figure 2.** X-ray diffraction patterns of individual composite layers.

Second-order stresses in the layers of composite under study

| Layer | Second-order stresses, MPa | |
|---|---|---|
| | ZrC | $Al_2O_3$ |
| ZrC | $672.6 \pm 28.1$ | – |
| ZrC–10 vol.% $Al_2O_3$ | $734.4 \pm 26.7$ | $577.5 \pm 16.1$ |
| ZrC–30 vol.% $Al_2O_3$ | $651.3 \pm 27.2$ | $549.3 \pm 20.4$ |
| ZrC–70 vol.% $Al_2O_3$ | $648.9 \pm 29.5$ | $501.4 \pm 15.7$ |
| $Al_2O_3$ | – | $280.1 \pm 9.2$ |

a formula for calculating residual stresses. Negative stress values evidence for the formation of tensile stresses, while positive ones indicate the formation of compressive ones. Since the difference in the ZrC and $Al_2O_3$ thermal expansion coefficients is negative, compressive stresses emerge in ZrC, while tensile stresses get formed in $Al_2O_3$.

For numerical determination of the fracture toughness of layered composites, paper [14] proposes a method for calculating apparent fracture toughness ($K_{app}$), which accounts for not only mechanical characteristics of individual components, but also residual stresses acting in the composite. Fig. 3 presents a numerical estimate of $K_{app}$, which reflects the behavior of stress intensity in the crack tip in the process of the crack development and approaching the layer interfaces. Layers of the composite in which the crack was initiated in $Al_2O_3$ are designated in the figure upper part ($L5$–$L1$); layers corresponding to the case when the crack is initiated in ZrC are designated in the lower part ($L1$–$L5$).

The study has shown that, when the crack arises in the $Al_2O_3$ layer and propagates to the opposite side of the composite (ZrC), the material's ability to resist the crack development reduces rapidly. The greatest decrease in the apparent fracture toughness (by more than 20 %) is observed when the distance from the $U$-notch to the crack tip increases from 0 to 0.5 mm; at the distance of 2.5 mm, the apparent fracture toughness is less than $2.4\,\mathrm{MPa}\cdot\mathrm{m}^{1/2}$. The fact that the ZrC thermal expansion coefficient is lower than that of $Al_2O_3$ makes the crack developing from the region of compressive stresses to the region of tensile ones, which provides favorable conditions for its growth. At the same time, in the case of crack initiation in the ZrC layer, a considerable increase in the stress intensity coefficient is observed when its tip approaches the interface between the ZrC and ZrC–10 vol.% $Al_2O_3$ layers. The highest apparent fracture toughness is achieved in the layer located at the distance of 2–2.5 mm between the $U$ notch and crack tip, which is because the crack tip crosses the region of residual elastic compressive stresses. Nevertheless, calculations showed that the observed increase in $K_{app}$, although exceeding the fracture toughness of ZrC, does not reach the values characteristic of $Al_2O_3$.

Experimental assessment of the composite's fracture toughness showed that a crack initiated in the outer $Al_2O_3$ layer has a significantly less curved trajectory than that initiated in the outer ZrC layer: $3.19 \pm 0.05$ and $7.83 \pm 0.10\,\mathrm{Pa}\cdot\mathrm{m}^{1/2}$ respectively. The obtained data show that fracture toughness in the case of crack initiation in the outer ZrC layer is more than twice as high as that when the crack arises on the side of $Al_2O_3$. Comparison of the fracture toughness values determined by two different methods showed that, in the case of crack initiation in the $Al_2O_3$ layer, the apparent fracture toughness is lower than when estimated by the three-point bending method. At the same time, the experimental value of fracture toughness of a composite with the crack initiated in ZrC is more than 2 times higher than the apparent fracture toughness.

Thus, we have found out that formation of residual compressive stresses due to a difference in the layers thermal expansion coefficients near their interfaces is an efficient mechanism for increasing the fracture toughness of the ZrC–$Al_2O_3$ composite having a layered-gradient structure. Evaluation of the apparent fracture toughness in the layered-gradient ceramics of the ZrC–$Al_2O_3$ system allowed us to define the character of crack propagation and fracture toughness variations from one composite layer to another. Our study has shown that the studied composite fracture toughness determined by the three-point bending method of a beam with a $U$-notch are higher than the apparent fracture toughness.

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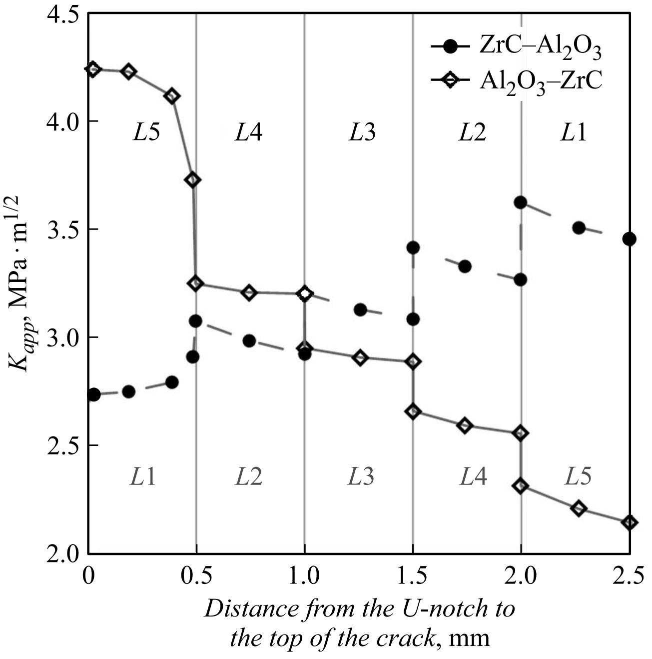

**Figure 3.** Estimation of the apparent fracture toughness of the composite under study.

## Conflict of interests

The authors declare that they have no conflict of interests.

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