PROPERTIES OF Al_2O_3 / ZrO_2 COMPOSITE FOR BIOMEDICAL APPLICATIONS

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Abstract

The paper presents the results of testing a composite based on Al_2O_3 with the addition of zirconium oxide ZrO_2 of 0 %, 5 %, 10 %, 15 %, and 20 %. ZrO_2 powder was stabilized with 8% mol Y_2O_3. The composites were made by compaction of the mixed powders at 60 kN with subsequent sintering of the obtained moulded pieces at 1600 °C for five hours. The examinations of the composites included: determination of density and porosity, microscopic examination, X-ray diffraction (XRD), hardness measurements by the Vickers method, and determination of crack resistance. Microscopic examinations were carried out using scanning electron microscopy (SEM). A SEIFERT 3003 T-T X-ray diffractometer and radiation of a cobalt anode lamp were used to examine the phase composition.

Keywords: Al_2O_3/ZrO_2 composites, XRD phase analysis, mechanical properties

1. INTRODUCTION

The development of modern civilization stimulates the demand for new materials with increasingly better properties and improving the properties of materials already known by modifying their chemical composition and structure. Technologies for the production of materials can be based on sintering powders, applying various types of coatings, and modification of the material surface (for example, using a concentrated energy source) [1-6]. The materials produced in this way offer an interesting alternative that can be used not only in the energy, aviation, and engineering industries (high-strength cutting tools) but also in medicine for medical tools or implants [7-10]. In addition to high bio-tolerance in the environment of tissues and body fluids, high hardness, and abrasion resistance, biomedical materials produced by sintering powders are also characterized by porosity which allows them to be overgrown by tissues and form durable connections. The materials in which Al_2O_3 and ZrO_2 are used play an important role in this respect due to their high biological neutrality [11-14]. In addition, ZrO_2 phase has unique properties associated with the transformations it undergoes during temperature changes. Of all ZrO_2 modifications, the most desirable is the tetragonal modification, as it is stable at high temperatures. However, stability can be maintained at room temperature by adding stabilizers such as yttrium oxide Y_2O_3. The occurrence of the tetragonal modification of ZrO_2 at ambient temperature in materials where its addition is used may lead to the change in their properties due to the phenomenon of toughening transformation of the phase [15].

2. MATERIAL AND METHODS

To obtain composites, the mixtures of powders with 0 %, 5 %, 10 %, 15 %, and 20 % ZrO_2 were weighed and then mixed for 4 hours. The prepared powders were compacted uniaxially, with its diagram shown in **Figure 1**. The compaction pressure was 60 kN whereas the compaction rate was 50 $N \cdot s^{-1}$.

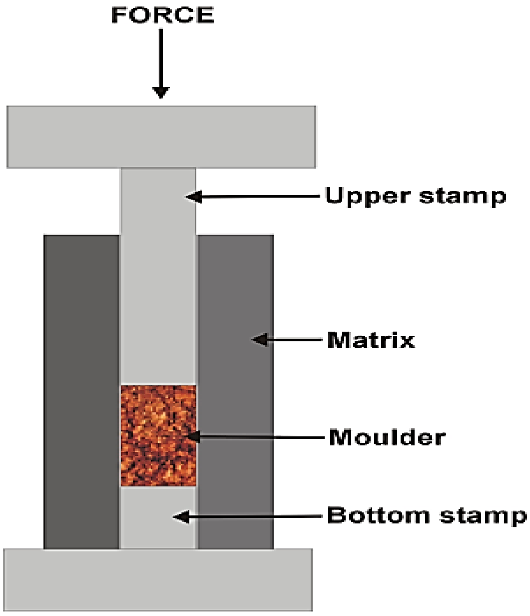

Figure 1 Diagram of uniaxial compaction [16]

The obtained moulded pieces were then subjected to sintering at 1600 °C for five hours. The examinations of the composites included:

- examinations of phase composition using X-ray diffraction (XRD) with a cobalt anode lamp with a wavelength of $\lambda_{Co} = 0.17902$ nm
- microscopic examinations using a scanning electron microscope (SEM)
- hardness measurements using the Vickers method carried out with a loading force of 98.07 N and determination of crack resistance coefficient.
- determination of density and porosity of composites using a hydrostatic balance.

3. RESULTS AND DISCUSSION

The powders used for testing had different particle morphology. In the case of Al_2O_3, the particles were of an irregular shape, while the ZrO_2 powder was characterized by a spheroidal shape, see **Figure 2**.

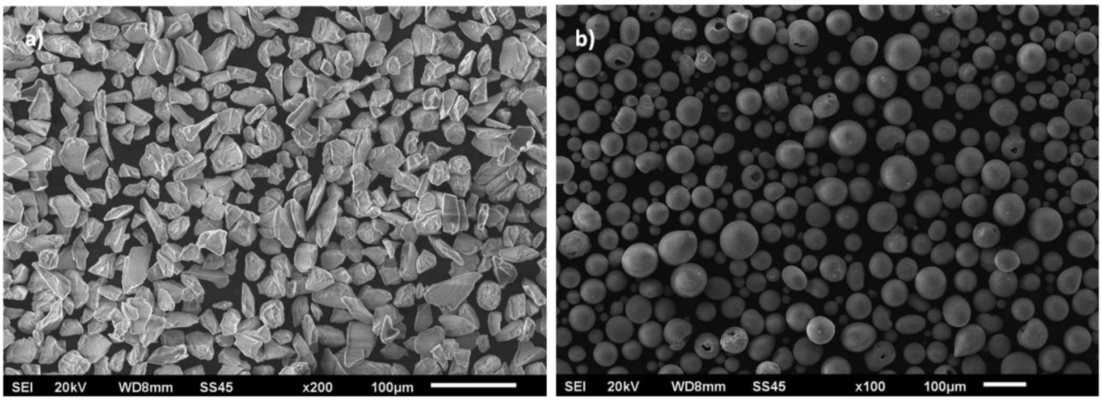

Figure 2 Powders used to make a composite: (a) Al_2O_3, (b) ZrO_2 + 8 mol% Y_2O_3

One of the most important properties of sintered materials is their density and porosity. The results obtained in this study showed that the density of the composites slightly decreased with the increasing content of zirconium phase, which resulted in an increase in porosity of the obtained composites, see **Figure 3**.

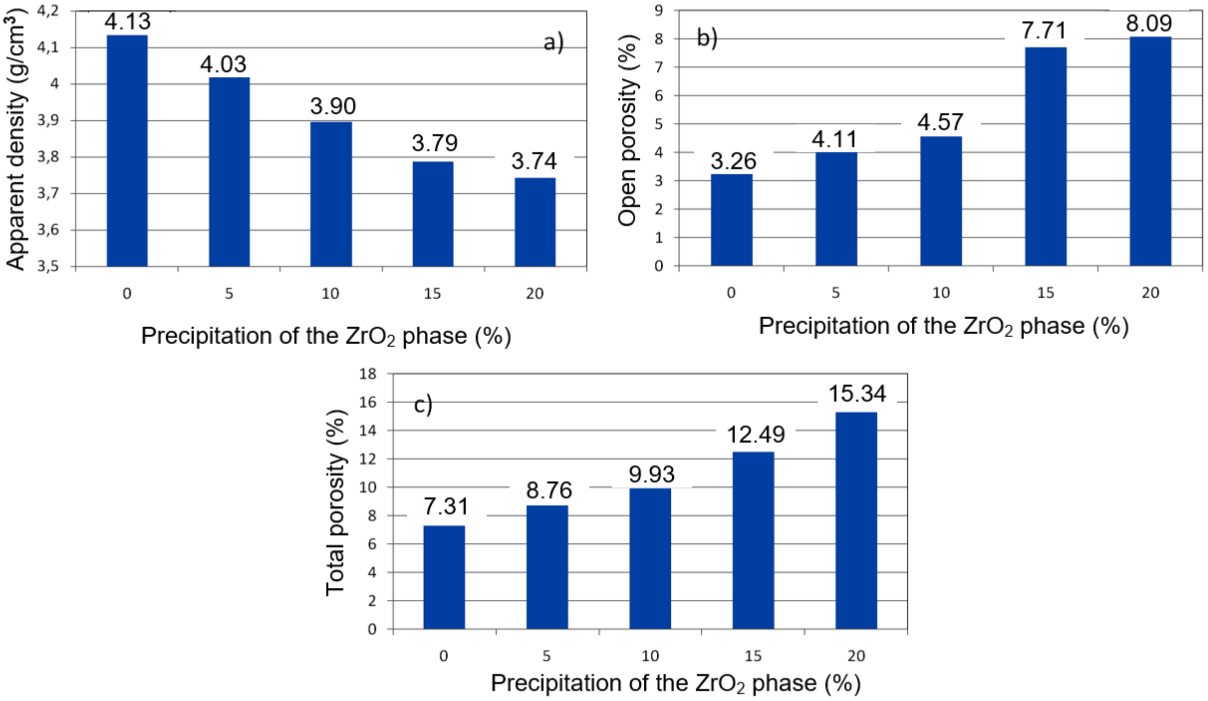

Figure 3 Measurement results: (a) apparent density, (b) open porosity, and (c) total porosity, depending on the ZrO_2 phase content in the composite

The increase in porosity is affected, among other things, by the difference in the shape of the powder particles used to obtain the composite. The investigations using the scanning electron microscope showed that irregular shapes of Al_2O_3 particles form a certain amount of free space between each other already in the matrix. The effect of an increase in the level of porosity of the composite may be additionally intensified at the interface between particles of Al_2O_3 with ZrO_2 with different shape morphology. Furthermore, despite quite a long time of powder mixing, the non-uniform distribution of the ZrO_2 phase particles in the matrix was observed, see **Figure 4**.

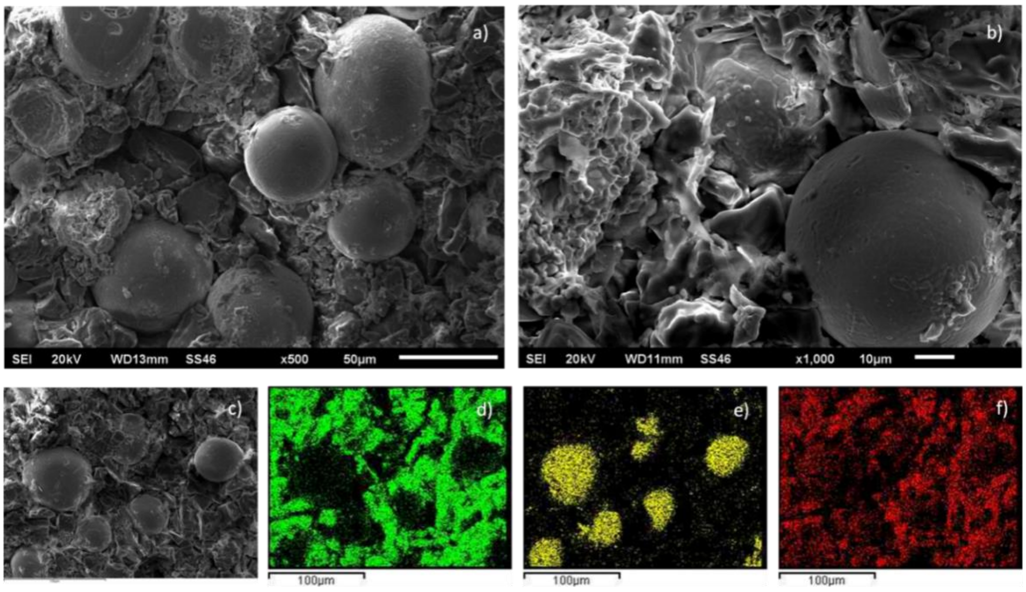

Figure 4 Example microstructure of the composite of Al_2O_3 – 15 % ZrO_2 (a-c), distribution of elements in the composite Al_2O_3 – 15% ZrO_2: d) Al, e) Zr, f) O

The X-ray phase analysis showed that in the obtained composites, no reactions leading to the formation of new phases occurred during the sintering process, but it was found that the addition of a stabilizer in the form of Y_2O_3 allowed for obtaining the tetragonal modification of ZrO_2 at ambient temperature, see **Figure 5**.

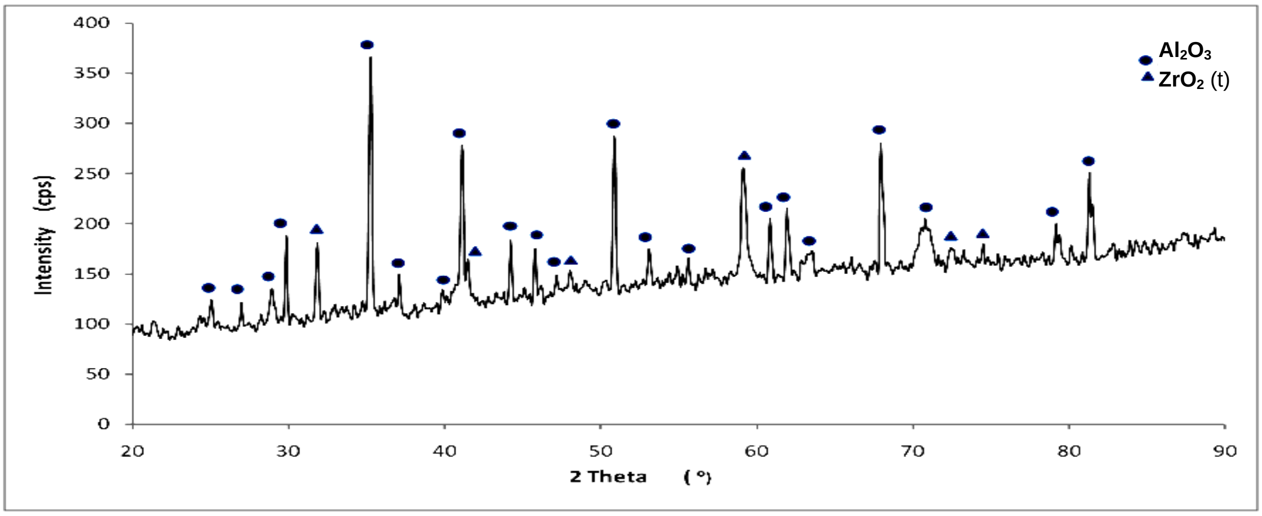

Figure 5 Example of X-ray diffraction obtained for the Al_2O_3 + 15 % ZrO_2 composite

In order to determine the effect of the addition of ZrO_2 on mechanical properties, hardness was measured using the Vickers method, and then K_{IC} crack resistance coefficient was determined based on the analysis of Vickers indentation. There is no unambiguous standardized method of crack resistance assessment with the use of Vickers' pyramid, but the one proposed by Niihare and Antis has been used most frequently [17,18]. The value of Young's modulus of E = 378 GPa for the Al_2O_3/ZrO_2 composite was adopted for calculations as determined and proposed in the paper [19]. The results of hardness and crack resistance tests are shown in **Figure 6**.

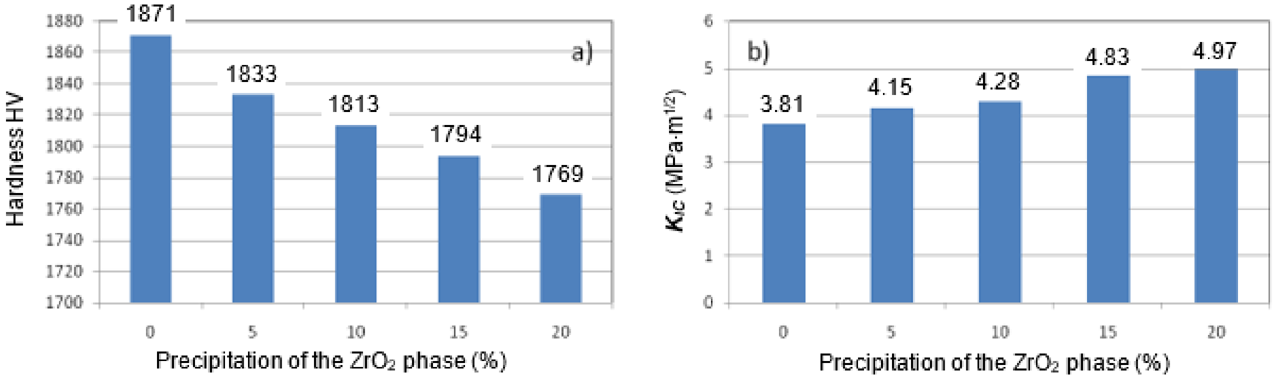

Figure 6 Measurement results: a) hardness, b) crack resistance

The data presented in **Figure 6** show that the increasing content of the ZrO_2 phase causes a decrease in hardness of the composites obtained and may have an effect on e.g. composite porosity. At the same time, a slight increase in crack resistance was observed along with an increase in the amount of the ZrO_2 phase. The increase in the K_{IC} coefficient can be explained by the fact of toughening transformation. This phenomenon consists in that the tetragonal phase ZrO_2 occurs in the structure in a metastable state, whereas the appearing cracks generate stresses, leading to the local transformation of the tetragonal modification into a monoclinic ZrO_2 modification. This transformation generates a local increase in the volume of the inclusions that are likely to block the formed gap. A similar effect was observed in the study [20], where it was found that the addition of 10 mol% ZrO_2 do Al_2O_3 caused a decrease in hardness compared to pure Al_2O_3, with a simultaneous increase in crack resistance.

4. CONCLUSION

The results of the study lead to the following conclusions:

- with the increase in the amount of ZrO_2 phase, the density of the composites decreases,
- the addition of zirconium phase modifies composite porosity,
- the hardness of the composites decreased with an increase in the ZrO_2 phase content; at the same time, an increase in crack resistance coefficient K_{IC} was observed.

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