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Abstract:
The mullite–zirconia composites were prepared by reaction–sintering of alumina and zircon powder. Besides, the slip casting method was employed for fabrication of these composites. Then, the effect of nano–TiO2 addition on the properties of these composites was investigated and the results were compared with micro-sized TiO2. Hence, the physical properties, phase composition, flexural strength and microstructure of these composites after firing at 1600 °C were studied. The results showed that the flexural strength of composite tends to increase with the addition of 0.5 wt.% nano–TiO2. It is attributed to the formation of larger size rod–like ZrO2 and enhanced ceramic bonding between them.

Keywords: Nano–TiO2, Composite, Mullite-zirconia, t–ZrO2, Microstructure

1. Introduction

The mullite–zirconia composites are materials with important technological applications due to their good properties such as toughness, chemical stability, high–creep resistance and good thermal shock resistance. In practice, they are employed in the glass industry and where a high chemical and corrosion stabilities are required [1–6]. The extensive use of mullite–zirconia composites is due to the fact that the zirconia dispersion in the mullite matrix improves the thermo-mechanical properties, leading to toughness by transformation and micro–cracking [5–9]. Hence, several researchers have been worked on the zirconia–mullite system due to its superior thermo–mechanical properties and different processing routes to prepare mullite–zirconia composites have been reported [6–12]. Generally, the reaction-sintering of zircon and alumina is a well–established method for producing mullite–zirconia ceramics. Without sintering aids or stabilizing oxides, the densification is achieved at 1450–1500 °C and the complete mullitization occurs at temperatures near 1600 °C [4–7, 13]. This reaction-sintering is difficult to control because of occurring chemical reactions, such as dissociation of ZrSiO4 and formation of mullite, as well as densification during sintering at various temperatures. Hence, various approaches to solve this problem have been adopted, including addition of sintering aid, reaction bonding and use of highly reactive powders [14]. Therefore, the role of different additives on the physical, thermo–mechanical properties and sintering behavior of mullite–zirconia composites have studied by several researchers [15–24]. It was observed that the additives like CaO and MgO, which do not form a solid solution with the matrix, encourage the sintering rate by a transitory liquid formation [17,18]. Cr2O3

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can form Cr-substituted expanded mullite lattice but, its role on the formation of the crystalline phases further from the amorphous phases is limited [19]. It was envisaged that the dysprosia helps in densification by liquid phase formation as well as by stabilization in tetragonal zirconia state [20]. Addition of CeO₂ enhances the formation of mullite and also increases the fraction of tetragonal zirconia. CeO₂ also forms a solid solution with zirconia [21]. As proved, TiO₂ is the most effective additive for improving sintering and mechanical properties of ceramic materials [22–24]. Therefore, many researchers have studied the system ZrO₂–Al₂O₃–SiO₂–TiO₂ [15 and 22–24]. In zircon/alumina/TiO₂ composition densification occurs through the formation of a transitory liquid phase. Moya et al. [15] found that for the composition located inside the solid solubility limit (0.25 mole TiO₂) the reaction–sintering process takes place in the absence of any liquid phase. Conversely, in the composition with the higher TiO₂ content than the solid solubility limit, the transitory formation of ZrTiO₄ occurs at temperatures around 1300 °C. Besides, TiO₂ has a considerable solubility in the matrix, which affects the densification either by the creation of defects or by the formation of a transitory liquid phase, depending on the amount of addition. It was shown that the addition of TiO₂ results in an important effect on the mechanical properties of the composites because it is the controlling parameter for the zirconia grain growth [15, 25, 26]. Ebadzadeh et al. [24] found that the addition of TiO₂ to mullite–zirconia composites prepared from different alumina sources leads to change of reaction sintering, densification and microstructure which can alternately alter the formation temperature and retention of t–ZrO₂ phase in these composites. The tetragonal phase was not detected in the mixture of α-alumina and zircon sintered below 1500 °C, while by adding TiO₂ due to the extent of reaction t–ZrO₂ peaks appeared at low sintering temperature (e.g.1400 °C) [23,24]. On the other hand, the use of nanometer oxides as additions to raw material mixtures or as nanometer starting material for the production of ceramic materials is reported in a wide range of literature since several years [27–30]. Nanometer materials are very reactive due to their better homogenization, high specific surface area and therefore the sintering starts at lower temperatures. Consequently, in the present work the influence of the nano–TiO₂ addition on the properties of mullite–zirconia composites is investigated and their results were compared with micro–TiO₂. For this reason, physical properties, phase composition, flexural strength and microstructure of these composites after firing at 1600 °C were studied. Besides, slip casting method was employed for fabrication of these composites due to obtain fine and homogeneous microstructures free of agglomerates and packing variations in the green composites.

2. Experimental procedure

2.1. Raw materials and composition

Starting powders were commercially available alumina (α–Al₂O₃, HTM 10 Fiberona Co., India) and zircon (ZrSiO₄, Mahlwerke Kreutz, Mikron, Germany) with average size particles 2.6 and 6.5 μm respectively. Table I shows the chemical composition of these starting materials given by the producers. The nano–titania and micro–titania powders are the product of AEROXIDE Co. (P25) and Tioxide Co. (A–HR), respectively. The chemical composition and physical parameters of used nano–titania and micro–titania are shown in Table II For the study of nano–titania addition on the properties of these composites, the TiO₂ addition was used in 0–1 %wt. range.
The mullite–zirconia composites were prepared from a mixture of 54.5 wt.% zircon and 45.5 wt.% alumina with respect to the stoichiometric ratio. The stoichiometric alumina to zircon weight ratio used corresponds to the stoichiometric \( \alpha-\text{Al}_2\text{O}_3/\text{ZrSiO}_4 \) mole ratio that forms mullite and zirconia after complete conversion at high temperature. The aqueous slips were prepared with 65 wt.% powder, 35 wt.% \( \text{H}_2\text{O} \) and different contents of deflocculant (Dolapix CE64, Zschimmers and Schwartz), with regard to the additive used at pH 9.1–9.2.

### 2.2. Preparation of samples

The starting powder mixes were ground and homogenized in a laboratory planetary mill for 2 h with distilled water, using corundum balls as the grinding bodies. The size distribution of slip particles was analyzed by a laser particle diameter analyzer (Honeywell Microtrac X–100) after milling. In this relation, 1.1 \( \mu \text{m} \) was measured as the mean particles size of milled slip. Generally, the changes in particles size distribution of slip have a direct effect on the rheological character of the slip [13, 14]. Therefore, the rheological character of the slip can affect with addition of micro–\( \text{TiO}_2 \) and nano–\( \text{TiO}_2 \). For this reason, slip cast bars were consolidated in a plaster mold from the optimal slip condition for each composition such as deflocculant content. On the other hand, the slips containing upper than 1 wt. % \( \text{TiO}_2 \) were not cast due to unacceptable rheological characteristics. Hence, the prismatic bars \( 7.5 \text{ mm} \times 7.5 \text{ mm} \times 50 \text{ mm} \) were produced from well dispersed suspensions by slip casting in a plaster molds. Dried probes were fired in an electric furnace with a heating rate of 10 °C/min up to 1600 °C for 5 h.

### 2.3. Test methods

The fired samples were tested for bulk density (B.D) and apparent porosity (A.P). The flexural strength at room temperature was measured on the fired rectangular bars by three-point bending method using an Instron–1185 universal testing machine. The most commonly used method of characterizing the scatter of ceramic strength is to apply Weibull statistical theory. On the other hand, at least 30 specimens should be tested for Weibull statistical analysis which, this might be impossible in such scientific studies. Therefore, the five samples
were tested to obtain the average strength and standard deviation. Crystalline phases present in fired bodies were determined by X-ray diffraction (Philips 3020, Cu Kα radiation and Ni filter at 40 kV and 20 mA). The microstructures of fired composites were studied by scanning electron microscopy (Cambridge S–360 SEM at 10–20 kV) on fracture surfaces.

3. Results and discussion
3.1. The effect of nano–TiO₂ addition on the physical properties

The effect of micro–TiO₂ addition on the physical properties of mullite–zirconia composite such as; B.D and A.P is shown in Fig. 1.

![Fig. 1. The effect of micro–TiO₂ addition on the B.D and A.P of fired mullite-zirconia composite.](image1)

The effect of nano–TiO₂ addition on the B.D and A.P of fired mullite-zirconia composite is shown in Fig. 2.

![Fig. 2. The effect of nano–TiO₂ addition on the B.D and A.P of fired mullite-zirconia composite.](image2)
With respect to results of Fig. 1, the addition of micro-TiO\(_2\) leads to increasing of A.P and then, decreasing of B.D. Although, TiO\(_2\) is the most effective additive for the improving of densification but, its role on the further densification of these composites is limited. Generally, bulk density depends on the formation of pores and development of different mineralogical phases in the sintered compacts \[19\]. Therefore, the increasing of A.P with TiO\(_2\) addition may be due to several factors like precipitated phase, secondary crystallization, lattice expansion, micro-crack formation etc. The effect of nano–TiO\(_2\) addition on the B.D and A.P of mullite-zirconia composite is shown in Fig. 2.

The results of Fig. 2 show that the bulk density of mullite-zirconia composites is decreased with the addition of nano–TiO\(_2\). Comparing Fig. 1 with Fig. 2, it can be found that variations of the densification are similar but, the nano–TiO\(_2\) addition has higher effect on the increasing of A.P.

### 3.2. The effect of nano–TiO\(_2\) addition on the phase composition

The XRD results of fired mullite-zirconia composites containing different amounts of micro–TiO\(_2\) are shown in Fig. 3.

**Fig. 3.** The XRD results of fired mullite–zirconia composites containing different amounts of micro–TiO\(_2\).

- ●: Mullite, ▲: m-Zirconia, ▼: t-Zirconia, □: Zircon, ■: Courundum

With respect to these results, the mullite phase exists in addition to monoclinic zirconia (m–ZrO\(_2\)) and tetragonal zirconia (t–ZrO\(_2\)) phases at all compositions. Besides, some zircon and alumina phases as raw materials exist. Generally, zircon disassociates to yield ZrO\(_2\) and amorphous SiO\(_2\) on heating. The amorphous SiO\(_2\) softens with increasing temperature and starts to dissolve Al\(_2\)O\(_3\) to form an amorphous aluminosilicate glass. Nucleation of mullite phase takes place after a critical alumina concentration is exceeded in the glass phase. On cooling through the transformation temperature (\(\sim 1100 \, ^\circ\)C), ZrO\(_2\) crystals transform from tetragonal to monoclinic form. On the other hand, the formation of t-ZrO\(_2\) at composition implies the existence of some particles below the critical size for transformation \[14, 26\]. On the other hand, the results reveal that the content of t–ZrO\(_2\) is considerably decreased with increasing of micro–TiO\(_2\) amount. Simultaneously, the content of m–ZrO\(_2\) is increased with decreasing of t–ZrO\(_2\) amount. This can be attributed to the decreasing of tetragonal→monoclinic transformation temperature by the formation of solid
solution of TiO_2 in t–ZrO_2 and the enlargement of zirconia particles higher than the critical size of tetragonal–monoclinic transformation [24, 31 and 32]. From the results of Fig. 1 and Fig. 3, one would expect to distinguish a connection between t–ZrO_2 retention and density of the mullite–zirconia composite. As can be seen, the higher compact composites obtain the higher t–ZrO_2 concentration. Generally, the martensitic transformation in zirconia, t–ZrO_2 to m–ZrO_2, is associated with 4%–5% volume expansion and then, micro-cracks formation, which possibly lowers the density for conventionally processed composites [33]. Besides, the results show that the content of zircon as remaining raw materials in the composite is decreased with increasing of micro–TiO_2 content. Ebadzadeh et. al [23 and 24] found that the dissociation of zircon and the formation of zirconia occur at lower temperatures in compositions containing TiO_2. In addition, the reaction between alumina and zircon enhances by introducing TiO_2 and the dissociation of zircon and formation of zirconia are shifted to the lower temperatures. The XRD results of fired mullite–zirconia composites containing different amounts of nano–TiO_2 are shown in Fig. 4.

![Fig. 4. The XRD results of fired mullite–zirconia composites containing different amounts of nano–TiO_2.](image)

As it can be seen from the results of Fig. 4, changes in the composition of phases with addition of nano–TiO_2 are similar to the results of Fig. 3. But, the small peak of rutile phase is detectable by XRD due to the high surface area of nano–TiO_2 particles. The high surface area of nano–TiO_2 particles can lead to agglomeration of these particles. Hence, the reaction of TiO_2 particles with other particles doesn’t take place. Generally, TiO_2 transforms from the anatase phase to rutile form with increasing of temperature. Therefore, nano–TiO_2 appears as rutile phase. On the other hand, the higher reduction of t–ZrO_2 is occurred with addition of nano–TiO_2 in comparison with micro–TiO_2 (Fig.3). This can be explained with respect to the replacement of t–ZrO_2 peak concerning to [101] plane. In Fig. 5, the effect of TiO_2 addition on the replacement of t–ZrO_2 peak concerning to [101] planes is shown. Generally, with replacement of Zr^4+ cation by Ti^4+ in the structure, the zirconia peak can shift to higher angels. Hence, Fig.5 indicates further evidence of the enhance effect of TiO_2 in the replacement of t–ZrO_2 peak concerning to [101] plane. On the other hand, Fig. 5 reveals, addition of nano–TiO_2 leads to the further replacement of t–ZrO_2 peak.

On the other hand, TiO_2 can enter into mullite grain by solid solution formation without much change in composition or amount of viscous phase. The substitution of Al^3+ by Ti^4+ probably in the octahedral sites of mullite crystals can cause aluminum ion vacancy...
according to following reaction:

\[(1 - X) \text{Al}_2\text{O}_3 + X \text{TiO}_2 \rightarrow 2(1 - X) \text{Al}_{1\text{Al}} + X\text{Ti}_{1\text{Al}} + X/3 \text{V}_{1\text{Al}}^{\text{III}} + (3 - X) \text{O}_2\]

According to this defect model, the electrical compensation is accomplished by the vacancy defect on the aluminum site.

Therefore, dissolution of TiO\(_2\) in the mullite structure can produce cations vacancies which, leads to increasing of diffusion and then, further formation of mullite. Besides, it reported that TiO\(_2\) can accelerate mullite formation by lowering the glass viscosity [36]. On the other hand, the aluminum ions vacancies increase with the TiO\(_2\) addition. The upper limit of solubility is controlled by the radius and the valence state of the cations. It found that when the mass fraction of TiO\(_2\) additives is less than 5\%, most of TiO\(_2\) particles are dissolved into ZrO\(_2\), and a small amount of TiO\(_2\) is dissolved into mullite grains [18, 34 and 35]. Besides, with replacement of Al\(^{3+}\) cation by Ti\(^{4+}\) in the structure, the mullite peaks can shift to lower angels. For further investigation, the replacement of mullite peaks concerning to [120] and [210] planes were studied. In Fig 6, the effect of TiO\(_2\) addition on the replacement of mullite peaks concerning to [120] and [210] planes are shown.

**Fig. 5.** The effect of TiO\(_2\) addition on the replacement of t–ZrO\(_2\) peak concerning to [101] plane.

**Fig. 6.** The effect of TiO\(_2\) addition on the replacement of mullite peak concerning to [120] and [210] planes.
Fig. 6 clearly shows the mullite peaks were shifted to lower angles by TiO$_2$ addition. Besides, addition of nano–TiO$_2$ leads the further replacement of mullite peaks in comparison with micro–TiO$_2$. Therefore, nano–TiO$_2$ can produce higher cations vacancies which, lead to higher increasing of diffusion and then, further formation of mullite.

3.3. The effect of nano–TiO$_2$ addition on the mechanical properties

The flexural strength of the fired mullite–zirconia composites as a function of TiO$_2$ amount are shown in Fig. 7.

![Image of Fig. 7](image-url)

**Fig. 7.** The flexural strength of the fired mullite–zirconia composites as a function of TiO$_2$ amount.

With respect to these results, with increasing of micro–TiO$_2$ amount the mechanical strength of the fired mullite–zirconia composite is decreased. Generally, several factors can affect the strength of ceramic composites, including bonding type, amount of porosity and phases developed during sintering [19]. With respect to physical properties results (Fig. 1), the A.P of fired composite tends to increase with increasing of micro–TiO$_2$ amount. Hence, the mechanical strength can decrease with increasing of A.P. On the other hand, it is known that the change from tetragonal to monoclinic generates micro–cracks in the microstructure that will negatively affect the flexural strength of the composite [26]. Therefore, with increasing of this polymorphic change and then, enhancing of micro–cracks amount the flexural strength can decrease. Comparing Fig.3 with Fig.7, it can be found that the reduction of t–ZrO$_2$ at room temperature in mullite matrix leads to the decreasing of strength. Oppositely, with increasing of nano–TiO$_2$ amount up to 0.5 wt. %, flexural strength is increased. This can be related to higher direct bonding of zirconia and mullite particles due to higher cations vacancies in the structure. On the other hand, flexural strength is decreased with further increasing of nano–TiO$_2$ amount due to formation of further micro–cracks and nano–TiO$_2$ agglomeration. However, there is a considerable scatter of the strength values. Generally, the strength of ceramic materials is statistically distributed according to the flaw size distribution in the material. Therefore, brittle fracture depends on the probability of a material to contain a flaw equal to or larger than the critical size for a given applied stress. Besides, fracture depends on the stressed area or the volume of the material, since larger areas or volumes increase the probability to present a critical flaw [37]. Therefore, the strength of ceramic composites depends on a wide range of random factors which results in a
considerable scatter of the experimental data.

3.4. The effect of nano–TiO₂ addition on the microstructure

The microstructures of fired mullite–zirconia composite without TiO₂ are presented in Figs. 8 and 9.

![SEM photomicrograph of fired mullite–zirconia composite without TiO₂.](image1)

**Fig. 8.** SEM photomicrograph of fired mullite–zirconia composite without TiO₂.

![SEM photomicrograph of fired mullite–zirconia composite without TiO₂.](image2)

**Fig. 9.** SEM photomicrograph of fired mullite–zirconia composite without TiO₂.

As shown, the microstructure of these composites comprises of bright zirconia grains which are uniformly distributed throughout the darker mullite matrix. Generally, zirconia has higher atomic number compared to aluminum and silica. So it appears clearly from its surrounding mullite matrix under SEM and shows a brighter image (white). It is observed that the mullite grains are acicular probably due to a considerable amount of glassy phases present during sintering. Two types of zirconia grains are present which, one is inter–granular zirconia located between the mullite grains and the other one is intra–granular zirconia present within mullite matrix. Many researchers [1, 6 and 31] are shown that intra–granular particles as spherical shape are tetragonal ZrO₂, while the inter–granular ZrO₂ is mainly
monoclinic. Rounded morphology of the ZrO₂ is due to the transient liquid phase. The stabilization of tetragonal zirconia in the composite microstructure is successfully accomplished by the uniform distribution of fine zirconia particles. The other obvious characteristic in these Figures is that there are very fine micro-cracks in the microstructure as a consequence of the ZrO₂ martensitic transformation. These micro-cracks will give their contribution to the improvement of the fracture toughness by micro-cracking toughening mechanism. The microstructures of fired mullite–zirconia composites containing 1 wt.% micro–TiO₂ are shown in Figs. 10 and 11.

**Fig. 10.** SEM photomicrograph of fired mullite–zirconia composite containing 1 wt.% micro–TiO₂.

**Fig. 11.** SEM photomicrograph of fired mullite–zirconia composite containing 1 wt.% micro–TiO₂.

The microstructural analysis of mullite-zirconia compositions containing micro–TiO₂ shows lower amounts of t–ZrO₂ particles in comparison with compositions without TiO₂. Besides, the mullite–zirconia composition containing TiO₂ shows enlarger ZrO₂ particles in comparison with composition without TiO₂, which is the reason for the reduction of t–ZrO₂ concentration. Therefore, TiO₂ addition is promoted the growth of ZrO₂ grains. The similar
behavior has been reported by Hong and Messing [38, 39]. On the other hand, microstructural evaluations of mullite–zirconia compositions show that the amount of micro–cracks in the microstructure tends to increase with addition of micro–TiO$_2$ which leads to decrease of mechanical strength (Fig. 7). The microstructures of fired mullite–zirconia composites containing 0.5 wt.% nano–TiO$_2$ are shown in Figs. 12 and 13.

![SEM photomicrograph of fired mullite–zirconia composite containing 0.5 wt.% nano–TiO$_2$.](image)

**Fig. 12.** SEM photomicrograph of fired mullite–zirconia composite containing 0.5 wt.% nano–TiO$_2$.

![SEM photomicrograph of fired mullite–zirconia composite containing 0.5 wt.% nano–TiO$_2$.](image)

**Fig. 13.** SEM photomicrograph of fired mullite–zirconia composite containing 0.5 wt.% nano–TiO$_2$.

From the scanning electron micrograph of the mullite–zirconia compositions containing nano–TiO$_2$ it was observed that enlarger ZrO$_2$ particles are formed in comparison with compositions containing micro–TiO$_2$. Besides, lower amounts of t–ZrO$_2$ particles in microstructure can be seen. In addition, some of the formed ZrO$_2$/ZrO$_2$ grains closed to each other and formed long necks and, consequently, change to one grain (grain growth). This means that ZrO$_2$/ZrO$_2$ grains form from sintering by direct bonding. Besides, the microstructure of the formed ZrO$_2$/ZrO$_2$ grains is uniform in grain size. Accordingly, the
properties can be improved because of this type of sintering and packing of formed m–ZrO₂. Hence, the higher nano–TiO₂ addition leads a larger size rod-like ZrO₂ to be developed, which has positive effect on mechanical properties of these composites. This is caused by a decrease in viscosity of the liquids formed, i.e., amorphous SiO₂ and amorphous mullite due to nano–TiO₂ addition. This liquid helps the phases formed to rearrange. Figs. 14 and 15 show the microstructures of fired mullite–zirconia composites containing 1 wt.% nano–TiO₂.

![SEM photomicrograph of fired mullite–zirconia composite containing 1 wt.% nano-TiO₂.](image1)

**Fig. 14.** SEM photomicrograph of fired mullite–zirconia composite containing 1 wt.% nano-TiO₂.

![SEM photomicrograph of fired mullite-zirconia composite containing 1 wt.% nano–TiO₂.](image2)

**Fig. 15.** SEM photomicrograph of fired mullite-zirconia composite containing 1 wt.% nano–TiO₂.

With microstructural evaluation of mullite-zirconia composite containing 1 wt.% nano-TiO₂, one can see that the agglomerates of fine particles exist. The EDX analysis shows that these agglomerates are rutile phase. The nano–TiO₂ particles have high surface area that leads to attraction forces between these particles. Therefore, the formation of agglomerates takes place in slips containing nano–TiO₂ particles. Besides, the high amounts of porosities with large size exist between particles, which have negative effect on mechanical properties.
of these composites.

4. Conclusions

The mullite–zirconia composites containing different amounts of micro and nano–TiO₂ particles were prepared by reaction-sintering of alumina and zircon powder. Besides, the slip casting method was employed for fabrication of these composites. Then, the physical properties, phase composition, flexural strength and microstructure of these composites after firing at 1600 °C were evaluated. This study confirmed the strong influence that nano–TiO₂ particles exert on the microstructure and then, flexural strength of mullite–zirconia composites. Nano–TiO₂ additive has a considerable solubility in the matrix and then, can create the ionic defects. Meanwhile, the bulk density of sintered samples falls with increase of TiO₂ amount. On the other hand, the amount of t–ZrO₂ phase in the composition is decreased with increasing of TiO₂ amount due to solid solution formation and then, ZrO₂ grain growth. Besides, micro–TiO₂ addition reduces the flexural strength as a result of increasing micro–cracks formation. But, the flexural strength of composite tends to increase with the addition of 0.5 wt.% nano–TiO₂. The microstructural analysis showed that nano-TiO₂ addition leads to development of larger size rod-like ZrO₂ and formation of necks between ZrO₂ grains. This leads to enhanced ceramic bonding between ZrO₂ grains, which has positive effect on mechanical properties of these composites.

5. References


Садржај: Композити мулт-цирконијум су припремљени реакционарним синтеровањем алумине и праха цирконијума. Поред тога, метод клизећег ливења је употребљен за добијање ових композита. Након тога, изучавани су ефекти додатка нано–TiO2 на својства ових композита и резултати су поређени са додатком микро–TiO2. Испитивана су физичка својства, фазни састав, отпорност на савијање и микроструктура након синтеровања на 1600 °C. Резултати су показали да отпорност на савијање композита расте са додатком 0,5 wt% нано–TiO2. То је приписано формирању већих штапићастих зрна ZrO2 и побољшаним везама између зrna.
Кључне речи: нано–TiO2, композити, мулт-цирконијум, m–ZrO2, микроструктуре