Colloid and Surface Science
2017; 2(1): 1-5
http://www.sciencepublishinggroup.com/j/css
doi: 10.11648/j.css.20170201.11

Surface Properties Modification of Zirconia Toughened Alumina by Using Titania Additives

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To cite this article:
Abdullah Al Mahmood, Abdul Gafur, Emdadul Hoque. Surface Properties Modification of Zirconia Toughened Alumina by Using Titania Additives. *Colloid and Surface Science.* Vol. 2, No. 1, 2017, pp. 1-5. doi: 10.11648/j.css.20170201.11

Received: December 17, 2016; **Accepted**: January 3, 2017; **Published**: January 19, 2017

Abstract: Aluminium oxide (Al_2O_3) were mixed with multicomponent such as Zirconia (ZrO_2) and Titania (TiO_2). The aim of this thesis was to investigate the effect of TiO_2 addition to Zirconia Toughened Alumina (ZTA). The physical, mechanical and microstructural behavior was characterized in this research. The percentage of Alumina was 83-85% and Zirconia used a fixed percentage of 15% for all samples where the percentages of Titania were used 0-1.5%. Each composition was weighted, mixed in pot mill with alcohol medium, heated, again mixed with polyvinyl alcohol binder and pressed using hydraulic press under 160 MPa into 10 mm pellets. The pellets were pre-sintered at 600°C for 2 hours and then sintered at 1450°C under pressureless condition. Bulk density, porosity and other properties of the samples were measured using standard procedure. Vickers hardness and fracture toughness of the sintered samples were measured using the Vickers indentation method. Phase analysis and microstructural analysis were carried out by XDR and SEM. By comparing ZTA ceramics with and without addition of MgO and TiO_2 the results found an increasing of fracture toughness and hardness of the materials. The XRD patterns of ZTA samples containing 15 wt% of ZrO_2, 0.5-1.5 wt% TiO_2 and sintered at 1450°C for 2 hours indicated that α-Al_2O_3, t -ZrO_2, and TiO_2 are the crystalline phases present in the composite samples. The microstructures show highly homogeneous microstructures without agglomerates, pores or abnormally grown alumina grains with less porosity when compared to only Zirconia Toughened Alumina. The approach adopted in the present study may provide an alternative to design Al_2O_3-ZrO_2-TiO_2 composites with improved mechanical properties.

Keywords: Alumina (Al_2O_3), Zirconia (ZrO_2). Titania (TiO_2)

1. Introduction

Zirconia Toughened Alumina (ZTA) provides mechanical strength and wear resistance and is used as the ideal intermediate solution between Zirconia and Alumina [1]. The main advantage of Zirconia Toughened Alumina (ZTA) is the additional strength and toughness over alumina [2]. Zirconia Toughened Alumina (ZTA) ceramics offer an exceptional performance to price ratio. Like Yttria Stabilized Tetragonal Zirconia Polycrystal (YTZP), Zirconia Toughened Alumina (ZTA) belongs to a family of ceramics that have a toughening mechanism due to transformation of the crystal structure under an applied stress. Their microstructures have been tailored to produce a significant enhancement of structural properties over basic alumina materials.

These improved properties are the result of a combination of factors, most significantly a phenomenon known as "transformation toughening". ZTA's carefully tailored microstructure with a uniform dispersion of YTZP particles in an alumina matrix results in a more fracture resistant structure than alumina alone [3]. Regardless of their advantages, ceramic materials exhibit very low toughness which eventually limits their overall applications [4–8]. The use of ZrO_2-based ceramics is one of the possible alternatives to circumvent the limitation of low fracture toughness [9, 10].

Most investigators concluded that a optimum amount of zirconia should be in the turn of 8-15 vol% (in ZTA) considering a trade-off between improvements in fracture toughness, decrease grain size and lower hardness [11-12]. Beside all advantages of using ZTA, it is difficult to have ZTA with high density and always the reported bulk density of ZTA is lower than mono phase ceramic of alumina and zirconia which lead to lower definitive properties in applied

engineering in the compare with research studies [12].

There exist many possible additives, mainly metal oxides which, with various efficiencies, act as sintering aids and grain growth inhibitors for alumina and in limit cases they are used for ZTA. One of the most used and also one of the most effective is TiO_2. The addition of TiO_2 promotes the sintering and grain growth of Al_2O_3 [13]. This advantage has been considered to be a result of the enhanced diffusivity due to the increasing concentration of the Al^{3+} vacancies which is generated by the Ti^{4+} substituting for Al^{3+}. As the quantity of additive approaches 0.15 – 0.35 mol%, i.e. the solubility limit, a further increase in the densification rate and grain growth can be observed. The grain growth of the Al_2O_3 and ZTA is encouraged by TiO_2 which is an important sintering additive, bringing about a completely dense and finer homogeneous structure.

The current research has been done to understand the role of TiO_2 in ZTA ceramic composites. The amount of TiO_2 were varied from 0 wt % to 1.5wt% with fixed amount of ZTA as the matrix. Hence, this study aims to enhance the surface properties and microstructural properties of TiO_2 doped Zirconia Toughened Alumina (ZTA) Ceramic Composites.

2. Experimental Procedure

Several compositions were prepared by mixing Al_2O_3, ZrO_2, and TiO_2 powders. The percentage of the ZrO_2 powder was fixed at 15wt% where the percentages of TiO_2 were varied from 0-1.5wt%. Al_2O_3 used as the matrix material.

At first ball milling was carried out for 24 hours in pure ethanol media in a motor driven ball mill. After mixing the powder mixture was removed with the help of a strainer and subsequently dried in an oven for 24 hours at 100°C. Hand milling was done for several hours to obtain a homogeneous distribution.

The green body (bulk sample) in the form of tablet of about 10 mm in diameter dimension was prepared by uniaxial compaction procedure. Before compaction, polyvinyl alcohol (PVA) binder was mixed with the powder in a mortar pestle to provide some green strength for subsequent handling. During green sample preparation, the right amount of binder and pressure selection was of high importance, otherwise the edge of the sample could be broken during handling or complexity may arise during ejection of the green sample from the die. In the present work, 1 drop of binder for every 0.8 gm of powder and 160 MPa of pressure for each sample were used. The pressing time used was 1 minute for every sample to maintain uniformity in thickness.

After the preparation of every sample the die was cleaned properly since any remaining powder inside the die may impart some resistance in the die movement for the subsequent pressing and the load distribution may not be uniform which results in breaking of the sample. The pellets were sintered at 1450°C for 4 hour at a heating rate of 5°C/minute in a pressureless condition.

Surface hardness of the sintered samples was measured by the Vickers indentation technique. the Vickers hardness was measured by taking the average of five different readings for each sample. The polished sintered samples were subjected to HV 50 kgf for 15 sec while the bulk density of the samples were calculated based on the Archimedes principle. A Field Emission Scanning Electron Microscope (FE SEM) was used to observe microstructures of the samples and their grain growth. The electrical conductivities of all test samples used in this research project were usually poor. To avoid this, the surfaces of all samples were coated by gold sputtering technique. The samples were bonded with conductive carbon tape on an aluminium stub. Then the stub and surface was further connected with highly conductive copper foil. For SEM analysis, 5 kV accelerating voltage was used. Under FE-SEM, various grain growths were observed and they were photographed.

3. Results and Discussion

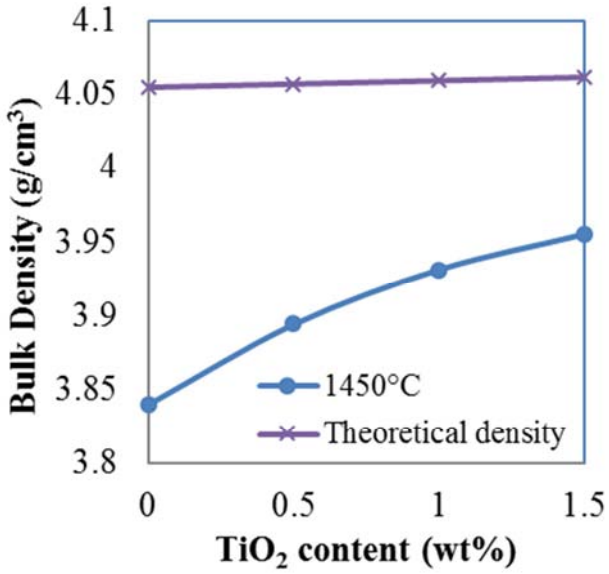

Figure 1. *Density variation with Titania content.*

Figure 1 shows that, with the addition of TiO_2, it increased the bulk density at sintering temperatures of 1450°C. TiO_2 have the ability to hinder the unusual grain growth of Al_2O_3 [14]. It also shows the pinning effect. As a result, initially addition of TiO_2 till 1wt% caused a rapid increment of the density. After that value the density increased very little amount at the higher sintering temperature where at lower temperature it increased comparatively at a higher rate. The highest value of the density found 3.955 g/cc when the TiO_2 was 1.5% and the sintering temperature was 1450°C.

Figure 2 shows the effect of TiO_2 addition to the porosity of ZTA ceramics. The lowest value of porosity (0.44%) was found when the Tio_2 addition was maximum (1.5wt%). At this point the density of the material was highest. So it is proved that, when the porosity is minimum, the density will be maximum. With the addition of TiO_2 to ZTA in a proper amount the porosity will be the minimum. Upto 1wt% TiO_2 addition the porosity reduced sharply where after 1wt%, it reduced slowly.

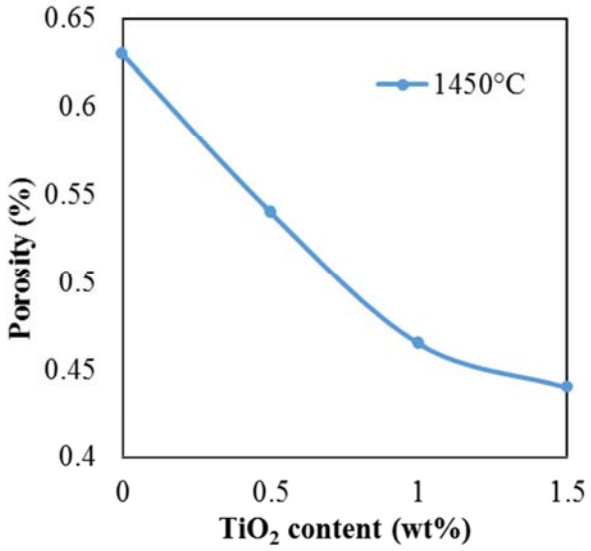

Figure 2. *Porosity variation with Titania content.*

Figure 3 represents the results of Vickers hardness for ZTA–TiO_2 ceramic composite with different TiO_2 contents. Vickers hardness was observed to gradually increase from 1389 HV (0 wt% TiO_2) to 1596 HV (1wt% TiO_2), indicating an improvement of approximately 14.9%. The results of Vickers hardness are directly related to the results of the bulk density, as shown in figure 1. After 1 wt% TiO_2 the value of the hardness increased slowly and the value is 1596 HV, which was found for TiO_2 content 1wt% and at the sintering temperature of 1450°C. The highest value of Vickers hardness, 1617 HV (1.5wt% TiO_2), also corelate with the highest density value (3.955 g/cm^3). The increase in hardness with the increasing TiO_2 content is related to an increase in densification

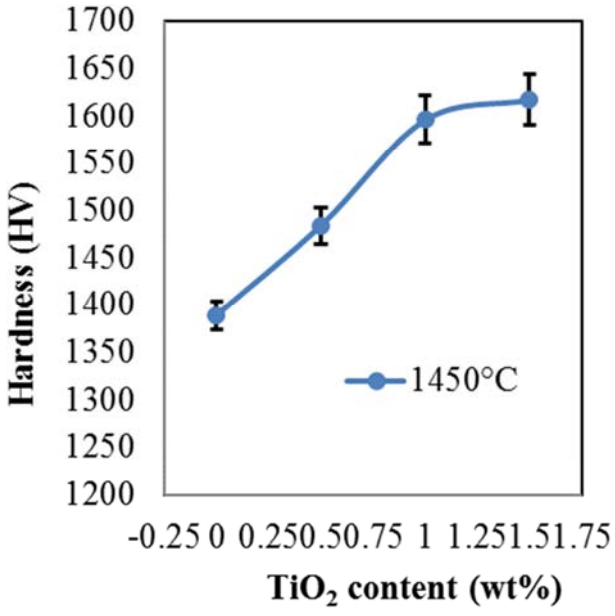

Figure 3. *Hardness variation with Titania content.*

Figure 4 shows the Comparison of XRD pattern of ZTA containing 0wt% Titania with raw alumina, zirconia and titania where Figure 5. Represents the Comparison of XRD pattern of ZTA containing 1.5wt% Titania with raw alumina, zirconia and titania. Both patterns showing the existence of alumina, zirconia and titania grains. The intensity of the peaks for the sample containing 1.5wt% titania is quite higher than that of the sample containing 0wt% titania. In both Figureures with the color variation. it represents the various components diffraction patterns.

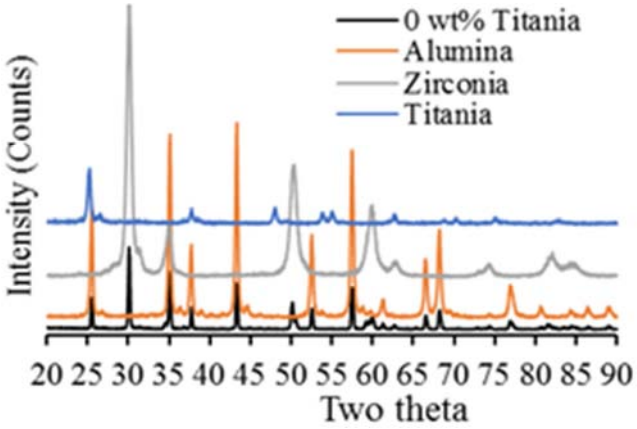

Figure 4. *Comparison of XRD pattern of ZTA containing owt% Titania with raw alumina, zirconia and titania.*

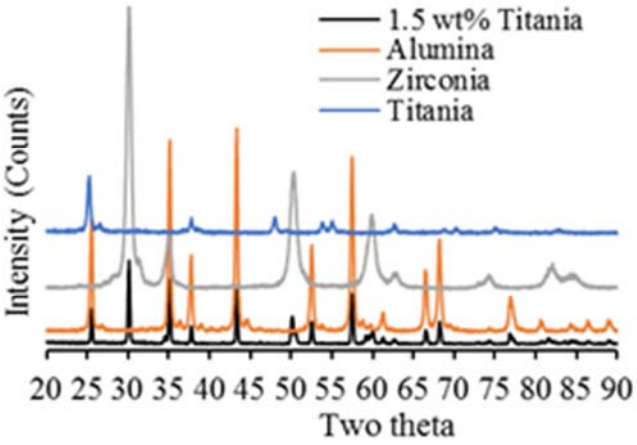

Figure 5. *Comparison of XRD pattern of ZTA containing 1.5wt% Titania with raw alumina, zirconia and titania.*

Figure 6 represents the micrographs of ZTA ceramics containing only alumina 85wt% and zirconia 15wt%. In Figure 7 representing the ZTA samples with 1wt% titania also used where in Figure 8 the ZTA sample contains 1.5wt% titania. By comparing all three Figureures it is clearly understood that, with the addition of more titania the porosity reduced. The titania content participating with zirconia content for hindering the unusual grain growth of alumina. This titania contents are located on the grain boundary of alumina. Titania content can show the pinning effect on the boundary, hence the smaller size alumina grains are obtained.

This refinement of microstructure by addition of titania would effect on the physical properties like density, porosity. The surface hardness was also increased due to the porosity

reduced by titania content addition to the ZTA. As there is a relationship with the density, porosity to other mechanical properties as well as wear resisting properties. Hence, those properties will also be enhanced by titania additive.

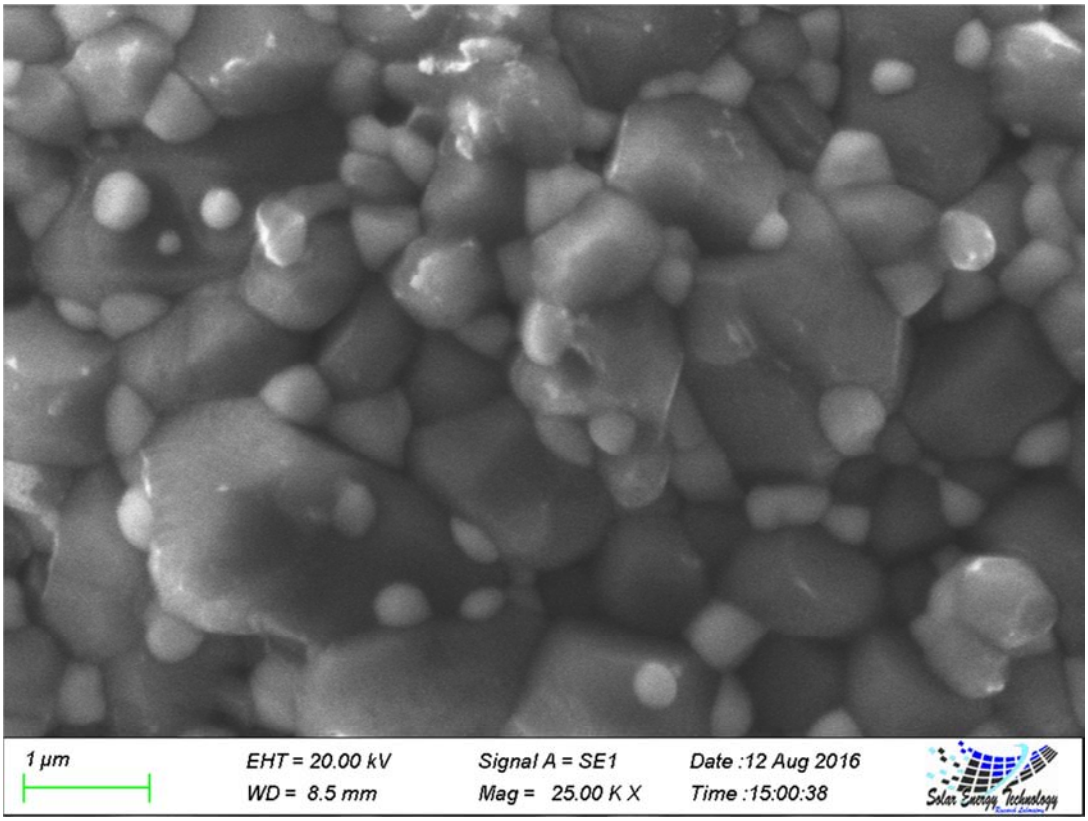

Figure 6. *SEM of ZTA containing 0wt% Titania.*

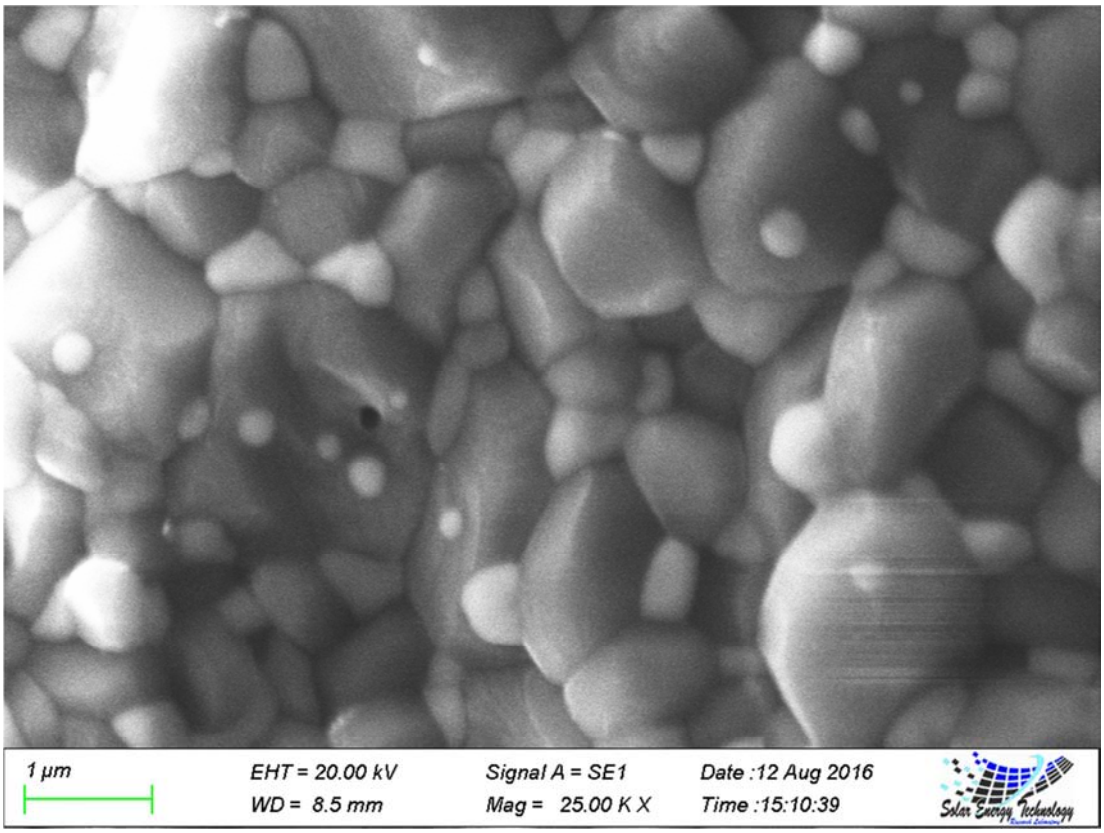

Figure 7. *SEM of ZTA containing 1wt% Titania.*

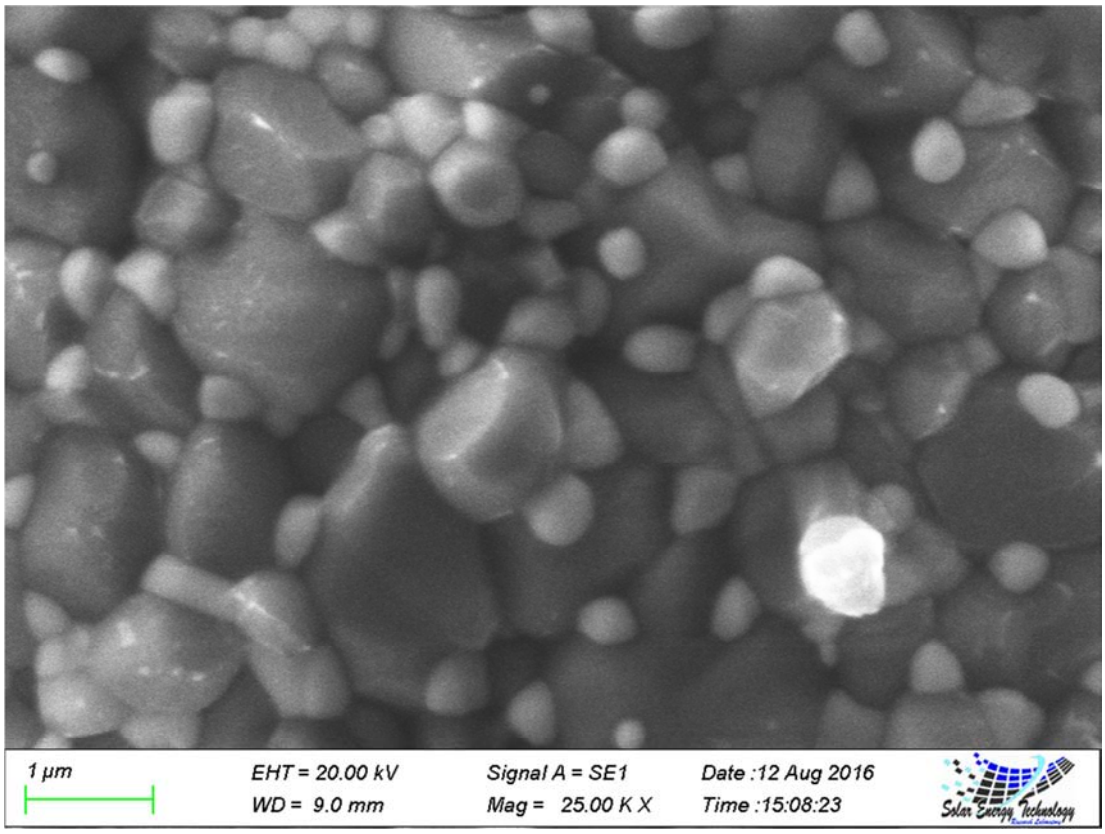

Figure 8. *SEM of ZTA containing 1.5wt% Titania.*

4. Conclusions

Sintered oxides ZTA–TiO_2 ceramics with different amounts of MgO addition were prepared and then analyzed. The surface properties and fracture behavior ZTA changed with the addition of TiO_2. The resulting microstructure differed in grain size and shape, depending on the amount of TiO_2 added. Bulk density improved with the addition of TiO_2 and it has found a suitable sintering temperature 1450°C. With the addition of TiO_2 porosity reduced and hence density and hardness of the composite were improved. In the Vicker hardness testing maximum value was 1617 HV in presence of 1.5% TiO_2. Where, without addition of TiO_2 the value of hardness was only 1389 HV. This 16.41% improvement of surface hardness is the great achievement of this research.

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