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Mechanical and Microstructural Properties of TiO_2 doped Zirconia Toughened Alumina (ZTA) Ceramic Composites at different TiO_2 contents

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ABSTRACT: Zirconia-Toughened Alumina (ZTA) is a glistening name for new generation of toughened ceramics for the past decade. In this experiment, microstructural and mechanical properties of ZTA ceramic were modified with TiO_2 as an additive which were constructed using a solid-sintering route. For various weight percents of TiO_2 (i.e. 0 wt%, 2 wt%, 3wt%, 4 wt%, 6 wt% and 8 wt%), corresponding constructed samples were dry mixed, uniaxially pressed and sintered at 1600°C for 1 hour in a pressureless condition. Properties like density, porosity, flexural strength, fracture toughness and Vickers hardness were measured for each sample. The grain growth was observed by using Scanning Electron Microscope (SEM). It was found that the flexural strength, fracture toughness and hardness have gradually increased with TiO_2 additions, reaching its maximum value at 4 wt.% and then decreased upon further addition of TiO_2. Scanning Electron Microscopy showed that the grain growth of Al_2O_3 was hindered significantly with the addition of 4 wt% TiO_2, but increased in size with further addition of TiO_2. Hardness and bulk density have also improved from 0wt% to 4wt% due to the fine microstructure, thus enhancing its properties.
Keywords: Fracture toughness, ZTA, TiO_2, sintering, microstructure, Flexural Strength

I. Introduction

Ceramics, the wonder materials are becoming an essential part of today's materials due to the advantages such as low density, strength, hardness and its inertness at high temperature [1]. They are extensively used as materials in making aircraft structures, electronic packaging to medical equipment, and space vehicle to home building. Regardless of their advantages, ceramic materials exhibit very low toughness which eventually limits their overall applications [2–6]. The challenge of increasing the toughness of ceramic based materials has been a key motivation in the field of ceramic research [6–8]. In this pursuit of improving toughness, Al_2O_3 based materials are often used as the benchmark due to its abundance, relative cheapness and excellent mechanical properties [9–11]. The introduction of the yttria stabilized zirconia (YSZ) toughening agent further increased the toughness of the zirconia toughened alumina (ZTA) ceramic composite [12]. Additionally, the use of additives was also introduced to reduce the sintering temperature, customise the microstructure as well as improve the product properties. It has been reported that the addition of TiO_2 promotes the sintering and grain growth of Al_2O_3 [13–15]. 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. Nevertheless, beyond its solubility limit, the contrary trend of decreasing densification and grain growth may be due to the pinning effect at the grain boundaries of the second phase, Al_2TiO_5 [15]. 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 authors' previous works investigated MgO, Cr_2O_3, CeO_2 and $CaCO_3$ for improving the mechanical properties of ZTA without diminishing properties of ZTA [3–6,16,17]. The current research has been done to understand the role of TiO_2 in ZTA ceramic composites. The amounts of TiO_2 were varied from 0 wt % to 8 wt % with fixed amount of ZTA as the matrix. Hence, this study aims to investigate the mechanical and microstructural properties of TiO_2 doped Zirconia Toughened Alumina (ZTA) Ceramic Composites.

II. Methodology

The raw materials used for this research were Al_2O_3 (Martinswerk, 99% purity), YSZ (Goodfellow, 5.4 mole% Y_2O_3 as stabilizer, >96% purity) and Anatase-TiO2 (Fluka, 99% purity, grain size: < 25nm). The initial mixture was 80 wt% Al_2O_3 and 20 wt% YSZ and was mixed with different amounts of TiO_2, whilst maintaining the ratio of Al_2O_3 to YSZ at 4:1. The mixtures were wet mixed using Ball Mixer Mill with ZrO_2 balls. The slurry was then dried for 24 h at 100°C in an Electrotherm oven, following which the dried cake was crushed and passed through a 100 μm sieve. The powders were then hydraulically pressed at 250 MPa for 120 sec into pellets of 14 mm diameter and 4 mm thickness without binder. The pellets were sintered at 1600°C for 1 hour at a heating rate of 5°C/minute in a pressureless condition.

Flexural strength of the samples were measured using three point bend test, where the dimensions of the samples were 50mm x 30mm x 25mm, keeping span length of 40mm and crosshead speed of 1mm/min. To measure the Vickers hardness and fracture toughness of the sintered samples, the Vickers indentation technique was used. With the *Hardness Tester Mitutoyo-model HV-114*, the Vickers hardness was measured by taking the average of five different readings for each sample. The polished sintered samples were subjected to HV 30 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.

III. Result and Discussion

Figure 1 shows the flexural strength of ZTA-TiO_2 ceramic composite with different TiO_2 contents. As shown in the bar chart, flexural strength gradually increases from 519.3 MPa (0 wt%) to 891.8 MPa (4 wt%), where flexural strength is the highest, and then decreases with further addition of TiO_2 content. This increase in flexural strength may be attributed to the fact that TiO_2 increases the pinning effect in the microstructure, therefore increasing the amount of stress required to bend the samples to rupture, and also due to the refinement of microstructure as shown in figure 2(d).

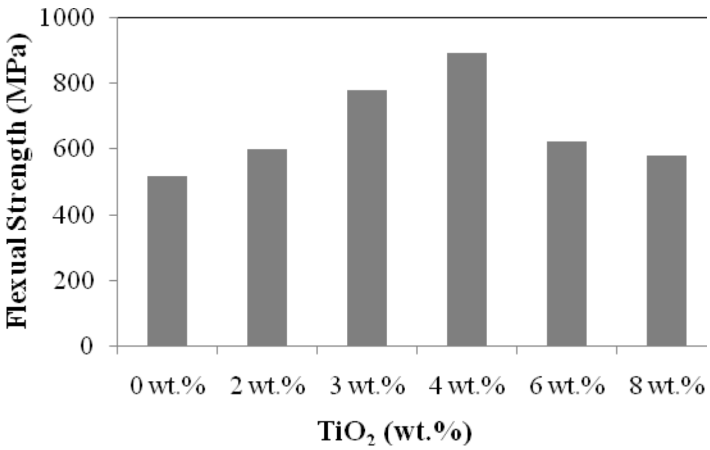

Fig. 1: Flexural strength of ZTA-TiO_2 ceramic composite with different TiO_2 contents.

Figures 2(a), 2(b), 2(c), 2(d), 2(e) and 2(f) show the Scanning Electron Microscope images of ZTA-TiO_2 ceramic composite samples with 0wt.%, 2wt.%, 3wt.%, 4wt,%, 6wt.% and 8wt.% TiO_2 respectively. According to the micrographs, it can be clearly observed that TiO_2 has influenced the grain growth of the ZTA-TiO_2 ceramic composites. The figures illustrate that as TiO_2 content was increased grain growth was hindered and at 4 wt.% TiO_2 grain size and grain growth was minimum, therefore successfully refining the microstructure. This observation also supports the fact that at 4wt.% TiO_2, flexural strength of the composite is maximum as shown in figure 1. Upon further addition, excess TiO_2 was no longer able to prevent the grain growth of the composites. According to Manshor *et al.* [18], excess TiO_2 will start to react with Al_2O_3 and form Al_2TiO_5 which in turn increases the grain size of the ZTA-TiO_2 ceramic composites.

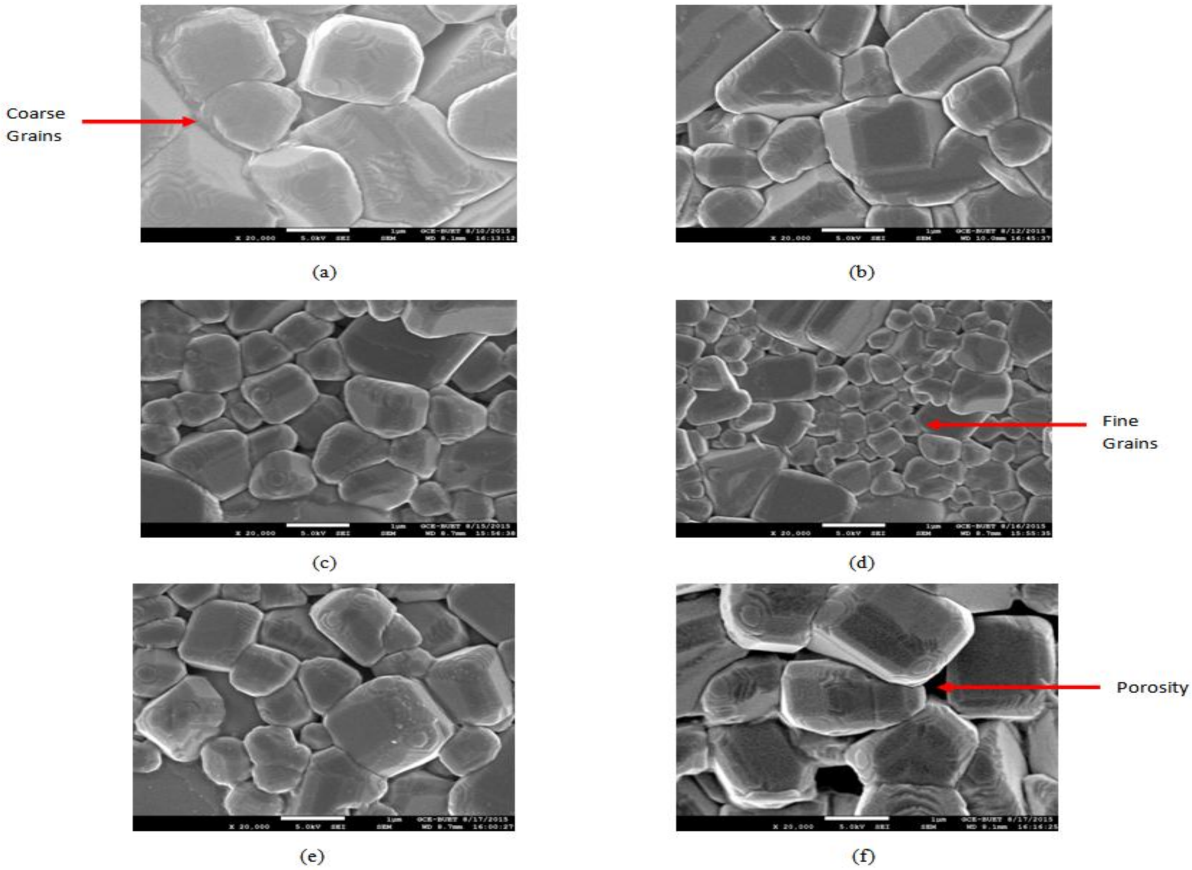

Fig. 2(a): SEM image of ZTA-TiO_2 ceramic composite with 0 wt.% TiO_2.
Fig.2(b): SEM image of ZTA-TiO_2 ceramic composite with 2 wt.% TiO_2.
Fig.2(c): SEM image of ZTA-TiO_2 ceramic composite with 3 wt.% TiO_2.
Fig.2(d): SEM image of ZTA-TiO_2 ceramic composite with 4 wt.% TiO_2.
Fig.2(e): SEM image of ZTA-TiO_2 ceramic composite with 6 wt.% TiO_2.
Fig.2(f): SEM image of ZTA-TiO_2 ceramic composite with 8 wt.% TiO_2.

Figure 3 presents the results of Vickers hardness for ZTA-TiO_2 ceramic composite with different TiO_2 contents. Vickers hardness was observed to gradually increase from 1503 HV (0 wt% TiO_2) to 1610 HV (4 wt% TiO_2), indicating an improvement of approximately 7 %. The results of Vickers hardness are directly related to the results of the bulk density, as shown in figure 4. The highest value of Vickers hardness, 1610 HV (4 wt% TiO_2), also coincides with the highest density value (4.18 g/cm3). The increase in hardness with the increasing TiO_2 content is attributed to an increase in densification. The Vickers hardness and bulk density both decreases with further addition of TiO_2. This is believed to be due to the presence of the secondary phase i.e. Al_2TiO_5 as indicated by Manshor *et al.* [18].

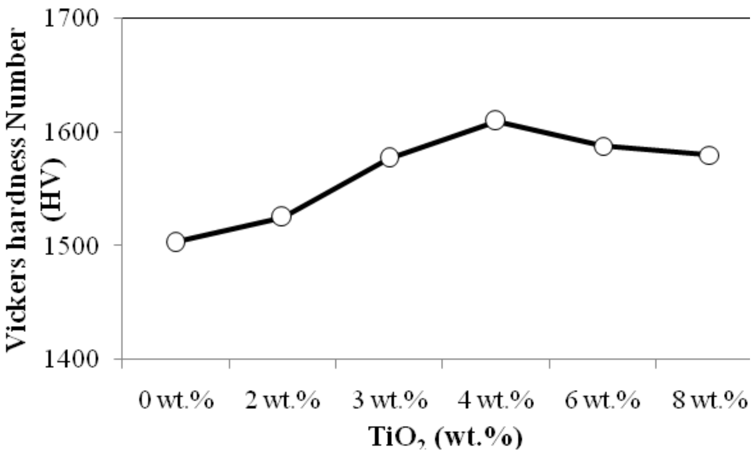

Fig. 3: Vickers hardness of ZTA-TiO_2 ceramic composite with different TiO_2 contents.

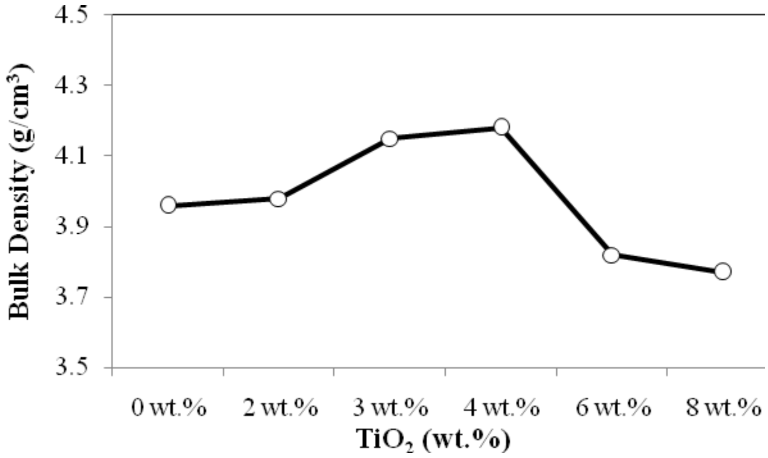

Fig. 4: Bulk Density of ZTA-TiO_2 ceramic composite with different TiO_2 contents.

Figure 5 shows the results of the fracture toughness for ZTA- TiO_2 ceramic composite. The fracture toughness gradually increased from 5.46 $MPa.m^{1/2}$ (0 wt% TiO_2) to 5.81 $MPa.m^{1/2}$ (4 wt% TiO_2), and then only had a minor increment after further addition of TiO_2.

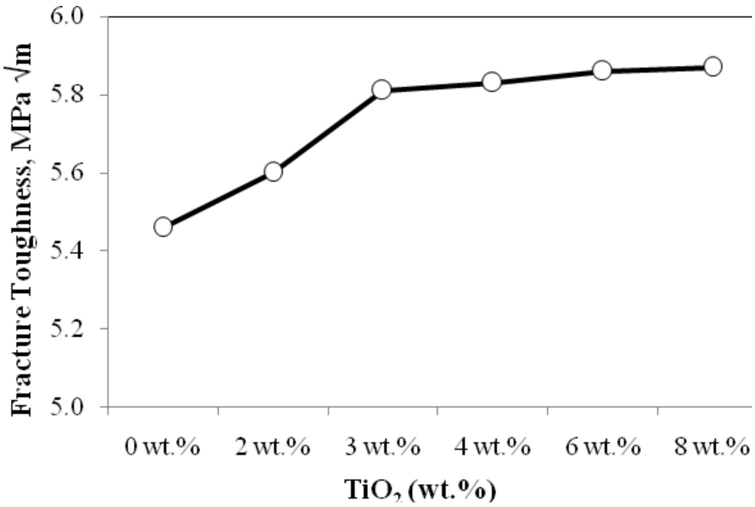

Fig. 5: Fracture toughness of ZTA-TiO_2 ceramic composite with different TiO_2 contents.

According to Wang *et al.*, [15], the TiO_2 addition to the ultrafine Al_2O_3 demonstrated that the toughness was reliant on the Al_2O_3 grain size, whereby the toughness raised from 4.4 to 5.2 $MPa.m^{1/2}$ as grain size increased from 0.6 to 2.44 µm due to the effect of TiO_2 additive.

IV. Conclusion

This experiment demonstrates the effects of TiO_2 particles on the mechanical and microstructural properties of ZTA-TiO_2 ceramic composites. The flexural strength of the composite was found to gradually increase when TiO_2 was added from 0 wt.% to 4 wt.%, reaching the maximum flexural strength at 4 wt.%, and then declines with further addition of TiO_2. According to the Scanning Electron Micrographs, grain growth is hindered maximum at 4 wt.% TiO_2, and grain size increases when TiO_2 content is greater than 4 wt.%, which is thought to be due to the formation of a secondary phase Al_2TiO_5. Hardness and bulk density were found to be the highest at 4 wt.% TiO_2, whereas fracture toughness tend to gradually increase from 0 wt.% TiO_2 to 4 wt.% TiO_2 and then shows no significant increase or decrease upon further addition.

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