# **Structural and Mechanical Properties of Zirconia Toughened Alumina (ZTA) Composites**

M. Moazzam Hossen<sup>1\*</sup>, F.-U.-Z. Chowdhury<sup>2</sup>, M. A. Gafur<sup>3</sup>, A. K. M. Abdul Hakim<sup>4</sup>

<sup>1</sup>Department of Computer Science & Engineering, International Islamic University Chittagong, Chittagong, Bangladesh. <sup>2</sup>Department of Physics, Chittagong University of Engineering and Technology, Chittagong, Bangladesh.

<sup>3</sup>PP and PDC, BCSIR, Dhaka, Bangladesh.

<sup>4</sup>Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka, Bangladesh.

Abstract - The Zirconia toughened Alumina (ZTA) composites with 0-16 wt % of ZrO<sub>2</sub> were prepared by slurry method and sintered at temperature (T<sub>s</sub>) 1500°C and 1600°C for 2 hours. The density, porosity, structural properties and mechanical properties of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> composites with respect to ZrO<sub>2</sub> content as well as sintering temperature have been explored in the present work. About 2% higher density of 16 wt % ZTA has been achieved and the microstructures are highly homogeneous and finer with less porosity when compared to pure Al<sub>2</sub>O<sub>3</sub>. The surface morphology of the samples was studied by using SEM. The effect of zirconia content on hardness and elastic modulus were investigated. At 1500°C, maximum hardness shows 15.79 GPa and at 1600°C, it is 19.76 GPa, which is observed for pure Alumina. However, at same temperatures, minimum hardness shows 11.24 GPa and 12.96 GPa, respectively, that is observed for 16 wt% ZrO<sub>2</sub>. Elastic modulus also shows same behaviour as shown for hardness. Flexural strength increases with the increase of both zirconia content and sintering temperature. The approach adopted in the present study may provide an alternative to design  $Al_2O_3$ -ZrO<sub>2</sub> composites with improved mechanical properties.

Keywords: ZTA; X-Ray Diffraction; SEM; Vickers microhardness; Flexural strength.

# 1. INTRODUCTION

The demand for composite materials over metals is increasing. Zirconia-toughened alumina (ZTA) composites have received significant scientific and technological attention during the last two decades for use in cutting tools, dies or prosthesis components due to their excellent room temperature strength, hardness, toughness and wear resistance [1]. More recently, ZTA has become increasingly important as a structural material for biomedical implants, such as hip prosthesis. A key issue for such implants is to increase their lifetime. For these reasons, research efforts currently focus on long-lasting devices based on new materials characterized by superior strength and toughness, optimal tribological properties and long-term biocompatibility [2].

In this frame, ZTA composites have demonstrated their effectiveness for orthopedic applications. In this system, alumina provides high strength and hardness, whereas tetragonal zirconia exerts a toughening effect, by controlling transformation into the monoclinic phase [3, 4]. It has been recognized that the magnitude of toughening and flexural

strength is completely dependent on the microstructure of the alumina–zirconia composite (i.e., volume fraction, size, shape, location and size distribution of  $ZrO_2$ ) [5, 6]. Generally, ZTA composites are prepared by slurry method, whose main issue is keeping a homogeneous microstructure in the final, sintered materials. In fact, zirconia aggregates can lead to localized aging phenomena [7, 8], whereas alumina ones could behave as preferential sites for

crack propagation. Zirconia particles can be embedded in a variety of different matrices to form transformationtoughened composites provided the zirconia particles have a sufficiently small size. Requirements are that the matrix will not react with the ZrO<sub>2</sub> or vice-versa and that the matrix has sufficiently high elastic modulus to maintain a low Ms (martensitic transformation) temperature. Usually, zirconia particles are likely to separate from each other and grow during sintering. However, evidence suggests that zirconia particles are mobile in nature and move within the grain boundary of the matrix. The presence of dispersed tetragonal zirconia particles in the matrix can affect the strength and toughness of the material in two ways: (a) the stress field of a crack can initiate the martensitic transformation and compressive stress on the crack surface is developed due to the expansion of transformed particles against the matrix, and (b) the transformation may take place near the surface of the macroscopic piece due to surface grinding and result in a compressive surface which leads to higher strength values. The increased volume fraction of zirconia is also responsible to reduce the stability of the tetragonal phase. Hence, an optimum amount of zirconia addition is expected to retain the favorable amount of tetragonal content which can enhance the thermal and mechanical properties of ZTA without any grain growth during sintering. Therefore, in order to control the properties of the product, it is important to understand these aspects at each and every stages of the process. Excellent control over composition can be achieved, most notably in the preparation of multi-component oxides that are homogenous at a near-atomic scale. On the basis of the above findings and importance, the particular interest of this study was to develop a series of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> composites through chemical route and characterize mechanical properties.

The present work deals with the structural and mechanical properties of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> composites by slurry method. Preparation of various Zirconia Toughened Alumina compositions was done. X-ray Diffraction (XRD) patterns and microstructures were analyzed. Physical properties such as density and porosity and mechanical properties such as microhardness, elastic modulus and flexural strength were determined.

### 2. EXPERIMENTAL

#### 2.1 Sample Preparation

The commercially available powders used in this work were the nanocrystalline,  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> powder and yttriastabilized ZrO<sub>2</sub> (3 mol%) powder from the manufacturing company Inframat<sup>®</sup> Advanced Materials<sup>TM</sup>, USA. Alumina presented crystal size ~40 nm and particle size ~150 nm and zirconia presented average particle size 30-60 nm. To prepare different composition, related powders were weighed by following unitary method, keeping the total weight constant.

Different compositions had been prepared by varying  $ZrO_2$  content from 4 to 16 wt% in the step of 4 wt%. A pure  $Al_2O_3$  sample was also prepared to compare its properties with those of ZTA composites. To attain the compositions, mechanical milling was performed in distilled water with 65 wt% of solid content for 24 hours in ball milling, with zirconia ball as a grinding media. Ammonium polyacrylate was used as dispersant. The slurry of the power mixtures was dried in an oven for 24 hours at 100°C and the dried lumps were crushed and passed through a plastic sieve. Hand milling was done for several hours to obtain a homogeneous distribution. Powder compacts with dimension of  $5 \times 6 \times 50$  mm were confirmed by uniaxially pressing at 60 MPa. Before compaction, polyvinyl alcohol (PVA) was mixed as a binder with the powder to provide some green strength for subsequent handling. The withdrawal of the binder was carried through in a muffle furnace with heating rate 6 °C/min at 600 °C per 180 min. The sintering was carried out in a box furnace at 1500 °C and 1600 °C for two hours with a heating rate of 20 °C/min. The bulk densities of the sintered samples were measured from the ratio weight/volume. The sintered specimens were machined longitudinally with a 325 grit-resin bonded diamond wheel at a depth of 5 µm/pass. The final dimensions of the specimens were  $3 \times 4 \times 35$  mm.

#### 2.2 Measurements

Phase identification was confirmed by powder X-ray diffractometry (XRD) technique using BRUKER X-Ray Diffractometer of Model D8 Advance with Cu- $K_{\alpha}$  radiation ( $\lambda$ = 1.54 Å). For each sample a scan has been performed from 20° to 75° with a step size of 0.02°.The volume fraction of monoclinic zirconia (V<sub>m</sub>) was calculated according to the following equation:

$$V_m = \frac{I_{(\bar{1}11)_m} + I_{(111)_m}}{I_{(\bar{1}11)_m} + I_{(111)_m} + I_{(111)_t}}$$

Theoretical density  $\rho_o$  of the sample was calculated from the actual density of the starting powders and their weight percentages using the rule of mixtures [9]:

$$\rho_o = \rho_A V_A + \rho_Z V_Z$$

where  $\rho_A$ ,  $\rho_Z$  are the density and  $V_A$ ,  $V_Z$  are the volume fraction of Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> respectively. Values of  $\rho_A$ = 3.97 gm/cm<sup>3</sup> and  $\rho_Z$  =6.10 gm/cm<sup>3</sup> have been used here to calculate  $\rho_o$ . The measured bulk density  $\rho_b$  was determined using the formula:

$$\rho_b = \frac{m}{\pi r^2 h}$$

Where m is the mass, r the radius and h the height of the sample.

The porosity P of the sample was determined using the relation.

$$P = (1 - \frac{\rho_b}{\rho_0}) \times 100\%$$

Microstructures for the samples of different compositions were examined by using Philips XL-30 scanning electron microscopy (SEM). Mean linear intercept

length l is used to determine the ASTM grain size number, G, by using the linear intercept method:

$$G = -3.2877 + 6.6439 \log_{10} l$$

where l is in millimeters at 1× Magnification.

The methodology used for the determination of hardness, was in accordance with ASTM C1327-99 [10]. Five Vickers impressions had been carried through in the surfaces of each one of the samples, which already were polishing, using an applied load of 2 kgf (19.6141 N) during six seconds. After the diagonal length measurement, the values of the Vickers hardness (GPa) were calculated, by the following equation [10, 11]:

$$H_v = 0.0018544 \times \frac{F}{d^2}$$

Where F is the load in N and d is the average length of the two diagonals of the indentations in mm.

The porosity dependence Elastic Modulus can also be measured using the Dewey-Mackenzie relation [12]:

$$E = E_{o}(1 - 2P)$$

where E is the effective elastic modulus of the porous composites,  $E_o$  is the elastic modulus of the dense composites and P is the porosity of the sample.

The flexural strength of the specimens was determined according with ASTM D790–10 by three-point bending method (Hounsfield Model H10KS-universal test machine), with a span of 30 mm and crosshead speed of 0.5 mm/min at ambient room temperature conditions. The flexural strength was calculated from the following equation.

$$\sigma_{flexural} = \frac{3WL}{2BD^2}$$

where W is the load (N), B is the width (mm) and D is the thickness (mm) of the sample.

# 3. RESULT AND DISCUSSION

### 3.1 XRD analysis

The XRD patterns of a ZTA sample containing 0, 4, 8, 12 and 16 wt%  $ZrO_2$  sintered at 1500°C and 1600°C are shown in figure 1. and figure 2. respectively which confirms the formation of multi-phase ZTA.



Figure 1. XRD pattern of ZTA composites containing different wt% ZrO<sub>2</sub> and sintered at 1500°C for 2 hours.

XRD analysis of the samples indicate that only  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, t and m-ZrO<sub>2</sub> are the crystalline phases present in the pure Al<sub>2</sub>O<sub>3</sub> and in the ZTA composites, respectively. It is observed that at 4 wt% and 8 wt% ZrO<sub>2</sub> content, there is no m-ZrO<sub>2</sub> phase and all are t-ZrO<sub>2</sub> phase, while with the increase of ZrO<sub>2</sub> content, m-ZrO<sub>2</sub> phase increases and the t-ZrO<sub>2</sub> phase decreases. However, t-ZrO<sub>2</sub> retention becomes much easier to trigger the transformation to monoclinic. The extent of toughening achieved in the composites depend on the

particle size of Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>, volume fraction of



Figure 2. XRD pattern of ZTA composites containing different wt%  $ZrO_2$  and sintered at 1600°C for 2 hours.

ZrO<sub>2</sub> retained in the metastable tetragonal phase as well as on the relative distribution of Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> in the matrix [13]. Finer particle size of both Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> will not only enhance the chances of a uniform Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> distribution, it also increases the possibility of ZrO<sub>2</sub> being retained as metastable tetragonal phase [14]. With the increase of ZrO<sub>2</sub> content, intensity of some t-ZrO<sub>2</sub> peaks are reduced, whereas some  $m\text{-}ZrO_2$  peaks are appeared and further intensity increases. So it can be inferred that monoclinic phase is indeed retained at the room temperature due to the presence of the hard alumina matrix and this phase is responsible for flexural strength improvement by transformation toughening mechanism. The effect of sintering temperature 1500°C and 1600°C on the XRD pattern of a ZTA composite containing 0, 4, 8, 12 and 16 wt% ZrO<sub>2</sub> has been shown in the related figure. From figure 1. and figure 2. it is observed that with the increase of sintering temperature more peaks for ZrO<sub>2</sub> have been found. It also reveals that with the increase of sintering temperature, more m-ZrO<sub>2</sub> phases appear and the intensity of t-ZrO2 decreases.

Densification of powdered particle is a thermally assisted phenomenon due to sintering at 1500°C and 1600°C. Figure 3 shows the result of density as a function of zirconia content. Presence of ZrO<sub>2</sub> results in a continuous increase in density. The theoretical density of ZrO<sub>2</sub> is around 6.10 gm/cc whereas that of Al<sub>2</sub>O<sub>3</sub> is 3.97 gm/cc, therefore the effective density values of the aluminazirconia rise with increasing ZrO<sub>2</sub> content and a compact structure is formed. Sintering in practice is the control of both densification and grain growth. With the continual supply of thermal energy, more and more contact points are created between the particles and the intervening porosity diffuses out of the contact area. Hence a substantial increase in density is resulted. In the mean time the existing grains inside the particle have an opportunity to grow in order to minimize the overall energy of the system.

Thus densification and grain growth occurs simultaneously. It shows that density increases with sintering temperature. It is known that matter transport during the sintering of polycrystalline composites occurs by diffusion, a thermally activated process, which can occur along different paths in the solid, giving rise to the different mechanisms of diffusion: lattice, grain boundary and surface diffusion. It is also known that there is an exponential dependence of diffusion coefficient (D) on temperature.



Figure 3. Theoretical and Bulk Density as a function of zirconia content sintered at different temperatures for 2 hours.

Rising in temperature increases the value of D and hence density increases with sintering temperature. The density is also influenced by phase transformation of monoclinic to tetragonal zirconia above 1170°C during heating. The more the tetragonal phase retains in the structure, the more the density of the composite attains. The density result reveals that the elimination of pore, simultaneous grain growth with grain boundary diffusion and lattice diffusion are the predominant mechanism to compact the material at high temperature. In this work, the theoretical density calculated from the rule of mixture was found around 94.21% for pure Al<sub>2</sub>O<sub>3</sub> and that of 94.51-96.11% for 16 wt% of ZTA sintered at 1500°C for 2 hours. In addition, about 98.65% of theoretical density was observed for pure Al<sub>2</sub>O<sub>3</sub> and 99.05-99.28% that for same ZTA sintered at 1600°C for 2 hours. About 3-6% density has been changed due to increase of 100°C for both pure Al<sub>2</sub>O<sub>3</sub> and ZTA. This resembles that after increasing sintering temperature particles become more compact and dense. Moreover, increasing temperature and Zirconia content, density increases more linearly than that of lower temperature and content.



Figure 4. Relative Density and Porosity as a function of zirconia content sintered at different temperatures for 2 hours.

Figure 4. shows the effect of  $ZrO_2$  content and sintering temperature on the porosity of ZTA composites, respectively. It is observed that the porosity is decreased with  $ZrO_2$  content as well as sintering temperature. It resembles that the composites are in more compact form at the higher temperatures. During the diffusion process of sintering, the intervening porosity diffuses out, resulting in reduction of porosity. It is observed that the porosity is decreased with  $ZrO_2$  content as well as sintering temperature. This porosity usually originates from entrapped pore during green compaction.

With the increase of temperature, sample becomes more homogeneous. During 100°C interval, porosity decreases sharply from low temperature to high temperature. It is also seen that the percentage of porosity is minimum at the sintering temperature 1600°C for all the composites than that of at 1500°C, i.e., it reveals that the sample is more compact at this temperature.

## 3.2 Scanning electron microscopy

Figure 5. (a)–(e) show the SEM micrographs of the ZTA composites containing different wt%  $ZrO_2$  at 1600°C. When  $ZrO_2$  was added, the grains become finer compared to that of pure  $Al_2O_3$ , as shown in Figure 5. (b)-(e). Observations show highly homogeneous microstructures without agglomerates, pores or abnormally grown alumina grains. The micrograph exhibits the  $Al_2O_3$  and  $ZrO_2$  grains as dark and whitish color respectively, where zirconia grains are embedded around the alumina grain and within the grain. However, small fractions of intragranular zirconia grain are also being noticed.

From the figure, it can also be observed that  $ZrO_2$  particles are uniformly dispersed throughout the alumina matrix. As the amount of  $ZrO_2$  content increases, the  $Al_2O_3$  grain size decreases significantly. The  $ZrO_2$  phase creates a pinning effect around  $Al_2O_3$  grain and obstructs its growth. When  $ZrO_2$  is added with  $Al_2O_3$ , evenly distributed fine  $ZrO_2$  grains act as grain refiner and led to smaller  $Al_2O_3$  grains. This is possibly due to increase in density as well as enhancement of mechanical properties. It can also be observed that the grain sizes of  $ZrO_2$  increase and hinders the grain growth of  $Al_2O_3$  at  $1600^\circ$ C, which contributes to the lowering of porosity. A fine grain size and refined microstructure are necessary for improved mechanical properties especially for wear resistance of dental implant.

It is observed from Figure 6 that, grain size of  $Al_2O_3$ and  $ZrO_2$  vary with the amount of Zirconia content added with the pure  $Al_2O_3$ . In the present work, it is seen that when  $ZrO_2$  was added to  $Al_2O_3$  then the grain size decreases slowly.

## 3.3 Hardness and elastic modulus

The Vickers hardness of  $Al_2O_3$ - $ZrO_2$  composites as a function of zirconia content and at temperature 1500°C and 1600°C, respectively are shown in figure 7. Figure 7. shows the variation in hardness of ZTA composites with the amount of zirconia content. Hardness decreases with the increase in  $ZrO_2$  content. Zirconia has lower hardness than

alumina and it can also obstruct the densification of the alumina phase. Again, amount of zirconia has an adverse affect on the hardness of the composites due to coarsening of the zirconia grains and formation of subsequent porosity. From the figure it is also clear that, for any particular composition, hardness increases with sintering



(a) Al<sub>2</sub>O<sub>3</sub>



(b) Al<sub>2</sub>O<sub>3</sub>+4 wt% ZrO<sub>2</sub>



(c) Al<sub>2</sub>O<sub>3</sub>+8 wt% ZrO<sub>2</sub>



(d) Al<sub>2</sub>O<sub>3</sub>+12 wt% ZrO<sub>2</sub>



(e) Al<sub>2</sub>O<sub>3</sub>+16 wt% ZrO<sub>2</sub>

Figure 5. Microstructure of Different Compositions sintered at 1600°C

temperature. As higher sintering temperature offers better densification, hardness value increases as a whole. Similar trend in hardness has been observed by R. P. Rana [15]. Similar trend in hardness has also been observed by Basu et al. [16]. The hardness increases with sintering temperature for all compositions. At 1500°C, maximum hardness shows 15.79 GPa and at 1600°C, it is 19.76 GPa, which is observed for pure Alumina. However, at same temperatures, minimum hardness shows 11.24 GPa and 12.96 GPa, respectively, that is observed for 16 wt% ZrO<sub>2</sub>. At the same temperatures, maximum and minimum hardness of 4, 8 and 12 wt% ZrO<sub>2</sub> are in between these values. In addition, for all the compositions, hardness increases moderately with T<sub>s</sub>. The above increase in the hardness value of the Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> composite is related to the density increment. It appears that the phases of the sintered composite have least influence on the hardness.



Figure 6. Variation of grain size with zirconia content at various sintering temperature



Figure 7. Variation of hardness and elastic modulus with zirconia content at different sintering temperature

Elastic modulus of the composite depends on the volume fraction of the phases present and their individual modulus of elasticity. At 1500°C, densification of the composites is improved by reducing pore volume fraction of the matrix and as a result of which the modulus of elasticity improves. Alumina has a higher modulus of elasticity compared to zirconia. So, samples containing 4 wt% zirconia have the highest modulus of elasticity, whereas specimen containing 16 wt% zirconia has lower levels of densification at a given sintering temperature compared to other samples having lower amount of zirconia. Similar phenomena also have been observed for 1600°C. Simultaneously, the grain size of zirconia increases and hinders the grain growth of alumina at 1600°C, which contributes to the lowering of elastic modulus. Nicholson [17] established that both Young's modulus and hardness of ZTA composites containing particles of zirconia stabilized with 3 mol% yttria followed a linear rule of mixtures.

#### 3.5 Flexural Strength

The Flexural strength increases linearly with increasing zirconia content as well as sintering temperature that has shown in figure 8. The flexural strength was found around 401 MPa for pure  $Al_2O_3$  and that of 656 MPa for 16 wt% of ZTA sintered at 1500°C for 2 hours. In addition, about 432 MPa of flexural strength was observed for pure  $Al_2O_3$  and 691 MPa for 16 wt% of ZTA sintered at 1600°C.



Figure 8. Variation of Flexural strength with zirconia content at different sintering temperature

The martensitic transformation and frontal process zone are predominant mechanisms to increase the flexural strength of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> nanocomposite. Since the tetragonal zirconia grains do not undergo the stress-induced transformation to the monoclinic form when grain size is smaller than a critical size. The particle sizes lower than the critical value does not affect the strength. The reduction of size improves the bulk density but not the transformability of the tetragonal zirconia grains. Usually, the effect of grain size on strength in transformation-toughened composites is a complex phenomenon [18]. An increase in grain size improves the transformability, therefore, increasing strength. Microstructure reveals that the zirconia grain size increases with increasing zirconia content. The m-ZrO<sub>2</sub> phase increases with increase in ZrO<sub>2</sub> content. The retention of t-ZrO<sub>2</sub> varies during fracture and t-ZrO<sub>2</sub> fraction decreases with increasing zirconia content. However, t-ZrO<sub>2</sub> retention becomes much easier to trigger the transformation to monoclinic. As a result, its contribution to transformation toughening predominates although retention of m-ZrO<sub>2</sub> aids to microcrack toughening.

The addition of zirconia is very effective in hindering the grain growth of alumina during sintering, even for the lowest zirconia content. The addition up to 16 wt%  $ZrO_2$ prevents grain growth without allowing the formation of zirconia agglomerates, which are very deleterious for the mechanical stability of the composite. However, the premature zirconia agglomeration can be observed in case of composites containing 15 mol%  $ZrO_2$  [16].

# 4. CONCLUSION

The Zirconia-toughened alumina (ZTA) composite has been successfully prepared by slurry method and they have been sintered at 1500°C and 1600°C for two hours. The sintering temperature and sintering time plays vital role in changing the properties. The X-ray diffraction studies clearly showed the formation of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, t and m-ZrO<sub>2</sub> phases both in the pure Al<sub>2</sub>O<sub>3</sub> and in the ZTA composites, respectively. It is seen that the ZrO<sub>2</sub> addition promotes composites with higher densities. The density has been found to increase with increasing zirconia content and sintering temperature, while porosity decreases with the same change of zirconia content and sintering temperature. SEM images show the microstructures of prepared ZTA which reveals that the grains are homogeneous and finer at higher T<sub>S</sub>. It can also be observed that the grain sizes of ZrO<sub>2</sub> increase and hinders the grain growth of Al<sub>2</sub>O<sub>3</sub> at 1600°C compared to that of at 1500°C, which contributes to the lowering of porosity. Hardness and elastic modulus was found to be increased with sintering temperature, while the addition of ZrO<sub>2</sub> decreases the hardness and elastic modulus of the composite. The Flexural strength increases with increasing zirconia content as well as sintering temperature. The martensitic transformation and frontal process zone are predominant mechanisms to increase the flexural strength of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> nanocomposite. The zirconia substitution in pure Al<sub>2</sub>O<sub>3</sub> and the change of sintering temperature was found to have a significant

effect on structural and mechanical properties.

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