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Contributed Paper

Physical and Mechanical Properties of Zirconia Toughened Alumina (ZTA) Composites Fabricated by Powder Injection Moulding

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ABSTRACT

Zirconia toughened alumina (ZTA) ceramic composites have been fabricated by powder injection moulding. The ZTA composites, having alumina with zirconia in range of 0-15 wt %, were mixed with polymeric binder to form feedstocks. The binder composed of polyethylene glycol (PEG), polyvinyl butyral (PVB), and stearic acid (SA). The plunger-typed injection moulding machine was employed in this work. The moulded specimens were subjected to PEG removal using water immersion method. The remaining PVB was eliminated during ramping up to the sintering temperatures. Pressureless sintering was done at temperature of 1600, 1650, 1700 and 1750 °C with a holding time of 2 hour. Physical and mechanical properties, i.e. density, hardness and flexural strength were evaluated. Microstructures of sintered components were characterized using scanning electron microscope. The results showed that sintering temperature strongly affected the physical and mechanical properties of the composites. The amount of zirconia also played an important role in the hardness and strength values of the specimens. Microstructures revealed that zirconia inhibited grain growth of alumina, consequently, enhanced properties of ZTA. The densities of ZTA ceramic composites obtained in this work were 97-98% of the theoretical value when sintered at 1650-1700 °C.

Keywords: ceramic injection moulding, water soluble binder, composites, zirconia, alumina

1. INTRODUCTION

Zirconia toughened alumina (ZTA) is one of the promising materials and is a material of interest due to its excellence properties such as high hardness, strength and good thermal shock resistance [1]. It has been widely used in various applications such as cutting-tools, biomedical implants, and structural parts [2]. The ceramic injection moulding (CIM) is one of the preferable ceramic processing because it can produce complex shape components with good dimension control, hence eliminate or minimize final machining. CIM normally

composed of 4 main steps, i.e. feedstock preparation, injection moulding, binder removal (debinding) and sintering. Generally, wax-based binder system can be employed in injection moulding of metallic, ceramic and composite powders. However, organic solvents, which are not environmentally friendly, would be required during solvent debinding step. Therefore, alternative water-soluble binder system composed mainly of polyethylene glycol (PEG) was developed [3]. CIM will allow complex-shaped ZTA components to be produced; moreover, using water-soluble binder system will lead to green fabrication process in industry.

In order to achieve a success fabrication of the products, the ZTA feedstock for the ceramic injection moulding required several adjustments of processing parameters. Therefore, this research investigated the effect of ZrO_2 content and other processing parameters on the physical and mechanical properties of ZTA composites fabricated by CIM route. The knowledge gained from this work will be beneficial for the preparation of small and complex shaped ZTA ceramics for various applications.

MATERIALS AND METHODS

Alumina powder (99.5%), having an average particle size of 1.63 μm, and zirconia powder (TZ-3Y-E) with an average particle size (D_{50}) of 1.93 μm were used in the study. The powders were mixed in four weight ratios of alumina: zirconia, that are 100:0, 95:5, 90:10, and 85:15, and are referred as compositions A1, A2, A3, and A4, respectively. The compositions were then mixed with polymeric binder to make feedstock having a powder loading of 50 vol%. The remaining 50 vol % of binder consists of polyvinyl butyral (PVB), polyethylene glycol (PEG), and stearic acid (SA) in the weight ratio of 20:78:2. Injection moulding was carried out with a plunger typed machine to form as-moulded components of ZTA feedstocks, i.e. green samples. The operating temperature for injection moulding was 180°C. The green samples were subsequently immersed in water to removed the water- soluble binder, i.e. PEG. The as-leached components, after PEG removal, were called brown samples. In order to study the rate of binder removal, specimens were weighed before and after leaching in water at several intervals. The weight difference will be calculated as percentage of PEG removed. The remaining backbone binder (PVB) and stearic acid (SA) were thermally debinded in the furnace. Pressureless sintering was done at temperature of 1600, 1650, 1700 and 1750°C with a holding time of 2 hour. The morphology was observed using scanning electron microscope (SEM). The density and porosity of the samples were determined using the Archimedes method according to ASTM C373. Vickers hardness was used to evaluate the mechanical properties of the sintered specimens. The flexural strengths of the samples were measured in three-point bending using a universal testing machine with the loading rate of 0.05 mm/min.

RESULTS AND DISCUSSION

The PEG losses versus time of A1-A4 green samples are presented in Figure 1. The PEG was quickly removed from samples in first 8 hours of immersion time then it was slowly removed until the theoretical PEG loss reach about 100%. However, the difference in ZrO_2 content in feedstock compositions does not show any significant effect on the PEG removal.

Figure 2a showed that the %theoretical density (TD) of the specimens increased from about 92% to about 97% when the temperature increased from 1600 to 1650°C

then stable and a bit fluctuate when sintering temperature was raised to 1750°C. Figure 2b presented the effect of zirconia content (from 0-15 wt %) on the density. It showed that TD fluctuated as ZrO_2 content increased. From the results, it is, therefore, confirmed that sintering temperature strongly affect to density of the ZTA specimens, whereas, the ZrO_2 content in range used in this work does not show any significant effect on the density. However, the fluctuation result observed in the present study was thought that it would be attributed to the non-homogeneity of the feedstock. This might come from the procedure of feedstock mixing, the powder loading of the feedstock, and injection moulding condition [4, 5].

For the mechanical properties, it was found that sintering temperature and ZrO_2 contents were strongly affected the flexural strength and hardness of the sintered specimens. Figure 3a showed that flexural strength of sample A4 (15 wt % ZrO_2) was increased from 259 to 355 MPa as the temperature rose from 1600 to 1700°C. After that the flexural strength dropped to 297 MPa as the temperature increased to 1750°C. Other samples (except A1) followed quite the same trend of flexural strength versus temperature, that is, the flexural strength increased up to the highest value and then drop as the temperature continued to increased. The increase of ZrO_2 contents, as seen in Figure 3b, resulted in an increase of the flexural strength as the temperature increased. The trend of hardness versus temperature also showed the similar trend.

Figure 4a presents an increase of hardness values of sample A4 from 1504 to 1766 HV as the temperature increased from 1600 to 1650□C and then drop to 1680 HV as the temperature continued to increase to 1750□C. The increase of ZrO_2 contents, as shown in Figure 4b resulted in an increase of hardness at all sintering temperatures employed in this work. Overall, sample A4 showed the highest values of flexural strength and hardness although its density was quite similar to other samples. The mechanical properties achieved in the current work are compared with research from other researchers' group as presented in Table 1. It was found that the properties of the present study are comparable with other ZTA products formed by other manufacturing methods.

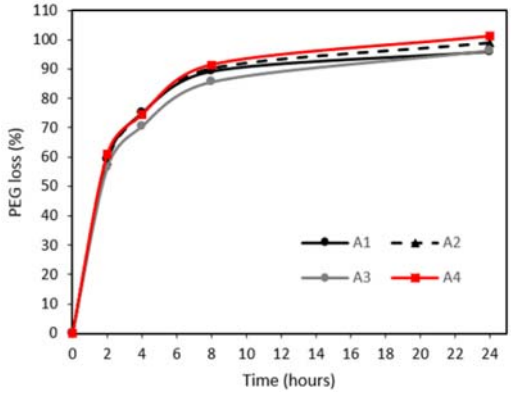

Figure 1. PEG loss vary with time.

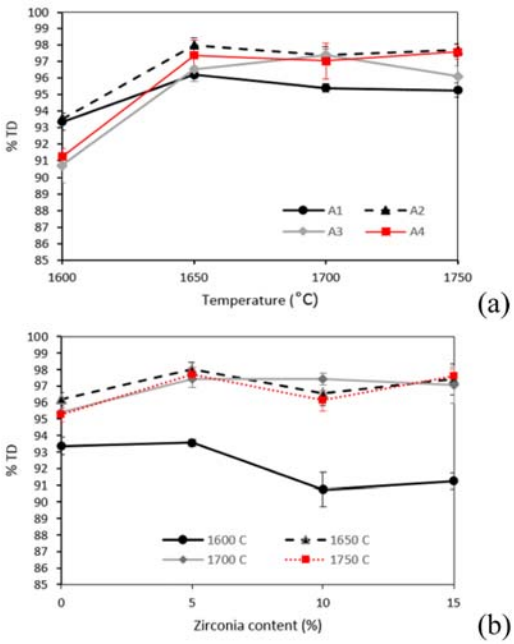

Figure 2. The effect of sintering temperature (a) and zirconia content (b) on the percentage of theoretical density.

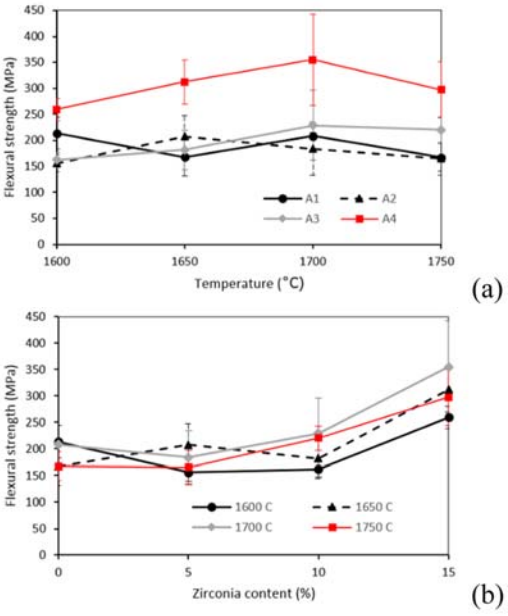

Figure 3. The effect of sintering temperature (a) and zirconia content (b) on the flexural strength.

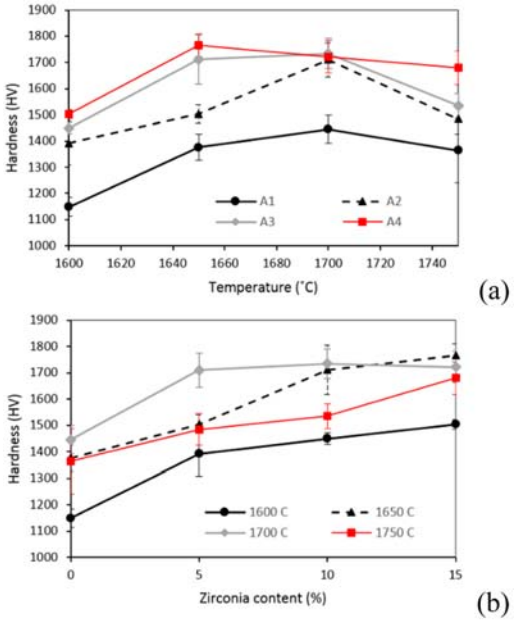

Figure 4. The effect of sintering temperature (a) and zirconia content (b) on the hardness.

Table 1. The mechanical properties of ZTA of this research compared to the others.

ZTA researches	Forming technique	Sintering temperature	Flexural strength of only ZTA	Hardness of only ZTA
ZTA produced by freeze dried granule [6]	Dry pressing	1650°C	150-430 MPa	1500-1540 HV
ZTA/Nb_2O_5 frabricated by sol-gel [7]	Dry pressing	1650°C	246 MPa	1438 HV
ZTA/BN fabricated by in-situ reaction [8]	Hot pressing	1800°C	643 MPa	13 GPa (eq. 1937 HV)
ZTA feedstock [9]	Injection moulding	1650°C	261-334 MPa	1999-2116 HV
ZTA of this study (A4)	Injection moulding	1700°C	355 MPa	1722 HV

The microstructure of sample A1 and A4 are shown in Figure 5. It was found that as the temperature was raised from 1600°C to 1700°C, the porosity of both samples decreased. However, further increment of the temperature to 1750°C resulted in alumina grain growth (as shown in samples A1), whereas samples A4 no grain growth was observed. This confirmed the experimental results that ZTA composites had better mechanical properties than those of alumina specimens. Moreover, there are also numbers of methods to control and suppress the grain growth regarding the

increase in temperature such as using the two-step sintering or doping with other oxide additives eg. MgO, Y_2O_3 [7]. Further work will be carried out to investigate the grain growth inhibition using these procedures.

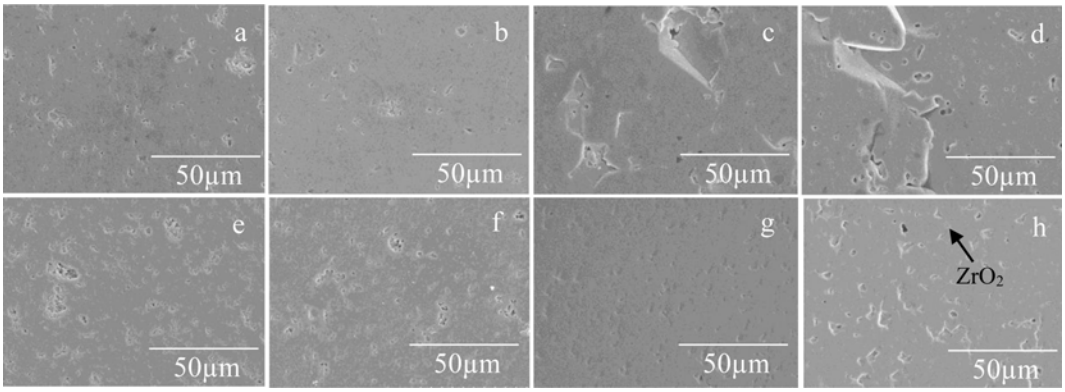

Figure 5. The SEM image of A1 with the sintering temperature of 1600°C (a), 1650°C (b), 1700°C (c), 1750°C (d) and A4 with the sintering temperature of 1600°C (e), 1650°C (f), 1700°C (g), and 1750°C (h).

CONCLUSIONS

ZTA composites were successfully fabricated by ceramic injection moulding method using water-soluble binder system. The results can be concluded as follows:

- The zirconia content does not affect much to the theoretical density while the sintering temperature strongly affects the density.

- The flexural strength and hardness are strongly affected by both zirconia content and sintering temperature. The higher mechanical properties values were obtained with an increase of zirconia content and sintering temperature. However, there was a limit of sintering temperature that the properties could be deteriorate when reach that temperature.

- The feedstock homogeneity, injecting time and pressure must be further studied as they can affect the perfection of green samples that will subsequently affect the properties sintered components.

- The highest properties of the specimens obtained from this work was from the ZTA with zirconia content of 15 wt %, using sintering temperature in range of 1650-1700°C. It had density of 97-98% of the theoretical values and the flexural strength of approximately 300 MPa.

This work can also be extended to the fabrication of more complex-shaped components such as parts for biomedical applications or some jewelry articles.

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