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Contributed Paper

# Powder Injection Moulding of Dental Ceramic Brackets Using Water Soluble Binder

**Nutthita Chuankrerkkul* [a], Thanakorn Wasanapiarnpong [b] and Khanittha Noomun [a]**
[a] Metallurgy and Materials Science Research Institute, Chulalongkorn University, Bangkok 10330, Thailand.
[b] Department of Materials Science, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand.
* Author for correspondence; e-mail: nutthita.c@chula.ac.th

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## ABSTRACT

Powder injection moulding (PIM) is one of mass production and cost effective near net shape processing, especially for small, complex-shaped components for high performance ceramic applications. Alumina has been generally known as one of the most widely used advanced ceramics due to its excellent properties such as high hardness, high strength, as well as its wear resistance and bioinertness. This work investigates PIM technique for a fabrication of alumina and zirconia toughened alumina compounds for dental ceramic brackets application. A water soluble binder system, composed of a major fraction of polyethylene glycol (PEG) and a minor fraction polyvinyl butyral (PVB), has been studied in a preparation of feedstock for PIM. Injection moulding was carried out with a homemade laboratory scale equipment. PEG can be removed rapidly by water leaching. The remaining PVB is removed by subsequent calcination and the samples were then subjected to sintering. The development of feedstock formulations and processing parameters for the powder injection moulding of ceramic brackets has been investigated. The preliminary study showed that the water soluble binder can be used for the fabrication of alumina and zirconia toughened alumina parts by PIM technique. Samples retain their shape after leaching of the PEG in water. The rate of PEG removal is also fast at the initial stage. Density and microstructure of samples depended on feedstock formulars and sintering conditions. The relationship between processing parameters, properties and microstructure of the injection moulded samples were systematically examined.

**Keywords:** ceramic injection moulding, alumina, zirconia, water soluble binder, dental bracket

## 1. INTRODUCTION

Alumina ($Al_2O_3$) ceramics are well known for their excellent properties such as high hardness, high mechanical strength, high wear resistance and bioinertness. However, the relatively low fracture toughness of alumina leads to its brittleness and limitation of applications. For this reason, zirconia is added into the alumina matrix to form zirconia toughened alumina (ZTA) [1]. Zirconia is one of the most promising biomaterials because it has very good mechanical and chemical properties suitable for medical or dental applications. Zirconia ceramics ($ZrO_2$) are also used in orthodontic dentistry. Zirconia stabilized with yttria ($Y_2O_3$) has been selected with the best properties for these applications [2]. Orthodontic brackets act like handles that hold the arch wires that move teeth. The brackets are typically bonded directly to the front of each tooth. They can be made of metallic or ceramic materials with a variety of styles and sizes. The shape of brackets is generally complex; therefore, powder injection moulding (PIM) is thought to be the most suitable technique for fabrication.

Powder injection moulding is a near-net shape manufacturing process that can produce large volume of products and also save the materials waste. PIM involves 4 main steps that are feedstock preparation, injection into the mould, debinding, calcination and sintering. Other ceramic injection moulding works employed wax based binder systems such as paraffin wax/polyethylene [3] paraffin wax/polypropylene/linear low-density polyethylene/stearic acid [4] that required the use of organic solvents during debinding step. In order to have an environmental friendly process, the water-soluble binder composed mainly of polyethylene glycol (PEG) system has been used in this PIM research at the Metallurgy and Materials Science Research Institute (MMRI), Chulalongkorn University. A variety of materials were successfully fabricated such as alumina, tungsten carbide and NiO-YSZ [5-7].

The aims of this work are to study on the effect of processing parameters for successful powder injection moulding of ceramic dental brackets using water soluble, PEG-based, binder and to investigate the properties of the specimens fabricated by this technique.

## 2. MATERIALS AND METHODS

Alumina powder (A32, Nippon Light Metal Co., Ltd, Japan) used in this work has a mean particle size of 1 μm. Zirconia with 3 mol% of yttria (TZ-3YE, TOSOH Corporation, Japan) has a mean particle size of 0.6 μm was also used. The ZTA powder mixture was composed of 80 wt% alumina and 20 wt% zirconia. The binder system is a mixture of a water soluble polyethylene glycol (PEG) 80 wt% and polyvinyl butyral (PVB) 20 wt%. The feedstocks were prepared with powder loadings of 52 vol% using a laboratory scaled pusher-type machine. A study of PEG removal in water was carried out at a 40 °C for several times interval. Specimens were weighed before and after leaching in water and these weight were used to calculate the percentage of PEG removal. Calcination and sintering were done in a furnace at temperature of 500 °C for 1 hour and 1600 °C with a soaking time of 2 hours, respectively. Density of the sintered specimens was determined using Archimedes method. Three-point bending test was used to measure flexural strength of as-moulded (green), as-leached (brown) and sintered specimens. Vickers hardness testing was done for sintered specimens. Scanning electron microscope was also employed for the microstructure evaluation of fracture surface.

## 3. RESULTS AND DISCUSSION

It was found that the mouldings specimens remain their shapes during and after leaching of the PEG binder in water. Figure 1 shows graph of PEG removal after leaching in water at 40 °C as a function of time. It was found that the rate of PEG removal decreased as the leaching time increased. The PEG molecules at surface easily interact with water and the PEG near surface can diffuse through the structure. This resulted in the rate of PEG removal was high at the beginning of water leaching and this was also in agreement with other previous findings [7, 8]. The PEG removal rate was then reduced due to the limitation of PEG diffusion outside from the samples. The pores occurred after PEG removal acted as pathways for the remaining binder (PVB in this case) to be removed during calcination in the following step. The PVB was removed by thermal debinding prior to sintering at high temperature.

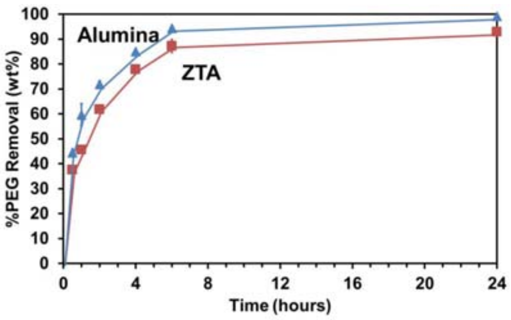


**Figure 1.** Graph of the percentage of PEG removal as a function of time for the $Al_2O_3$ and ZTA specimens.

The sintered density and hardness values of the specimens as well as the green, brown and sintered strengths of the specimens were presented in Tables 1 and 2, respectively. The theoretical density of $Al_2O_3$ and ZTA were 3.95 g/cm$^3$ and 4.25 g/cm$^3$, respectively.

**Table 1.** Density and hardness of specimens sintered at 1600 °C for 2 hours.

| Specimen | $Al_2O_3$ | ZTA |
|---|---|---|
| Density (g/cm$^3$) | 3.78±0.02 | 4.12±0.03 |
| Theoretical density (%) | 95.4 | 96.9 |
| Hardness (kgf/mm$^2$) | 1608±97 | 2093±154 |

**Table 2.** As-moulded, as-leached and sintered strength of specimens sintered at 1600 °C for 2 hours.

| Specimen | $Al_2O_3$ | ZTA |
|---|---|---|
| As-moulded strength (MPa) | 5.2±1.5 | 9.9±1.3 |
| As-leached strength (MPa) | 11.3±0.8 | 13.3±0.9 |
| Sintered strength (MPa) | 205.2±37.9 | 334.2±90.3 |

The sintered density of $Al_2O_3$ and ZTA specimens were 95.4 % and 96.9 % of the theoretical value, respectively. This can be implied that some porosities were in the specimens. However, in order to increase the density, specimens could be sintered at higher temperature. For the mechanical properties of sintered specimens, it was found that the flexural strength and hardness are strongly affected by zirconia addition (20 wt% in this work). The microstructural evaluation revealed that zirconia inhibited the grain growth of alumina. The ZTA specimens had flexural strength of 334.2 MPa while $Al_2O_3$ specimens had exaggerated grain growth and a flexural strength value was 205.2 MPa. The microstructure of sintered specimens is shown in Figure 2. In addition, the preliminary prototype of dental ceramic brackets fabricated by powder injection moulding are shown in Figure 3.

From this work, it was found that adding zirconia added in alumina enhanced the properties of the specimens. The results of this work was in agreement with previous findings where the mechanical properties (flexural strength value and fracture toughness) of specimens were improved by the addition of zirconia in alumina matrix [9]. This improvement was resulted from the stress-induced phase transformation in materials, i.e. from the tetragonal structure to monoclinic structure of the zirconia. This phase transformation has an associated volume expansion that creates compressive stress, which counters the tensile stress required for crack growth and therefore, increased the fracture toughness of the ZTA composite specimens.

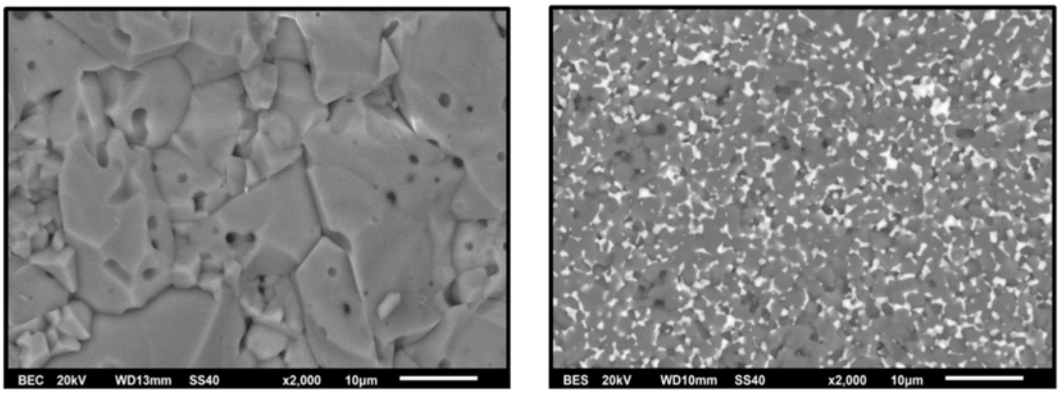


**Figure 2.** SEM micrographs of (a) alumina specimen (b) ZTA specimen.

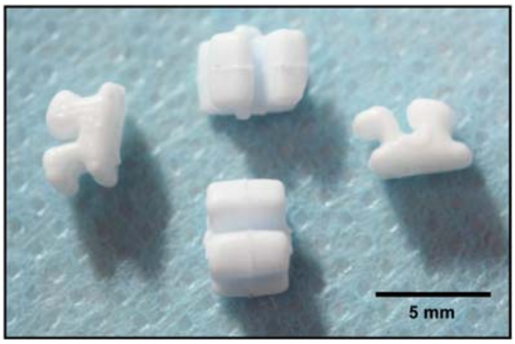


**Figure 3.** dental ceramic bracket fabricated by powder injection moulding.

## 4. CONCLUSIONS

This study focused on powder injection moulding process for dental ceramic brackets. The physical and mechanical properties and microstructures of powder injection moulded (PIM) alumina and zirconia toughened alumina (ZTA) specimens were investigated. The mouldings were successfully prepared using feedstocks containing a water soluble binder system. Water leaching could be employed for PEG removal prior to thermal debinding to remove PVB. The mechanical properties of sintered specimens that contain zirconia were higher than those of alumina specimens. The ZTA microstructure revealed normal grain growth with a small average grain size due to the added zirconia that plays an important role as a grain growth inhibitor. Whereas microstructure of alumina specimens had abnormal grain growth with a larger average grain size than that of the ZTA specimens. For this study, it was concluded that powder injection moulding can be employed in fabrication of dental ceramic brackets. The ZTA specimens provide preferable microstrusture and higher mechanical strength than those of alumina specimens prepared with the same condition.

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