

Fabrication and Characterization of Hydroxyapatite Reinforced Alumina-Based Foams

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ABSTRACT

Alumina (Al_2O_3) based porous ceramic materials are widely used in filters, sealing elements, refractors, and biomaterial applications. Among them, biomaterials have a great attention for artificial bone and filler materials. Hydroxyapatite (HA) is the most preferred calcium phosphate based powders to enhance the biocompatibility of the bioceramics. This study aims to fabricate HA reinforced alumina-based porous ceramics and investigate their mechanical properties. In this study, porous organic sponges were used as foaming template. The foam materials were sintered at different temperatures ($T_s=1600-1700-1750$ °C) for 2 hours. The microstructure of the foam materials was analyzed by scanning electron microscopy (SEM). The compressive strength of foams was determined by compressive test machine depending on heat treatment. From the results, the mechanical strength of ceramic foams improved with increasing sintering temperature. As a result of the analysis, it was determined that the compressive strength of foams increased from 0.1 to 0.4 MPa with increasing sintering temperatures.

KEYWORDS: Ceramic foam, Alumina, Hydroxyapatite, characterization, compressive strength

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I. INTRODUCTION

In recent years, ceramic foam materials were extensively studied by researchers due to its high melting point of ceramic materials, high corrosion resistance, high wear resistance, low thermal conductivity, low density and low dielectric properties of ceramic materials [1]. One of these materials is aluminum oxide (Al_2O_3) ceramics. Alumina (Al_2O_3)-based materials are fabricated in various microstructures (porous, dense structure) due to their chemical resistance and thermal stability. Alumina-based ceramics are widely used in many industrial applications such as biomaterials, biomedical-health applications, gas burning systems, thermal insulation application, diesel engine exhaust filtration, catalytic applications, sealing elements, cutting tools, refractors, porcelain, white goods, and light construction materials industry [2-4].

Three methods are generally used in the production of alumina-based ceramic foam. They can be classified as foam production by using a polymeric sponge, foam production with gel casting, and foam production by using spacer organics. Ceramic foams are produced by using ceramic powders in these three methods. In the foam production by using polymeric sponge method, produced ceramic mug covered the foam structures. After this process, drying and heat treatment were performed and then, porous ceramic structures were fabricated. In the gel casting method, foam structures were produced with using additional foaming agent by the mechanical effect. In the foam production by using spacer organics method, foam structures were fabricated by the use of pore-forming balls that were removed from the system by temperature [5-8]. Among the foam production methods mentioned, the most preferred method is the ceramic foams production method by using polymeric sponges. In this method, it is possible to produce ceramic foams with a pore size of less than 1 mm, which affects the mechanical properties of the foams. The major disadvantage of the alumina foams produced by this method is their low strength and low fracture toughness due to their wide pore size. Therefore, either reinforcement elements (fiber) are used or mechanical properties are controlled by sintering temperature to increase the mechanical properties. The sintering temperature is the most important process parameter affecting the porosity and mechanical properties of foams. Generally, the sintering temperature of alumina foams changed between 1450°C and 1700°C and their effects on mechanical properties was not studied detailed. Also, biocompatibility is very important than mechanical properties during applications. HA is a good candidate to increase biocompatibility, osseointegration due to its similar chemical composition with real bone [9-13].

In this study, metal ion (Ag-Zn) and hydroxyapatite (HA) reinforced alumina (Al_2O_3)-based foams were fabricated by polymeric foam method to develop the biocompatibility of foam. The microstructure and compressive strength of alumina-based foams were investigated with different sintering temperatures (1600, 1700, 1750 °C).

II. EXPERIMENTAL PROCEDURE

The fabrication scheme of alumina-based HA foams was given in Fig. 1. Firstly, hydroxyapatite (HA), alumina (Al_2O_3) and metal ion doped solid powders (% weight ratio: Al_2O_3 :HA-83:17) were added to pure water. Also, the organic binder was added up to 0.083% weight percentage of the total mixture. Then, HA and Al_2O_3 powders uniformly mixed and ground in ball milling during 15 h. After grinding, alumina based ceramic mixture was impregnated with the polymeric sponge and ceramic foams were fabricated. Alumina-based foams were sintered at a certain sintering temperature ($T_s=1600, 1700, 1750$ °C) and sintering time ($t_s=2$ h) after drying. The microstructure analysis of fabricated foams was performed by scanning electron microscope (SEM). The compressive strength of foams was determined by the compressive test unit (GUNT WP300). By using this test machine (compressive capacity: 2 ton), the load was applied manually.

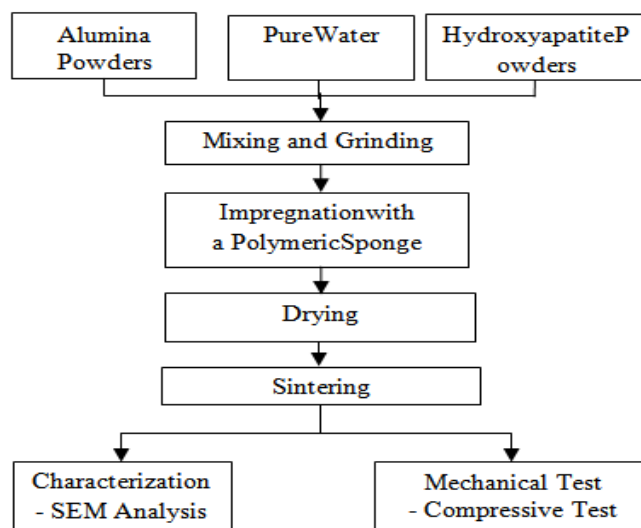


Fig. 1: Fabrication scheme of alumina-based HA foams

The rectangular test specimen (17x17x17 mm) was replaced the test machine for the compressive test. The samples sizes were written in the software. Data acquisition rate were set as per second in the software. The load was manually applied until the sample is deformed. The stress values were transferred to the computer via the software for each specimen. Hence, the maximum compressive strength value was determined for each foam specimen.

III. RESULTS AND DISCUSSION

The microscopic image of a stereo microscope for polymeric foam was given in Fig. 2a. As shown in the figure, the pore distribution of the foam varied between 300 and 700 μm which is similar to real bone. Also, it completely decomposed at 400°C from the green line termogravimetrik analyses (TGA) (Fig. 2b).

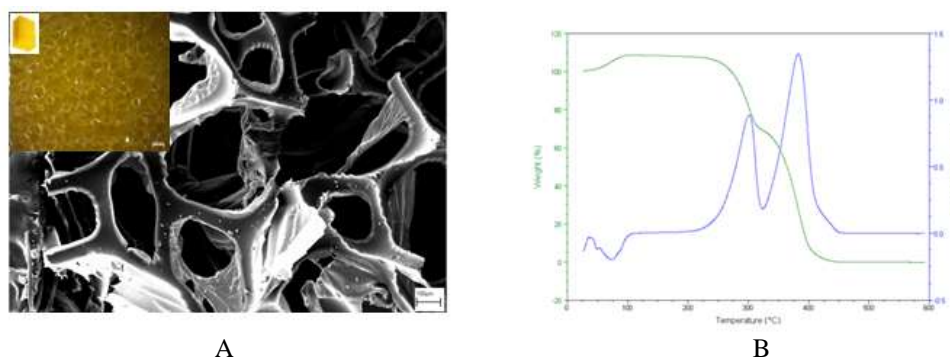


Fig. 2: The microscopic image (a) and TGA plot (b) for polymeric foam

The image of the alumina-based foams after drying and heat treatment was shown in Fig. 3. The foam shrank nearly 25-35% according to the sintering temperature (T_s). As a result of the density measurements, the porosity percentage decreased from 86% to 80% with increasing the sintering temperature (Fig. 4). This is due to the fact that the alumina foams exhibit higher shrinkage behavior by increasing the sintering temperature.

Increase in the shrinkage of the foam, the porosity size of the foam decreases. The decrease in the porosity has a positive effect on the fabrication of the foam according to the increase in the sintering temperature [9].



Fig. 3: The image of alumina-based foams after drying and heat treatment

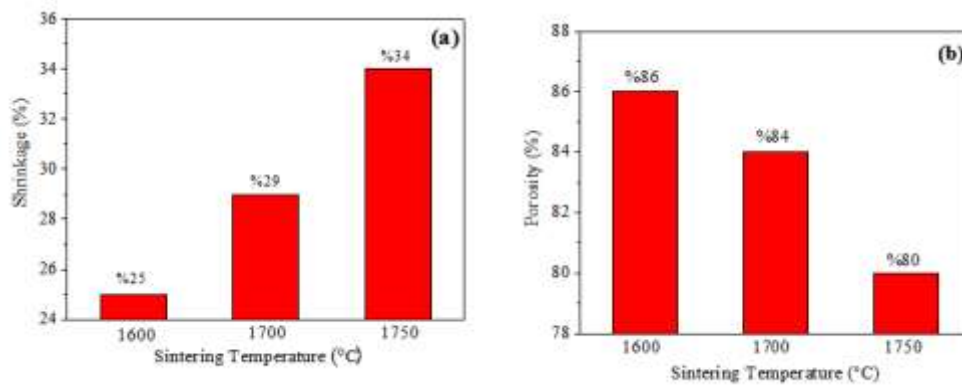


Fig. 4: Shrinkage (a) and porosity (b) quantity of alumina-based foams

Intergranular bonding and the porosity amount between grains are one of the most important factors affecting the strength of the foam in order to fabricate alumina foams. Also, the thickness of the strut wall and the intense sintering of the grains affect the mechanical properties of the foams positively [9, 10]. Therefore, the interaction between the grains joining the porous regions is controlled by the sintering temperature. The fracture surface scanning electron microscopy images of the metal ion doped alumina-based ceramic foams at the different magnifications (x100, x1000, x10000) were given in Fig. 5. At the certain sintering temperature ($T_s=1600^\circ\text{C}$) with the high magnification, no intense sintering between grains forming the porous regions was observed when the SEM images were investigated. From the SEM images, pores between the grains were seen clearly. Furthermore, it was determined that grains sintered intensively and the porosity between grains decreased with increasing sintering temperature. From the SEM images ($T_s=1750^\circ\text{C}$), it was seen that the bonding between alumina grains was strong. This leads to an increase in the strength of foams.

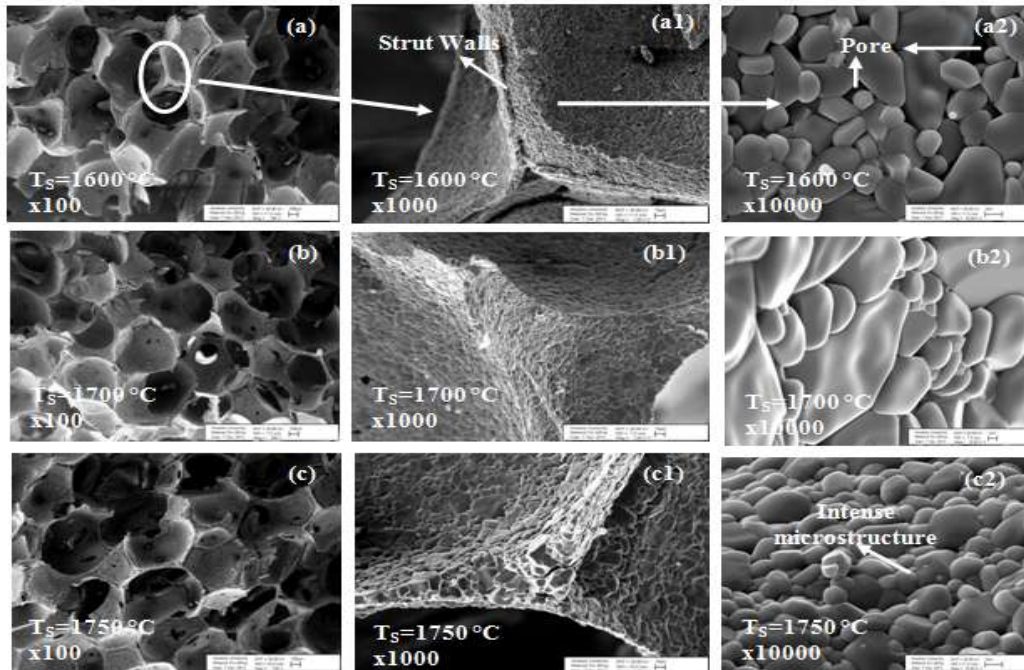


Fig. 5: SEM images of alumina-based foams with different sintering temperatures ($T_s=1600\text{ }^\circ\text{C}$ (a, a1, a2), $1700\text{ }^\circ\text{C}$ (b, b1, b2), $1750\text{ }^\circ\text{C}$ (c, c1, c2))

Fig. 6 gives the variation of compressive strength for alumina-based foams. The compressive strength of hydroxyapatite (HA) reinforced alumina-based foams was determined as nearly 0.11 MPa at $T_s=1600\text{ }^\circ\text{C}$. The compressive strength of the foam increased to 0.41 MPa when the sintering temperature increased to $T_s=1750\text{ }^\circ\text{C}$. The variation of the compressive strength confirmed the SEM images. There are three reasons for increasing strength. These reasons can be expressed as the amount of shrinkage, quantity of pore and grain growth after sintering [9]. The quantity of shrinkage increased with increasing sintering temperature. This causes the decrease in the amount of the pore. In addition, the interactions between grains with each other increased with increasing the sintering temperature. Hence, both a decrease in the gap between the grains and an increase in the sizes of the grains performed. This leads to an improvement in the compressive strength.

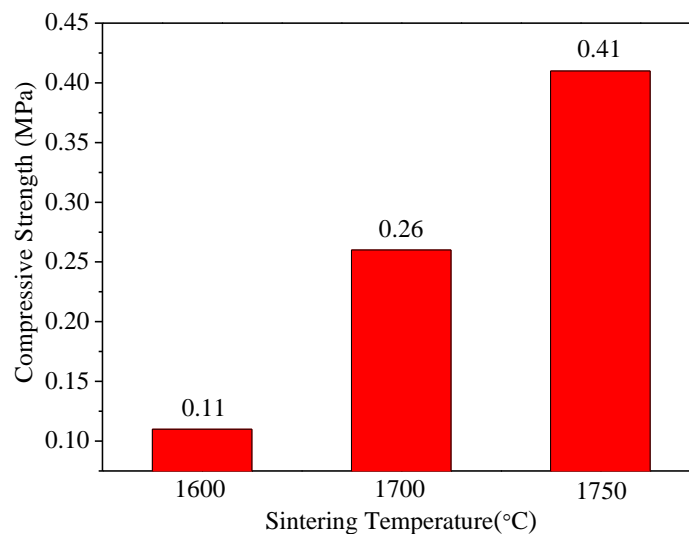


Fig. 6: Variation of compressive strength for alumina based foams

IV. CONCLUSIONS

In this study, hydroxyapatite reinforced alumina-based ceramic foams were fabricated by sponge method to develop the biocompatibility of foam. The test results showed that compressive strength of alumina-based foams increased with sintering temperature. Also, alumina grains were intensely bonded with increasing the sintering temperature from SEM analysis. The best compressive strength was obtained at a certain sintering

temperature ($T_s=1750$ °C). It was reported that the produced foams can be used in biomedical applications and filtration systems. It is aimed that the studies will continue to improve the mechanical properties and its biocompatibility of foams.

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