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Mechanical Properties of Dense Artificial Bone Fabricated by Powder Processing

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Abstract – Osseointegration and osteoconduction are essentials for artificial bone tissue production. Hydroxyapatite has been extensively used in orthopedic application due to its similar composition with bone. It is a highly biocompatible calcium-phosphate based bioceramic which could be shaped into bone like structure with powder processing techniques. Gradually changing porosity pattern of bone maintains both ease of transmission of bodily fluids and structural strength. Due to fracture type, in some instances bone tissue could not be recoverable and aligning large bone fractals requires dense bone tissue to place fixation apparatus. In this study, the aim is to produce a dense hydroxyapatite artificial bone and the effects of sintering duration and temperature were investigated. Metal ion doped calcium phosphates in hydroxyapatite form (<100nm) were used as powder. Two different types of Polyvinyl alcohol (PVA, with molecular weights of 10000 and 60000g/mol) were used as plasticizers to obtain strong green samples. The samples were sintered between 1000°C to 1200°C for 60-300 min. Hardness and compression test were performed to observe effect of the process parameters. From the SEM images and density measurements, highly dense samples were fabricated at 1200°C for 120 minutes. Compressive strength and hardness were enhanced up to 71 MPa and 4.5 GPa, respectively.

Keywords – Hydroxyapatite, Artificial bone, Bioceramic, Powder processing, Biomechanic

I. Introduction

Biomaterials have been used and engineered for almost five decades to treat, repair or to replace dysfunctioning organs and tissues. Engineering materials are needed to meet certain criteria to find use in vivo, besides strength by means of biocompatibility. Biocompatible material is a kind of material that does not produce an immunological or toxic response when exposed to any kind of living tissue and bodily fluids. Manufacturing a biocompatible implant is possible in many methods like powder processing and coating when it is necessary. By using conventional and unconventional manufacturing methods, it is possible to develop desired surface and inner geometric patterns. Therefore, as improvements in powder processing technology emerges the biomaterial varieties, the skeletal system has become a major attraction for biomechanics in context of artificial bones. Earlier applications with metallic and ceramic materials were performed poorly in terms of biocompatibility and blending into bone tissues.

Artificial bone should be biocompatible, osteoconductive and highly porous in general. Although porosity allows a high osteoconductivity rate, porous materials show much less strength than dense materials. Natural form of a bone consists of both dense and porous structures. Inner section of a bone is called cancellous bone and it is spongious, while outer section is dense and gives desired strength to the bone to sustain torsional and compressive forces mainly.

Hydroxyapatite [Ca10(PO4)6(OH)2] is one of the most stable forms of calcium phosphate and it is a major component that makes up about 65% of bone [1]. There are several methods of manufacturing or gathering hydroxyapatite and it is widely used in artificial bone studies because of its biocompatible nature. Osteoconductivity capability of hydroxyapatite makes available to have long-lasting bone implants and dissolution rate of about 10 wt% per year is significantly slower than the growth rate of newly formed bone [2]. Although being a natural bone element and excellently biocompatible, hydroxyapatite is hindered by its brittle nature [3].

The aim of this study is to manufacture a highly dense and biocompatible hydroxyapatite structure by changing sintering duration and temperature, besides observing plasticizer additive effect.

II. Materials and Method

In this study silver doped hydroxyapatite powder with <100 nm average particle size is used to manufacture samples. The reason behind choosing hydroxyapatite as material of this study is its extensive use as a biomaterial in many applications recently. Although porosity enhances osteoconductive properties [4], a dense structure of hydroxyapatite is capable of acting as a solid matrix phase for powder composites, thus brittle behavior of artificial bones could change to ductile through conscious decisions of reinforcement type.

The effects of sintering duration and temperature were observed by sintering green samples with 60, 120, 180 and 300 minutes of sintering and combinations with sintering temperatures of 1000, 1100 and 1200°C. Density and hardness measurements, compressive tests were applied to test samples and microstructural imaging was carried out by scanning electron microscope (SEM).

Density Measurement

Densities of sintered and nonsintered samples which have a regular geometric shape were calculated through measuring with precise measurement tools and porosity ratios were measured with Archimedes' scale. Densification rate effect of samples calculated by given formula.

$$\text{Densification rate, } \rho_{relative} = \frac{\rho_{final} - \rho_{initial}}{\rho_{initial}}$$

Hardness Measurement

Sintered samples' surfaces were prepared on a grinding and polishing machine and then Hardness measurements were carried out by a micro Vickers hardness tester properly. Every load was applied at least 5 different regions on the surface and the average value was taken as a representative value and a force of 250 gf was used.

Compression Test

In order to determine the compressive strength, sample dimensions were measured and a compression force was applied in the testing machine with an acceptable feed rate for ceramic materials. Results were obtained with a tailored software through computer.

Microstructural Imaging

On the purpose of microstructural imaging, a scanning electron microscope was used. In order to compare experimental and theoretical porosity, fracture surfaces of the specimens were scanned.

Sintering

Sintering is a temperature controlled phenomenon and heat treatment plays a key role on neck formation that enhances mechanical properties. Owing to the higher surface energy of nanoparticles, that kind of formations between particles occur easily [5].

III. Results

Densification rates, compression and micro Vickers hardness test results and SEM images are shown below. As given in Fig. 1, sintering duration and temperature have positive effect on the densification. The best compression behavior (75MPa) is observed at 1200°C for 2h. Similarly, the greatest hardness is observed as 467 HV (4.51 GPa) at same process parameters.

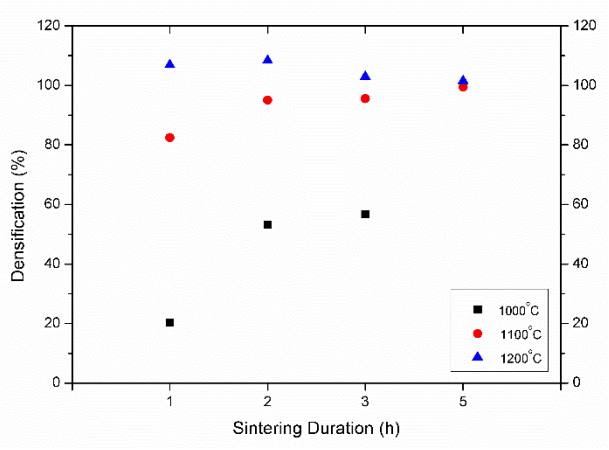

Fig. 1 Post-sintering densification rates of samples

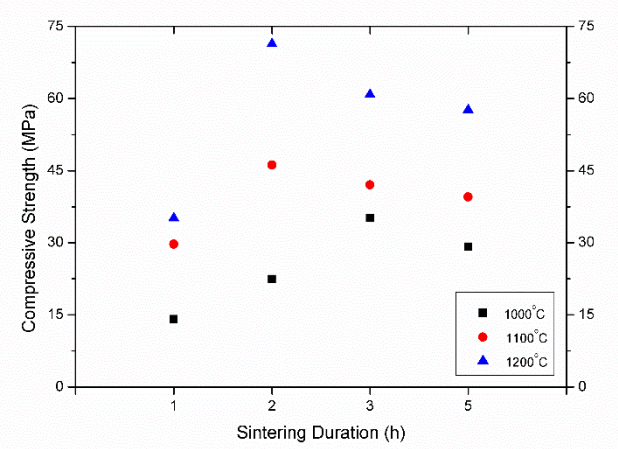

Fig. 2 Compression test results of samples

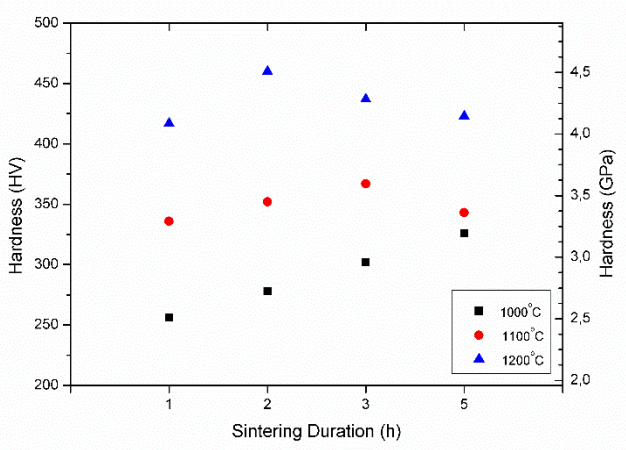

Fig. 3 Hardness measurements of samples

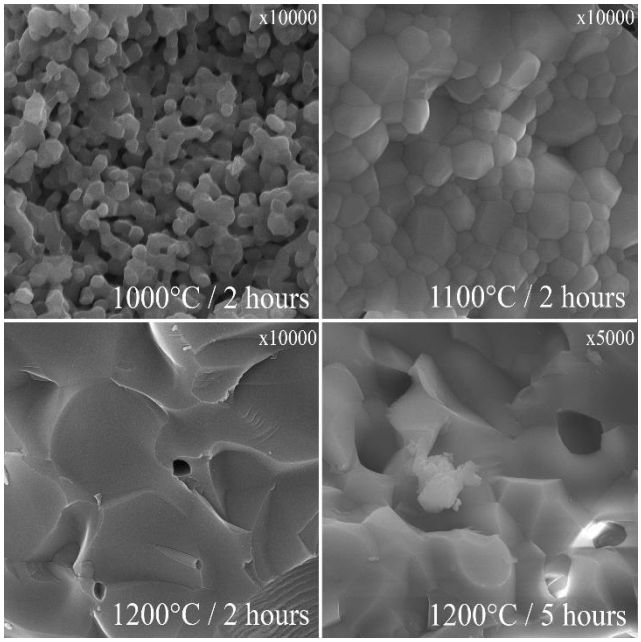

Fig. 4 SEM images of four different sintering regimes

IV. DISCUSSION

Particle size of hydroxyapatite powder used in this study is lower than 100 nm, therefore, as the smaller particle in size has a better atomic movement ability, higher sintering temperature causes a better heat treatment. It is not likely to observe necking and pore formation with lower sintering temperatures. Since particle size gets smaller, structural mechanic properties enhances due to grain boundary strengthening.

As it could be seen in Fig. 4 a porous structure is formed at 1000°C and also interparticle bridging is developed poorly. In addition to these, it could also be observed that increasing sintering temperature enhances density and material's mechanical properties by increasing necking and grain growth. This could be explained by particle diffusion mechanism. Since diffusion is a temperature controlled mechanism, at higher temperatures translation movement between nano particles occurs faster, thus results an advanced microstructure. On the contrary longer sintering durations results in an excessive grain growth that worsens mechanical properties.

V. CONCLUSION

In this study, the effects of sintering time and temperature were investigated. Metal ion doped hydroxyapatite form were used starting powder. From the SEM images and density measurements, highly dense samples were fabricated at 1200°C for 120 minutes. Compressive strength and hardness were enhanced up to 71 MPa and 4.5 GPa, respectively.

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