Fabrication of Porous Segments Using Ti-6Al-4V Chips for Orthopaedic Applications

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Abstract
Different methods have been evaluated for manufacturing the porous Ti6Al4V alloys according to decreasing stress shielding phenomenon and increasing mechanical compatibility between the metallic components and the host tissue. For this purpose, in this work Ti6Al4V alloy chips were pressed under 400 MPa pressure and then samples were categorized and heated into two groups at 1000 and 1150°C under a vacuum about 10⁻⁴ mbar. The results implied a porosity about 35%, significant reduction of density in prepared segments comparing to bulk ones and a reduction in the elasticity modulus and better matching with the host cancellous bone tissue. According to the results one can expect to manufacture porous Ti6Al4V orthopaedic and dental implants using the discussed method.

Keywords
Hard Tissue Replacement, Ti6Al4V, Chips of Alloy, Orthopaedic Implant

1. Introduction
Titanium (Ti) and its alloys are widely used in biomedical applications such as orthopedic implants for bone repairing [1] because of their excellent corrosion resistance and biocompatibility. High specific strength and good mechanical properties also make them suitable for using in the mentioned applications [2]. One of the issues that limits them for using in the recent applications is the biomechanical mismatching between implanted materials such as commercial purity (CP) Ti or Ti–6Al–4V and hard tissue because of their Young’s modulus differences [3]. It led to stress shielding phenomena which weakens the host bone. This can be overcome through the use of porous structures [4].

Paying attention to some requirements such as biocompatibility of the material, accurate design of the porous biomaterial and it's similarity to the surrounded tissue make them applicable for orthopedic implants. Porous biocompatible Ti6Al4V is one of the porous structure which has been developed it's using in the recent years [5]. They are different methods to fabricating porous components such as rapid prototyping [6], powder metallurgy [1], hollow spheres [7] and electron beam processing [8]. In all of them it should be considered that at first the minimum pore size suitable for bone replacement is larger than 100 µm [9], secondly, the suitable mechanical properties including a higher compressive strength and an appropriate Young’s modulus are essential [10]. At the present literature, a porous structure fabricated from Ti6Al4V chips was
Fabrication of Porous Segments using Ti-6Al-4V chips for orthopaedic applications, pp. 59-65

studied. For this purpose at first the metallic foams were manufactured by cold pressing and sintering of the chips in a cylindrical mold and then characterized. It is expected to promote interactions between the foam and the surrounding tissue. Surface pores facilitate the bone tissue to have a mechanical interlocking with the implant and the host bone, enhancing mechanical stability at the interface [11].

2. Material and experimental procedures

2.1 Removing pollution
Ti6Al4V alloy chips (figure 1) were used to achieve a porous structure in this work. To remove contaminants from the surface of the alloy, the chips were immersed in the Kroll’s solution ($4HNO_3 + HF + 5H_2O$). Then they were dried after washing with distilled water.

![Figure 1. The used Ti6Al4V alloy chips](image)

2.2 Sample preparation
The chips were placed horizontally and then vertically layer by layer in a steel mould with 10mm diameter. Each sample was cold pressed (SPECAC 15.011, 15 ton hydraulic press) under 400 MPa pressure. Then they were categorized and heated into three groups (the first group for 4 hours at 1000, with a vacuum about $10^{-4}$ Torr. the second group (examples of the first group) for 3h at 1150°C at the same atmosphere and the third ones for 2h at 1150°C in argon atmosphere controlled furnace (it should be noticed that the chips at the last samples had been milled for 17h with 200rpm under argon atmosphere before pressing). All of the samples heated and cooled with 10 k/min. Some of the samples were cut in both vertical and horizontal sections by the wire cut machine in order to achieve SEM and optical microscopic images.

2.3 Characterization
Micrograph images were performed by SEM (Philips, XL30, combined with EDS analysis) and optical microscopy (Carl Zeiss, Axiolab A, 450909). The phase constituents of samples were studied by XRD analysis. Compression tests were carried out on samples having 35% porosity at room temperature with 30 ton mechanical testing machine and stress-strain curves have been presented.
3. Result and conclusion

3.1 Density and porosity percentage of samples

The density of the samples were measured. According to the obtained density (2.8-2.9 gr/cm$^3$) in compare with the density of the compacted alloy (4.34 gr/cm$^3$), it reduced dramatically and became closer to the density of host tissue (0.1-1gr/cm$^3$ for cancellous bone and 1.8gr/cm$^3$ for cortical bone). Using equation (1) the percent of porosity is obtained 35% and 33% for the first and the second group of samples respectively.

$$\% \text{porosity} = \frac{\rho_b - \rho_s}{\rho_b} \times 100$$

where $\rho_b$ and $\rho_s$ are density of bulk alloy and porous segments respectively. The obtained porosity (33-35%) is also suitable for insertion in the bone tissue.

3.2 Morphology

The SEM images of samples 1 and 2, respectively, are shown in figure 2. Images of horizontal and vertical sections of the samples are also shown in Figures 3 and 4. The Images implied presence of open cell and also close cell porosities in the microstructures. Size of porosities is various and their distribution is Heterogeneous. Connection between the chips is established. This led to the creation of components with a strength near to a cancellous bones. In addition, most of pores are interconnected that is useful to ingrowth the host tissue to the interconnected pores and allows mechanical interlocking of bone with the implant and making it fixed.

The distribution of the pores in samples belongs to group 3 is more homogenous (figure 5). It is related to the smaller size chips. The same chip size was obtained by grinding in a short period of time and low rotation speed, under a protective atmosphere of Ar (99.998% pure) in a planetary ball mill. But in a long time and high-speed milling unavoidably increase the oxidation of samples. On the other hand milling process of chips to uniform the size can result in uniformity of size and distribution of pores.

Figure 2. SEM micrographs of the surface of samples group a) 1 and b) 2
3.3 XRD analysis

XRD patterns of the used chips after milling, the porous component (sample 3) before and after metallography are illustrated in figure 6. The results show that sintering the pressed sample make the surface oxidized that is removed after polishing. The thermo mechanical processing of the sample makes the peaks related to Ti6Al4V sharper. The layered microstructure of $\alpha$ and $\beta$ phases is obviously visible in the image (Figure 7).
3.4 Mechanical test

Mechanical properties were investigated via compression test on porous samples with length to diameter ratio of 6/10 and 35% porosity. The compressive stress-strain curve is shown in figure 8. The curve includes three region, the first is linear elastic region. Elastic modoulus was measured from slope of linear elastic region. Average of elastic modoulus was 1.52 GPa. The second area of curve is plateau stress region that plastic deformation has occurred on the pore walls. The plateau stress average for samples was measured about 0.406 MPa. The third area is densification region that cell walls densified and sample behave as a bulk sample. Hence the sudden increase in the slope of the stress-strain diagram occurs after the second stage.
4. Conclusion

In this project, the porous segments were fabricated successfully by pressing and sintering of Ti6Al4V alloy chips. Among various methods for fabricating porous titanium alloys, using chips of titanium alloy as primary materials and sintering process is suitable and economic technique to produce porous samples having good micro and macroscopic structure and mechanical properties for orthopaedic applications. Samples group 1 and 2 were achieved from as received chips without milling that have heterogeneous pore distribution while milling process with low rotation speed and short time can decrease the chips size without oxidation compounds arrival and these qualities lead to uniform size and distribution of pores.

5. References