THE EFFECTS OF HAP ADDITION ON THE PROPERTIES AND MICROSTRUCTURE OF F-75/HAP COMPOSITES

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Co-Cr-Mo alloys (F-75) are widely used in biomaterials implants due to their high performance in mechanical properties, and corrosion while hydroxyapatite (HAP) powders have been used as filler because HAP is one of the most effective biocompatible materials with similarities to mineral constituents of bones and teeth. This research examines the effects of HAP addition on the properties and microstructure of F-75/HAP composites fabricated by powder metallurgy. HAP powders (chemical formula $Ca_{10}(PO_4)_6(OH)_2$) have been added to Co-Cr-Mo alloys in the composition of 0, 2, 6 and 10 wt.%. The mixtures were then milled at 154 RPM, before cold compacted at 550 MPa. Sintering was performed at 1150^{0}C in a tube furnace with sintering time set to two hours. Physical properties were measured by means of density and porosity. A scanning electron microscope equipped with energy dispersive spectrometry was used for determination of microstructure and interface analysis. The composites with higher addition of HAP showed better density and porosity than those with lower addition of HAP and suitable for biomedical application. Line scanning analysis showed that, as the amount of HAP is increased, there was good bonding between HAP particles and matrix F-75.

Keywords: Co-Cr-Mo alloys, Hydroxyapatite, Powder metallurgy, Microstructure

INTRODUCTION

Biomaterials are used to make devices to replace a part or a function of the body in a safe, reliable, economic, and physiologically acceptable manner [1]. They have been trusted to be used in human or animal body due to their excellent biocompatibility, including replacement of a body part, which has lost function due to disease or trauma, to assist in healing, to improve function, and to correct abnormalities. According to Geetha *et al.* [2], three major factors that can lead to the success of a biomaterial or implant are (i) the properties (mechanical, chemical and tribological) of the biomaterial, (ii) biocompatibility of the implant and (iii) the health condition of the recipient and the competency of the surgeon.

Most of the metallic materials used cobalt based alloys, austenitic stainless steels, titanium and titanium base alloys and magnesium based alloys. Co-Cr-Mo (F-75) alloys are widely used in implants such as prosthetic hips and knees due to their mechanical properties, good wear, and corrosion resistance as well as biocompatibility [3-4]. The application of metals and alloys in orthopaedic is very important, as they play a very predominant role in fulfilling almost every difficult factor that arises in implant applications [5]. Calcium based biomaterials like hydroxyapatite (HAP with chemical formula $Ca_{10}(PO_4)_6(OH)_2$), have been used since 30 years ago in medicine due to their excellent biocompatibility, structural and chemical similarity with bone mineral compositions [6]. As reported by Navarro *et al.*, 2008 [7], the application of this ceramic material as bone substitutes started around the 1970s and has mainly used as bone defect fillers. In order to improve its physical properties, without deteriorating its biocompatibility, HAP could be used in combination with another metal/ceramic phase [6].

With this background, the main goal of this research is to fabricate a composite material of Co-Cr-Mo (F-75) alloy as a matrix with HAP as reinforcing and bioactive phase using powder metallurgy route. The paper also describes the effects of HAP addition to F-75 in terms of physical properties and also microstructure of this composite.

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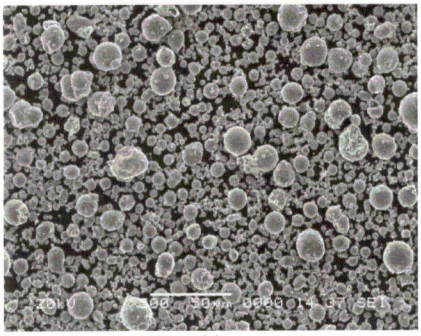
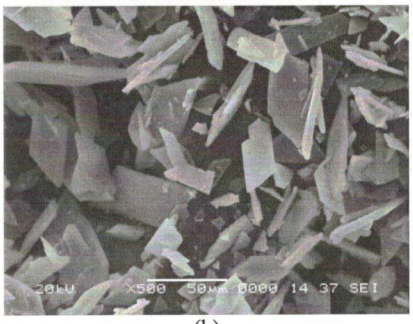
MATERIALS AND METHODS

The lab work presented the fabrication of composite Co-Cr-Mo powder filled with hydroxyapatite by powder metallurgy method.



(a)



(b)

Fig. 1. Morphology of the particles (a) Co-Cr-Mo (F-75) and (b) HAP at 500x magnification.

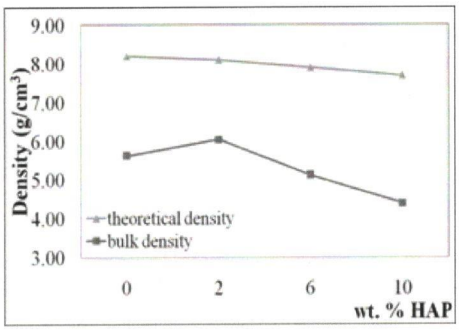
Fig. 1 shows the morphology for F-75 and HAP powders at 500x magnification. It can be seen that F-75 (Fig. 1 (a)) alloy powder exhibiting nearly spherical particles. F-75 was obtained in powder form (particle size of 22 micron) from Sandvik Osprey Ltd, UK. HAP powder (Fig. 1 (b)), purchased from Merck Company, revealed a flaky-acicular shape. The lab work was carried out on Co-Cr-Mo powders with the addition of 0, 2, 6 and 10 wt. % of HAP. 3 wt. % stearic acid was added as a binder.

The samples were milled in rotary milling machine for 20 minutes at 154 rpm. The samples were cold compacted under the pressure of 550 MPa between hardened steel dies and then sintered at sintering temperature of 1150^{0}C for 2 hours in a tube furnace under argon atmosphere. The heating rate was set at 3^{0}C/min up to the temperature of 400^{0}C and soaking for 30 minutes. The heating rate was then increased to 5^{0}C/min up to sintering temperature of 1150^{0}C and soaking for two hours before cooling down to the room temperature. The density and porosity of the sintered samples were estimated by Archimedes principle method.

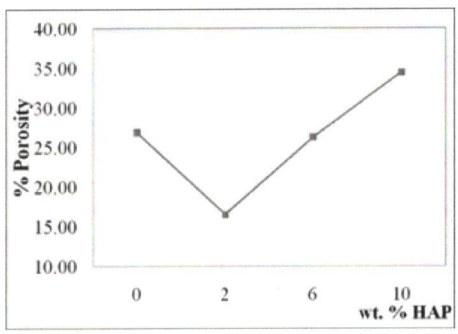
Microstructural observation was carried out using scanning electron microscope (SEM) (model JEOL, JSM-6420LA) equipped with EDS (energy dispersive spectrometry) elemental analysis equipment. For SEM, the sample was prepared by the standard metallographic methods of wet rotary grinding on a series of SiC papers (240, 400, 600, 800 and 1200 grit). The composites were then polished to produce a deformation free surface that is flat, scratch free and mirror like in appearance. The samples were polished starting with 6 micron diamond pastes (BUEHLER, USA), followed by 3 micron and 1 micron, on a soft napped cloth (BUEHLER, USA). The polished samples were again cleaned in ultrasonic cleaner with distilled water and dried by using a dryer. A line scan technique was used to analyze the elements in the microstructure [8] and the bonding between HAP and F-75.

RESULTS AND DISCUSSIONS

Physical properties



(a)



(b)

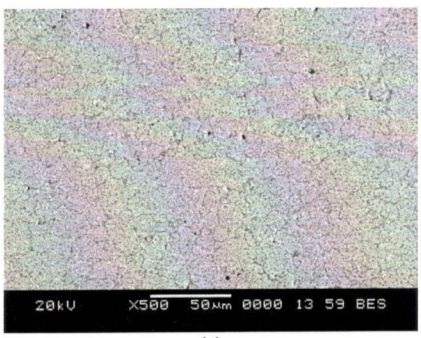
Fig. 2. The results of (a) theoretical density and bulk density (b) % of porosity for the samples sintered at 1150^{0}C, with different wt. % of HAP

The influence of HAP addition on the bulk density and porosity of the F-75/HAP composites after sintering at 1150^0C is presented in Fig. 2 (a) and Fig. 2 (b). The theoretical density (Fig. 2 (a)) of the composites was calculated from the following formula [9]: X% × the theoretical density of the Co–Cr–Mo alloy + Y% × the theoretical density of HAP. The theoretical density of the Co–Cr–Mo alloy is 8.2 g cm^{-3} whereas the theoretical density of HAP is 3.16 g cm^{-3} [10]. If the percentage amount of X is 98 wt. % of the F-75 alloy, then the value of Y is 2 wt. % of HAP.

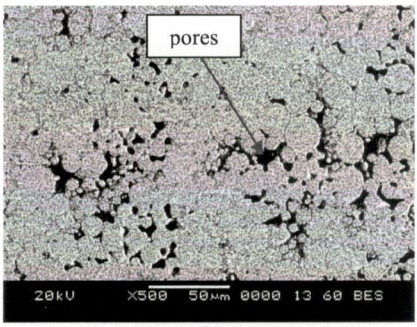
In Fig. 2 (a), the bulk density of composite is increased when 2 wt. % of HAP is added to pure F-75 alloy but decreased when higher amount of HAP is added. The highest value of bulk density is obtained for the sample F-75/2 HAP (with 2 wt. % HAP) which is 6.0489 g/cm^3. The relatively low density of the samples, due to the addition of HAP can be explained by internal friction of the composite powder increases which hinder consolidation of the material [9]. So, with more addition of HAP, the composite becomes more difficult to consolidate and results to low density.

In biomaterials, pores are necessary for tissue formation, because they allow migration and proliferation of cells, as well as vascularization [12]. Fig. 2 (b) presents the plot for percent of porosity calculated from the value of density in Fig. 2 (a). The percent of porosity plot shows that the porosity value increases with increasing HAP content. According to the plots, F-75/2HAP showed the lowest percentage of porosity (16.51%).

Microstructural Observation



(a)



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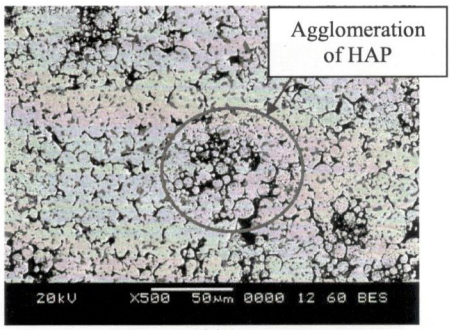
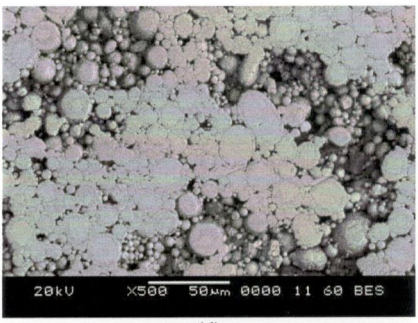


(c)



(d)

Fig. 3. The microstructure of (a) F-75, (b) F-75/2HAP, (c) F-75/6HAP, and (d) F-75/10HAP composites sintered at 1150^0C.

The SEM micrograph, Figure 3, shows that addition of higher amount of HAP created more pores in the microstructure. Many small pores can also be seen in the micrographs. These pores are associated with agglomeration of HAP as shown in Figure 3 (c). This is consistent with the increase in porosity with increase in HAP content as shown in Figure 2 (b). These homogeneous pores are suitable for biomedical application [13].

The composite with lower amount of HAP shows higher density value (Figure 2 (a)). This shows that during heat treatment, diffusion reaction occurred between the F-75 powder and HAP particles [9].

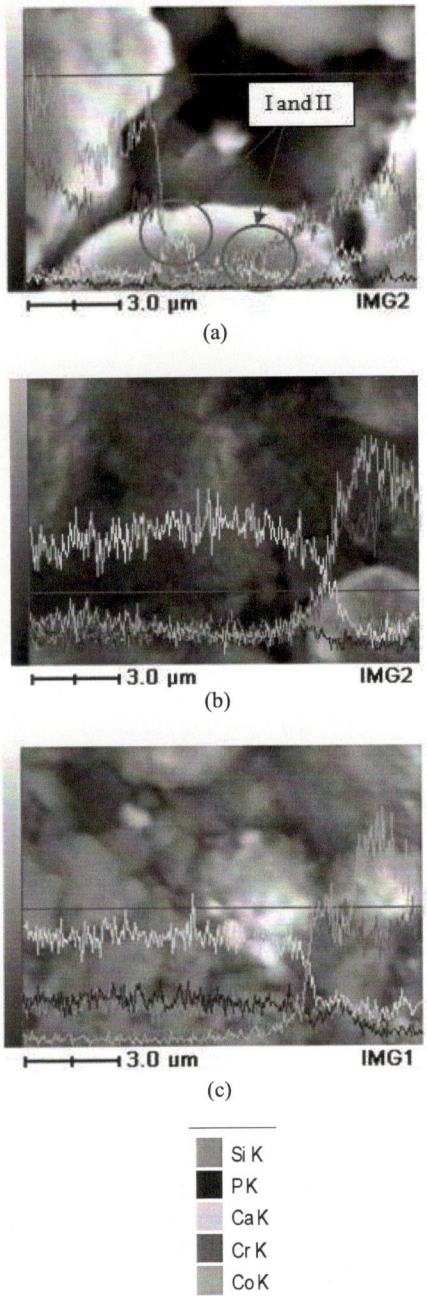


Fig. 4. SEM- EDS Line scan of sample (a) F-75/2HAP, (b) F-75/6HAP and (c) F-75/10HAP.

SEM-EDS analysis (Fig. 4) revealed that two main metallic powder elements (Co and Cr, presented in light blue and purple line colour) react with Ca from HAP (in yellow line) and diffuse towards the amorphous structure of reinforcing phase (HAP). Therefore, it can be summarized that bonding between two composites constituents exists that influences the density and properties of the specimens [9]. From Fig. 4, we can see that the composite with 2 wt. % of HAP (F-75/2HAP) shows better bonding. This sample exhibits two bonded area (indicated as I and II) suggesting good bonding between F-75 and HAP [9].

CONCLUSIONS

The F-75/HAP composite material has been successfully prepared using powder metallurgy method. After sintering at 1150^0C, F-75/HAP composites with good properties were obtained with HAP addition between 2-6 wt. %. The microstructural observation shows that more addition of HAP can create more pores. Good bonding between HAP particles and F-75 was observed for the sample with HAP addition between 2-6 wt. %.

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