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Effect of Sintering Temperature on The Adhesion Quality of Hydroxyapatite on Porous Tantalum for Cancellous Bone Implant Application

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Abstract
Porous tantalum has been recently recognized as a conventional orthopedic implant for bone substitute applications besides stainless steel, cobalt-chromium, titanium, and other metallic alloys. Porous tantalum has excellent mechanical and osseointegration properties similar to cancellous bone structure. To optimize the utilization, the dip coating technique was applied to coat porous tantalum with hydroxyapatite (HA). X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques investigate the coating characterization. HA is a bioceramic material that used for bone substitutes due to its chemical and structural similarity to bone minerals. Coated porous tantalum requires the sintering process of porous tantalum to adhere to HA. During the sintering process, various temperatures (650°C, 750°C, 850°C, 950°C, and 1100°C) were used to determine the optimum temperature for porous tantalum coated with HA. The heating rate is 1°C/1 min and the holding time is 60 minutes. The result shows that the optimum temperature of HA-coated porous tantalum is at 850°C. The morphology structure of the HA-coated porous tantalum shows that the adhesion between porous tantalum and HA is in good condition. The element in the HA-coated porous tantalum shows that the existence of HA is high.

Keywords: Hydroxyapatite, Porous Tantalum, Sintering Temperature, Cancellous Bone

1. Introduction

The development of Porous tantalum is an open-cell tantalum structure with an appearance similar to cancellous bone [1]. Porous tantalum is a transition metal that remains relatively bioinert and the porous designs have been developed to enhance biological fixation to the bone implant. Biological fixation is defined as the process by which prosthetic components become firmly

bonded to host bone by ongrowth or ingrowth without the use of bone cement [2]. To achieve better biocompatibility with bone, metal implants are coated with bioceramics that have been used to modify the surface of implants [3].

The similarity of chemical structure between Hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$, and natural bone makes it one of the most attractive materials to treat and replace the broken parts of human bones due to its good biocompatibility and bioactivity [4]. implants using hydroxyapatite as coatings and porous tantalum as substrates become the focus because of the combination of superior biological and mechanical properties [5]. Hydroxyapatite also can easily be composed at a certain temperature, determination on the optimum temperature is essential for the purpose of sintering [6].

Cancellous bone is less dense than compact bone and is composed of a honeycomb-like network of bones called trabeculae. Cancellous bone actually looks like a sponge. Cancellous bones are designed to bear stress caused by bending or stretching [7-8]. Damage to cancellous bone can be treated with implant.

Implant materials are designed to stay in the body permanently and can cause complications such as allergy and sensitization when used for temporary applications. An implant has to remove after the tissue is healed is not preferable either since there are always risks and cost issues involved with extra surgery. In order to overcome this problem, biocompatible material would eliminate these risks and the cost for secondary surgeries. Biocompatibility defined as the ability of a material to perform with an appropriate host response in a specific application [9-12]. The success of a biomaterial or an implant is highly dependent on three major factors; (i) the properties (mechanical, physical, and chemical) of the biomaterial (ii) biocompatibility of the implant and (iii) the health condition of the recipient and competency of surgeon [13 - 14].

Tantalum has been widely used in clinical applications Tantalum has been used as a radiographic bone marker, cranioplasty plates, pacemaker electrodes, gastrointestinal stents, dental reconstruction, vascular clips, spine surgery, sutures, and wire, foil, and mesh for nerve repair and various applications for porous tantalum in bone substitutes [15-17].

Porous tantalum has open-cell structure of repeating dodecahedrons that fabricate via carbon vapor deposition/infiltration of commercially pure tantalum onto vitreous carbon scaffolding. Clinical and histological evidence from retrieved implants clearly demonstrate that these porous surfaces enhance bone tissue in-growth and are effective in supplementing the stability of the implant by biological fixation [18-19].

Properties of Tantalum elastic modulus is 185 Gpa, yield strength is 165 MPa, elongation is 40%, tensile strength is 205 MPa, density 16.9 g/cm^3, Melting Point 3000^{o}c and hardness is 100 Hv [20]. Porous tantalum used also can cause adverse effect to the surrounding tissue that resulted from the metallic ion released. In order to make metal surfaces interact to the surrounding tissue (bioactive), coating calcium phosphate especially hydroxyapatite have been used to modify the surface of implants [21].

Implants using hydroxyapatite as coatings and porous tantalum as substrates become the focus because of the combination of superior biological and mechanical properties. Hydroxyapatite coatings can not only benefit to the building-up of new tissue but also fix the implants inside the body and protect surrounding bone against metal-ion release from metallic implants [22-23].

Coating is a covering that is applied to the surface of an object, usually referred to as the substrate, dip coating techniques can be described as a process where the substrate to be coated is immersed in a liquid and then withdrawn with a well-defined withdrawal speed under controlled temperature and atmospheric conditions [24-26].

2. Materials and Methods

The methodology throughout this project with the flow chart and work breakdown as well as the materials used. The method starting from samples preparation, samples process, and lastly data analysis. Flow chart shown in figure 1 that is.

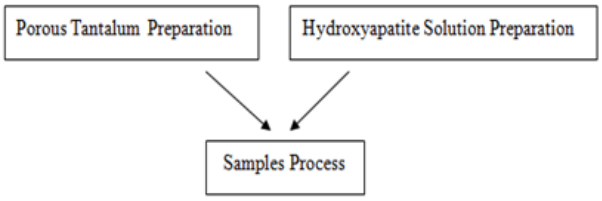

Figure 1. Main flowchart of the project

2.1 Porous Tantalum Preparation

Samples of porous tantalum with radius of 1cm will be use as substrate of coating as seen in Figure 3.3. As show in Figure 3.4, these samples were

ultra-sonically cleaned in immersed acetone, ($(CH_3)_2CO$) for 15 minutes at temperature = 30°C. Cleaning process is required for remove the foreign material or dust that contain in the samples. After that, these samples were washed by using distilled water and then were dried in oven for one hour with temperature at 40°C. Flowchart of porous tantalum preparation shown in figure 2 that is.

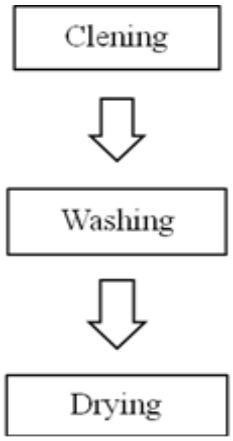

Figure 2. Flowchart of porous tantalum preparation

2.2 Hydroxyapatite Solution Preparation

Illustrates that the hydroxyapatite HA ($Ca_{10}(PO_4)_6(OH)_2$) solution were prepared by mixing 40 ml of ethanol, (C_2H_5OH) and 5g of hydroxyapatite powder which was stirred together using magnetic stirrer. The hydroxyapatite solution is used for the dip coating of the sample. Method to prepare hydroxyapatite solution shown in figure 3 that is.

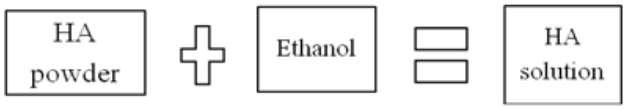

Figure 3. Method to prepare hydroxyapatite solution

2.3 Sample Process

A belt and pulley type apparatus was built and used in this study for dip coating process. The apparatus had two way electronic switch, to descend and ascend the porous tantalum samples immersed into the Hydroxyapatite solution at constant speed 150mm/min for three times. The samples coated with the hydroxyapatite solution were immediately dried in air oven with temperature at 100°C. Lastly, the samples are sinter in a vacuum furnace at various temperatures starting from 650°C, 750°C, 850°C, 950°C, and 1100°C and dwelled for 1 hours, using heating and cooling rates of 1°C/min. The coated porous tantalum was characterized by using scanning electron microscope and Energy Dispersive X-ray. Flowchart of samples process shown in fig 4 that is.

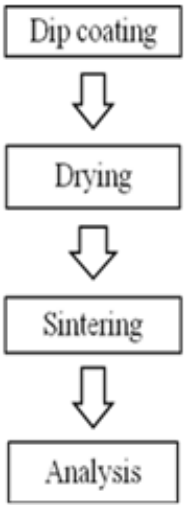

Figure 4. Flowchart of samples process

3. Result and Discussion

The analysis of coated porous tantalum with hydroxyapatite by using Scanning electron microscope and Energy Dispersive X-ray.

3.1 Scanning Electron Microscope (SEM)

In this study, trials of different temperature were used to find the optimum sintering temperature for porous tantalum coated with HA. Physical observation was done in order to evaluate the physical appearance of coated porous tantalum after sintering process. Meanwhile, SEM observation revealed the morphologies of the surfaces of coated porous tantalum. Figure 5a shows that the uncoated porous tantalum as control sample and interconnected structure of the pores that has similar structure in cancellous bone was shown in Figure 5b. Porous surface can enhance bone tissue ingrowth and are effective in supplementing the stability of the implant by biological fixation.

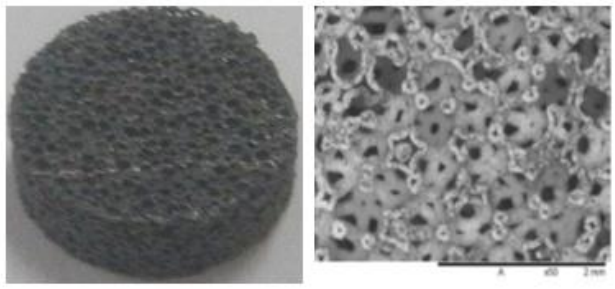

Figure 5. (a) Uncoated Porous Tantalum (b) SEM image

Sintering process was performed at different temperature (650°C, 750°C, 850°C, 950°C, 1100°C), it can be seen that the physical observation and the morphology of coated porous tantalum from Figure 6 until Figure 10. At 650°C, the pores structure can be seen and some of the pores are covered by hydroxyapatite as shown in Figure 6b. The adhesion between them is not strong due to the cracking when the sample was contacted.

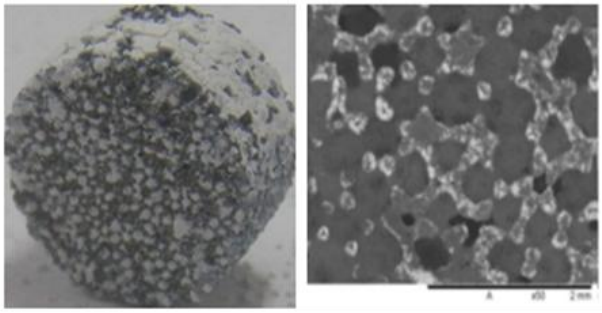

Figure 6. (a) Physical observation and (b) SEM image at 650°C

From Figure 7, it can be seen that the hydroxyapatite covered the porous tantalum more completely at sintering temperature 750°C compared to previous temperature. This sample also does not adhere completely because the sample still cracking when contacted.

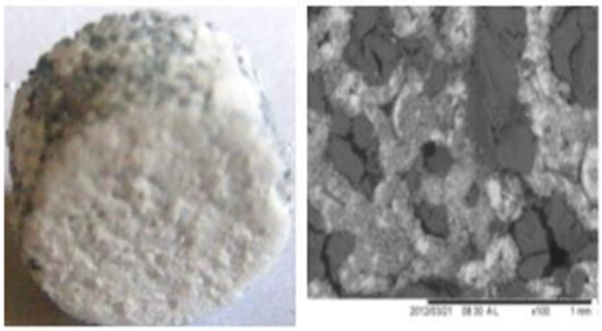

Figure 7. (a) Physical observation and (b) SEM image at 750°C

Hydroxyapatite covered porous tantalum completely at sintering temperature 850°C as shown in Figure 8. It can be shown that the optimum sintering temperature for hydroxyapatite adheres with porous tantalum is at 850°C. Adhesion between hydroxyapatite and porous tantalum are strong at this temperature because there are no cracking when contacted.

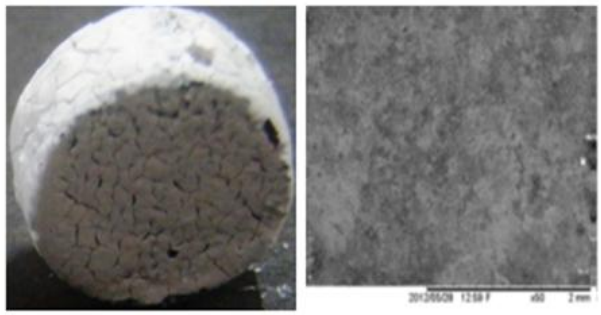

Figure 8. (a) Physical observation and (b) SEM image at 850°C

Figure 9 and 10 show that the morphology of inner porous tantalum at temperature 950°C and 1100°C respectively. The results show that the corroded structure of coated porous tantalum. That is because of the mechanical properties changes of hydroxyapatite and porous tantalum at higher temperature.

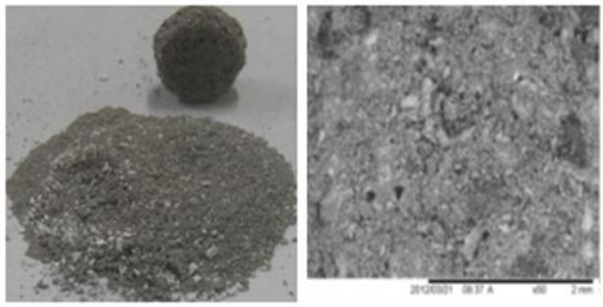

Figure 9. (a) Physical observation and (b) SEM Image at 950°C

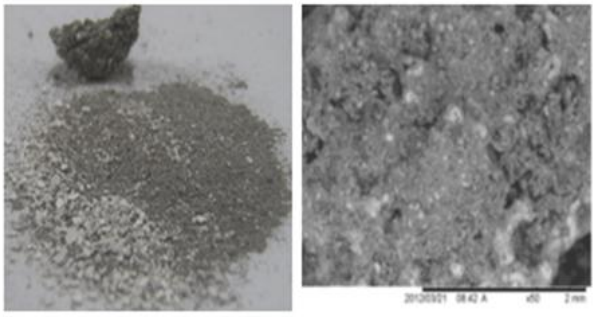

Figure 10. (a) Physical observation and (b) SEM Image at 1100°C

3.2 Energy Dispersive X-ray (EDX)

The weight of element consists in the samples can be evaluate by using the energy dispersive x-ray machine. Figure 11 until 16 show that the result of weight element consists in the coated porous tantalum at different temperature. For the control sample, pure porous tantalum consist about 53.18% weight of tantalum as seen in Figure 11. In this study, consideration in existence of tantalum, phosphorus and calcium elements only focused.

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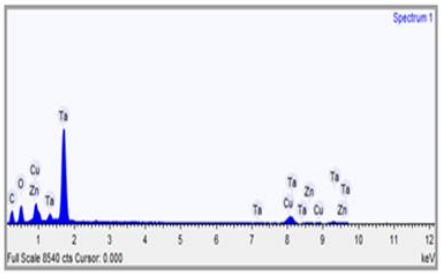
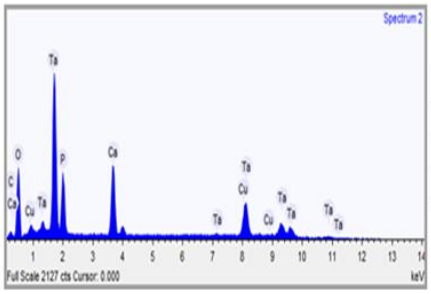
Actually, there other elements consist in the sample such as carbon, oxygen, copper, and rubidium.



Element	Weight %
Tantalum	53.18

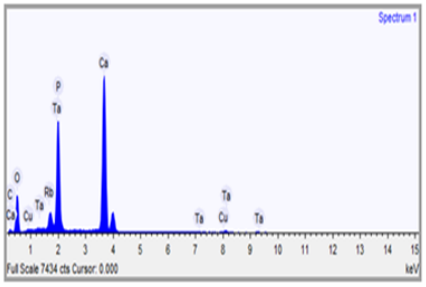
Figure 11. EDX result for uncoated porous tantalum

From Figure 12 and 13 it can be compared that the element of calcium and phosphorus (hydroxyapatite element) at temperature 650°C is less than compared to the temperature at 750°C. It means that the hydroxyapatite are not covered the porous tantalum after sintering process. The result is due to the cracking when the sample was contacted.



Element	Weight %
Phosphorus	8.12
Calcium	12.52
Tantalum	39.63

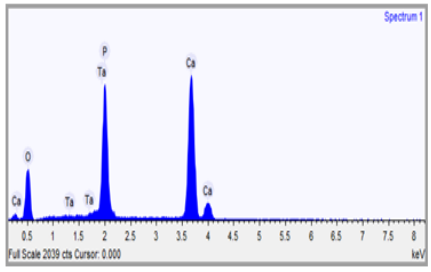
Figure 12. EDX result of coated porous tantalum at 650°C



Element	Weight %
Phosphorus	11.56
Calcium	20.03
Tantalum	23.15

Figure 13. EDX result of coated porous tantalum at 750°C

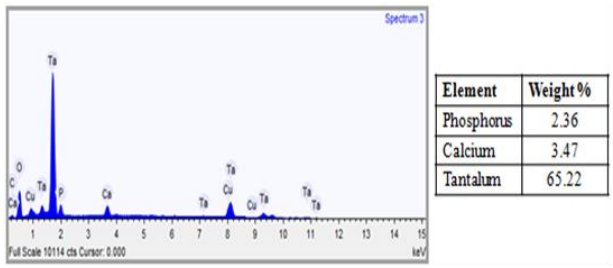
Hydroxyapatite covered the porous tantalum strongly when sintering temperature at 850°C. The weight of hydroxyapatite at that temperature is high which resulted in the successfully coated porous tantalum. It can be prove in EDX results as shown in Figure 14.



Element	Weight %
Phosphorus	18.64
Calcium	32.47
Tantalum	0.65

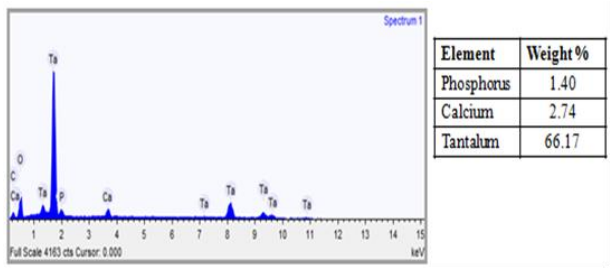
Figure 14. EDX result of coated porous tantalum at 850°C

Sintering temperature above 850° C caused the weight of tantalum increase rapidly as show in Figure 15 and 16. Its means that the there are no hydroxyapatite coated at the porous tantalum. It shows that the unacceptable sintering temperature of the coated porous tantalum.



Element	Weight %
Phosphorus	2.36
Calcium	3.47
Tantalum	65.22

Figure 15. EDX result of coated porous tantalum at 950°C



Element	Weight %
Phosphorus	1.40
Calcium	2.74
Tantalum	66.17

Figure 16. EDX result of coated porous tantalum at 1100°C

3.3 Weight Versus Temperature

Figure 17. shows that the graph weight of elements consists in coated porous tantalum versus temperature. It summarize that the overall observation of the tantalum, calcium and phosphorus consist on the coated porous tantalum.

Coated porous tantalum requires the sintering process of porous tantalum to adhere to HA. During the sintering process, various temperatures (650°C, 750°C, 850°C, 950°C, and 1100°C) were used to determine the optimum temperature for porous tantalum coated with HA. The heating rate is 1°C/1 min and the holding time is 60 minutes.

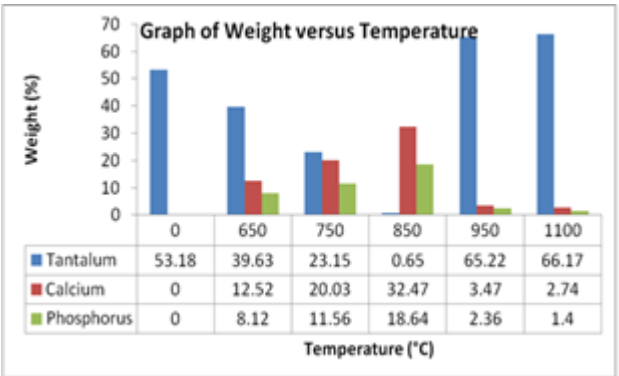


Figure 17. Graph weight of elements consist in coated porous tantalum versus temperature

Show in Figure 17 that is at temperature of 850°C, the weight of tantalum is 0.65%, the weight of calcium is 32.47% and the weight of phosphorus is 18.64%. This shows good adhesion between porous tantalum and hydroxyapatite. The result shows that the optimum temperature of HA-coated porous tantalum is at 850°C. The morphology structure of the HA-coated porous tantalum shows that the adhesion between porous tantalum and HA is in good condition.

4. Conclusion

Sintering temperature is the important part to investigate the quality adhesion between hydroxyapatite and porous tantalum. After tested the various temperature during the sintering process, the optimum temperature accessible is at 850°C. The morphology of the coated porous tantalum at this time shows the strongly adhesion between it. It also shows the higher weight of hydroxyapatite during the EDX characterization.

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