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Producing Stainless Steel (SS16L) with Hydroxyapatite (HA) via Slurry Method

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Abstract: Stainless steel is now widely used metal in biomedical applications such as implants, which are used to replace structural features of the human body. The bioactive and biological advantage of hydroxyapatite (HA) for the bony tissue impact is to be established as the material of choice for implant applications. The slurry process was used to fabricate stainless steel-hydroxyapatite foam with various Hydroxyapatite powder compositions of 1 wt%, 3 wt %, 5 wt %, 7 wt% , and 10 wt %. Binders such as Polyethylene Glycol (PEG), Carboxymethyl Cellulose (CMC), and distilled water were used. Raw materials was stirred using a mechanical stirrer, and polyurethane (Pu) cylinders with dimensions of 22-24mm length and 15-16mm diameter was submerged in SS316L-HA slurry until completely coated. The coated material was dried inside a drying oven at 40 ° C for 24 hours before being sintered at 1200 ° C in tube furnace containing argon gas. A mechanical stirrer was used to mix the raw materials, and polyurethane (Pu) cylinders measuring 22-24mm in length and 15-16mm in diameter were immersed in the SS316L-HA slurry until fully coated. The coated material were dried for 24 hours at 40 ° C inside a drying oven before even being sintered at 1200 ° C in an argon-gas-filled tube furnace. The result of density are in range of 0.857g/cm³-2.335g/cm³ and porosity in range of 56.20%-84.39%. SS316L-HA were successfully developed by using slurry method.

Keywords: Biomedical, Composition, Metal, Implant, Ceramic.

1. Introduction

Metal alloys include metals like cobalt-chrome alloys, titanium alloys, and stainless steel 316L, which are commonly a dental device and orthopaedics and also implanting materials [1, 2]. The SS316L alloy, which is a medically acceptable metal, allows of external fixation [3, 4]. In addition, due to properties such as ease of manufacture, mechanical characteristics that are extremely cost-effective, low corrosion resistance, biocompatibility, high fracture toughness, and high strength, SS316L is commonly used as implant devices [5]. Foams as well as other porous materials developing a cellular structure wide range for intriguing physical and mechanical properties, as excellent adhesion combined low increase in the thickness or gas permeability and heat conductivity are both high. The replication of polyurethane (PU) foam is among most successful methods for producing high porosity, which is suitable for structure's interconnectivity resemblance cancellous bones [6]. Hydroxyapatite (HA), are

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large inorganic bone component, are widely used in bone regeneration and biomedical implants regarding its biodegradable, bioactive, and osteoconductive properties. [7, 8, 9]. In addition, HA are better mechanical characteristics, such brittleness and poor fracture toughness, which prevents them from being used in load-bearing applications [10]. There are two types of implants which is permanent implant and temporary implant. Permanent implant very important to ensure the bonding between the implant material and living tissue are strong enough and safe. However, the current metal implant is still weak. As a result, tough metal-ceramic composites are made of metallic materials to improve mechanical properties [11]. The HA and SS316L composites provide strong biomaterial substitute implant applications [12]. The aim this research is to develop SS316L-HA composites in order investigate the physical and mechanical properties as well as the microstructure behavior of composites in tube furnace. As a result, the study's purpose is to investigate the effects of different hydroxyapatite powder compositions sintered in an argon gas furnace.

2. Materials and Methods

The Stainless Steel (SS316L) and Hydroxyapatite (HA) [SS316L-HA] were the materials used in this analysis. Polyethylene Glycol (PEG), Carboxymethyl Cellulose (CMC), and distilled water are used as binders. For 3 hours, a mechanical stirrer was used to combine the ingredients. Various parameters were used in this analysis, including 5 different HA parameters, which are all stated in table 1 below.

Table 1: Parameter used in this study

Stainless Steel (SS316L wt.%)	Hydroxyapatite (HA wt.%)	Carboxymethyl Cellulose (CMC wt.%)	Polyethylene Glycol (PEG wt.%)	Distilled Water (wt.%)	Sintering Temperature (˚C)
60	1	2.5	2.5	31	
60	3	2.5	2.5	32	
60	5	2.5	2.5	30	1200
60	7	2.5	2.5	28	
60	10	2.5	2.5	25	

Polyurethane (Pu) cylinders with dimensions of (24-25mm) length and (15-16 mm) diameter were dipped in slurry for 20 minutes, squeezed to extract excess slurry, and dried for 24 hours in drying oven at 40°C. The method of dipping and squeezing was repeated many times. Make sure the PU foam design was entirely coated and fully engulfed in slurry. Sintering method was the final step in this analysis, and all samples sintered at 1200°C in a tube furnace with argon gas. In this study, a two-stage profile of sintering used, with the 1st stage involving raising the temperature to 600°C and soaking for 1 hour. The temperature rises to 1200°C in the second level, but the soaking time remains the same at 1 hour. The sintering profile for SS316L-HA foam is shown in figure 1.

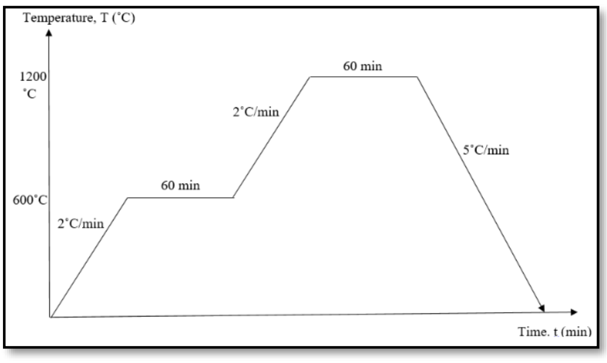


Figure 1: The SS316L-HA foam sintering profile

3. Results and Discussion

Numerous methods [13] can be used to calculate the density and porosity. Nonetheless, the Metler Toledo measuring system was used to calculate the porosity and density percentages of SS316L-HA foam based Archimedes Principle. Table 2 shows 4 samples from each of the SS316L-HA compositions before and after sintered in a tube furnace, this material was used in an experiment. According to Table 2, HA samples with a composition of 1 wt. % and 3 wt. % have good structure but its fragile and black in colour. Therefore, sample were too fragile and easily collapse when touched. For the composition of 5 wt.%, 7 wt.% and 10 wt.% all the samples were fragile and entirely collapse after sintering process. Table 3 has shown the average porosity and density of SS316L-HA. Meanwhile, the porosity and density SS316L-HA foam was compared in figure 2.

Table 2: Shows illustration of four samples from each of the SS316L-HA compositions used in this experiment.

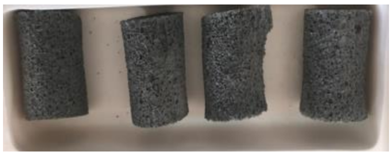
HA	Before sintered	After sintered	Observation
1 wt.%			The sample's appearance were black,fragile.
3 wt.%			The sample's appearance were black,fragile.
5 wt.%			The sample's entirely collapse after sintering
7 wt.%			The sample's entirely collapse after sintering but the appearance grey.
10 wt.%			The sample's entirely collapse after sintering

Table 3: Shows the average porosity and density of SS316L-HA foam

HA	Average of Density, (g/cm³)	Average of Porosity, (%)
1 wt.%	0.956	74.49
3 wt.%	0.857	73.5
5 wt.%	1.282	84.39
7 wt.%	2.335	56.20
10 wt.%	1.552	66.9

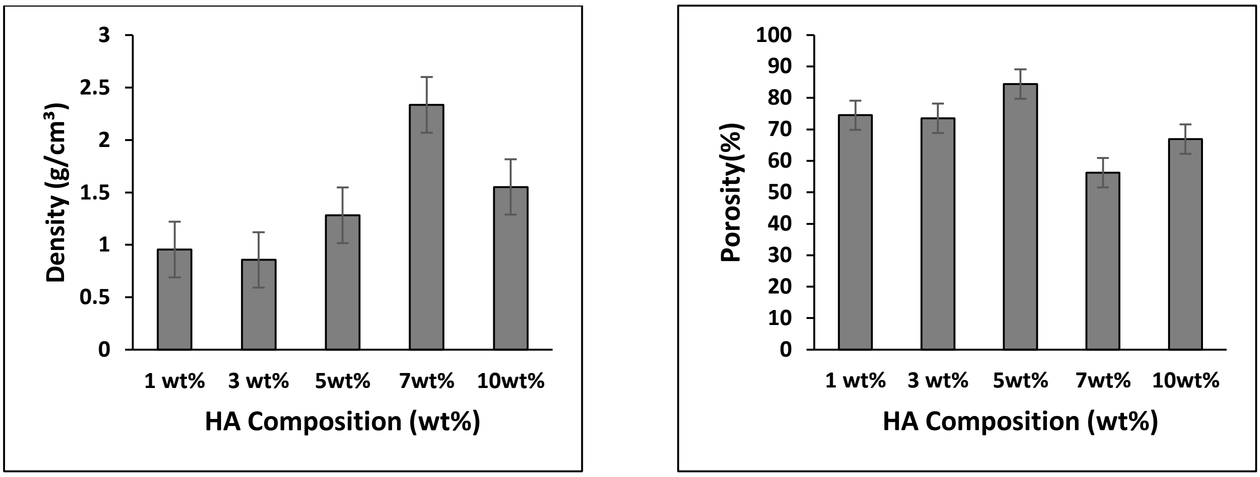
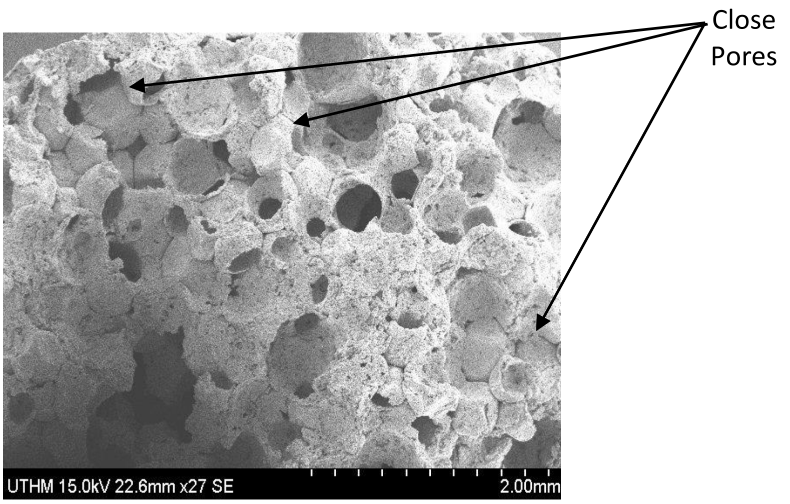


Figure 2: Shows the Average Density and porosity of SS316L-HA foam

According to table 3 and figure 2, the highest density value is 2.33 g/cm3, contains 60 wt% of SS316L and 7 wt% of HA, while the lowest average density value is 0.956 g/cm3, contains 60 wt% of SS316L and 1wt % of HA. The average highest of porosity for SS316L –HA foam are 84.39% which contains 60wt% of SS316L and 5wt% of HA and lowest value of porosity is 56.20% contains 60wt% of SS316L and 7wt% of HA are shown in table 2 and figure 3. Theoretically, the value of density increase meanwhile the porosity decreased while solid loading increase [14]. From figure 2 the density increased while porosity decreased as the loading increase 5wt% to 7wt% of HA.

The structure of the graphs, as seen in Figure 2 and 3, corresponds to the density and porosity theory, in which the density varyingly related to porosity of sintering process [15]. The density and porosity of SS316L-HA can be affected by the thickness of the slurry coated on PU foam struts [16]. This is because when the composition of HA increase slurry become thicker. Implant density must between 0.05-1.0 g/cm3 and for porosity between 75% and 90% for biomedical applications [17].

According to the results of the SEM and EDX analyses, the shaped SS316L-HA foam is closed pore. Closed pore foam has a higher density advantage since the weight is heavier. The EDX and SEM analysis for the composition SS316l-HA foam are shown in table 4 and figure 3. The density increase due to more closed pores makes it makes to increase the value of density. Because of this phenomenon, the foam became stronger and had a lower porosity value. The porous implant's open and interconnecting pores will facilitate body fluid movement and so encourage tissue ingrowth inside the open network. The expansion of the particle size associated with the increasing density value, resulting the removal of pores within the particles. This situation decreased the porosity value [18]. Due to oxidation during the sintering process, all of the samples became brittle, weak, and collapsed.



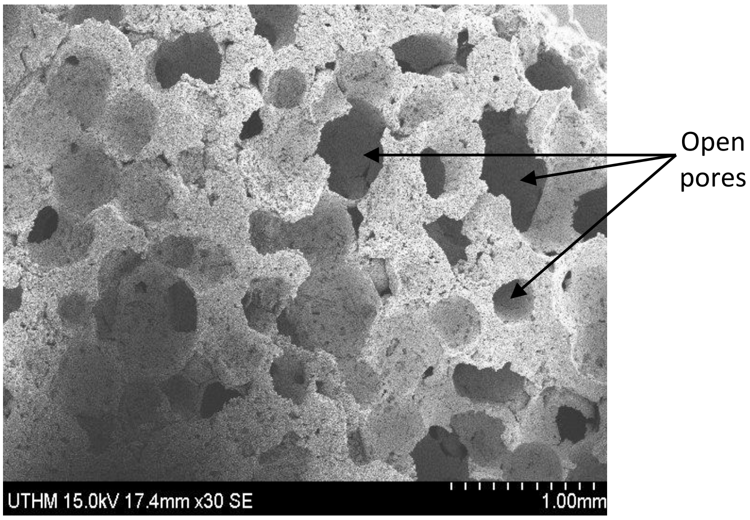


Figure 3: The analysis of EDX and SEM

From EDX analysis shows it found that the contaminate element which is O, Al, and F. Other than that is where element of SS316L-HA component is shown in table 4. From EDX result, which analyses elevated higher oxygen content in samples as seen in figure 4, may also confirm the oxidation. The interstitial element contents, such as nitrogen, carbon, and oxygen which influenced by sintering temperature, cooling rate and atmosphere determine corrosion resistance [19, 20]. S. W. Kim et al., stated that it is critical to minimize contamination levels, therefore cannot eliminated completely [21]. These contaminants form on the surface of polymeric burnout the sintering process [22].

Table 4: Shows the composition of SS316L-HA foam determined by EDX

Element	Wt.%	At.%
O	47.84	51.61
C	13.2	13.10
Cr	6.8	5.64
Fe	18.56	17.45
Ca	11.8	11.4
P	1.8	0.8
Total	100	100

4. Conclusion

Using the slurry process, the SS316L-HA foams were successfully developed and fulfil the requirement of biomedical implant. The density and porosity with similar to biomedical implant which is hydroxyapatite of 1wt% and 3wt%. After sintering, the samples were found to be oxidised by SEM and EDX analysis. The metal foams had a closed pore rather than an open pore, and the powder particles did not expand well enough to combine with each other, according to SEM diagrams. The thickness and regularity of the SS316L-HA coating on the PU foam struts, the openness of the cell, and the capacity to preserve its shape after the polymer foam is removed during the sintering process are all affected by the slurry's properties. As a result, the metal foam should have an open pore for this analysis to work.

Furthermore, the samples must be sintered thru vacuum furnace to avoid oxidation, suggestion for future research improving the properties of SS316L-HA foam. Therefore, PU foam would have a large enough pore size to aid in the production of SS316L-HA open pore foam.

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