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Processing of Stainless Steel (SS316L)-Hydroxyapatite (HA) Powder Composite through Powder Injection Molding

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Abstract. The combination of a metallic bioinert material, stainless steel 316L (SS316L) and a bioactive material, hydroxyapatite (HA) can produce a composite material which has superior properties for orthopaedic applications. The main objective of this study is to investigate the effects of sintering temperature and time on the physical and mechanical properties of the sintered part. 50wt.% of SS316L and 50wt.% of HA were mixed with a binder system which consisted of palm stearin (PS) and polyethylene (PE) for powder loading. Rheological properties show the pseudoplastic behavior of the feedstock, where viscosity decreases with increasing shear rate. The feedstock was injection moulded into a tensile bar shape while thermal debinding was carried out at 320°C and 500°C. The brown part was sintered at 1000, 1100, 1200 and 1300°C, with three different sintering times of 1, 3 and 5 hours in the furnace. The highest sintered density measured was 95.61% of the theoretical density. The highest hardness and Young's modulus measured were 150.45HV and 52.61GPa respectively, which is higher than the human bone. The lowest percentage of carbon content was 0.022 wt.% when the sample sintered at 1300°C for 1 hour. Mechanical and physical properties show that the SS316L/HA composite was successfully produced through the PIM process and can be potentially used for medical applications.

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1. Introduction

Powder Injection molding (PIM) is a method that can produce a small, complex geometry and net shaped products at low cost of ceramic or metal powder[1]. This PIM process is a combination from plastic injection moulding and conventional powder metallurgy (PM) processes [2]. The PIM process has four main steps, which are mixing, injection moulding, debinding and sintering[3]. The powder and the binder with a certain ratio will be mix together in order to produce a homogenous feedstock through the mixing steps. Then the green part will be produce through injection molding process where the feedstock will flow into a mould cavity under heat and pressure [4]. The debinding step is where the binder system in the green part will be removed and the last step in PIM is sintering which the brown part will be sinter at an elevated temperature to shrink in an isotropic way to achieve full density[5]).

An alternative to natural bone grafts is a material that is biocompatible, osteoconductive and can withstand mechanical loading. Bioceramic, which is referring to hydroxyapatite (HA), is a very good

candidate as it has a similar chemical composition ²⁸ to the mineral component of natural bone[6]. This bioceramic has been proved to have an excellent biocompatibility and tissue bioactivity properties [7]. However, the disadvantages of HA are brittleness and low fracture toughness, which is limited to load bearing applications. Thus, HA with a good in mechanical properties are required in order to widen their application in the medical field in the future.

The improvement idea is to add a mixture of metallic biomaterials in HA which producing metal-ceramic composites. By combining these metallic biomaterials with HA, it can produce a composite with good physical and mechanical properties that ¹³ succeed in long-term load-bearing applications. An austenitic stainless steel (SS316L) has been widely used as an implant material in orthopaedic applications due to its good corrosion resistance at high temperatures[8]. This SS316L was available at low cost, ease to fabricate and have the mechanical properties that can be related to natural bone[9]. The other advantages of SS316L is it ⁵ as proved biocompatible material and the thermal coefficient properties is close to HA ceramic[10]. The objective of this study is to determine the effect of sintering temperature and holding time on the mechanical properties of the sintered part of SS316L/HA composite.

2. Experimental procedures

In this study, 50 wt.% of SS316L powder, purchased from ¹¹ Epson Atmix Corporation, Japan with an average ³¹ particle size of 6.75 μm was mixed with 50 wt.% of HA powder that obtained from Sigma Aldrich, with an average particle size of 5.34 μm using Brabender mixer. Based on critical powder ²⁷ time percentage (CPVP), the powder loading used in this study was 61.0 vol.% with 39.0 vol.% of binder system. The binder system used consists of 60 wt.% of palm stearin (PS) and 40 wt.% of polyethylene (PE).

Rheological properties were determined by using CFT-500D capillary ²⁶ rheometer with a diameter of 1 mm at 150, 160, 170 and 180 °C. The SS316L/HA feedstock were then injected using a DSM Xplore injection moulding machine producing in dumbbell shape. Next, the green body were debinded in order to remove the binder system. In this process, two stages of temperatures were used which are 320 °C to remove PS and 500 °C to eliminate PE under argon atmosphere. The brown part produced were sintered at four different temperatures; 1000, 1100, 1200 and 1300 °C and the sintering time were 1, 3 and 5 h under air environment with heating rate of 3 °C/min. Morphological analysis were carried out on the sintered parts as well as density, hardness, Young's modulus and percentage of carbon content to assess the performance of the physical and mechanical properties.

3. Results and discussion

Rheological test were carried out to identify the flow ⁹ characteristics of the feedstock. Viscosity is the most important properties in rheology because it ⁴⁰ very sensitive to temperature and shear rate[11]. Based on graph in Fig. 1, it was shown that the viscosity of the feedstock decreased with increasing shear rate indicating pseudo-plastic flow behavior. Huang et al. 2003[12] stated that the ¹⁶ feedstock with pseudo-plastic flow behavior can be excellent in PIM applications and it's also found that the decreasing of the viscosity with increasing shear rate will produce the green part with free defects. The viscosity of 61.0 vol.% powder loading feedstock shown between 35.01 to 229.20 Pa.s while the shear rate is between 1070 to 11210 s⁻¹. German and Bose, 1997[13] stated that the viscosity of the feedstock should be not more than 103 Pa.s while shear rate is between 102 -105 s⁻¹.

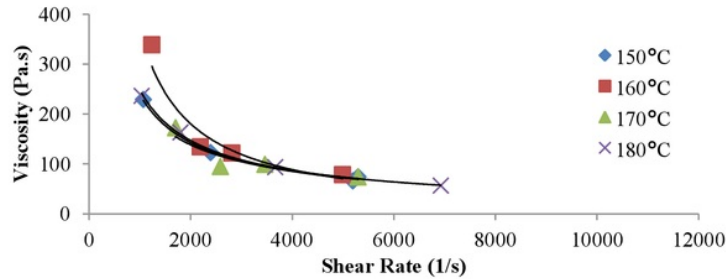


Figure. 1 Rheological properties of SS316L/HA feedstock for 61.0 vol.%

Sintering can give the most important factor which can give an impact on the properties of the sintered part. Sintering was conducted at the temperature of 1000, 1100, 1200 and 1300 °C with 1 h sintering time. Fig. 2 (a), (b), (c) and (d) shows the morphology of the sintered part based on variable temperatures. Based on Fig. 4 (a), the structure is porous and HA particles were began to defuse to each other through necking process. The pores were connected from one to another and at the same time, the necking area between HA and SS316L was formed. It can be seen that the open pores remain and occurs at all sintering temperatures. This happened due to the necking process that occurred on the SS316L/HA composite during sintering process. This porous structure is important because it can allow the body fluid circulation and absorption of protein in the bones where it can promote the bone growth [14].

When the temperature increased, the grain size increased become denser. At temperature 1300 °C, the grain size of both SS316 and HA was interconnected to each other and separated by grain boundaries and caused by the densification process [15].

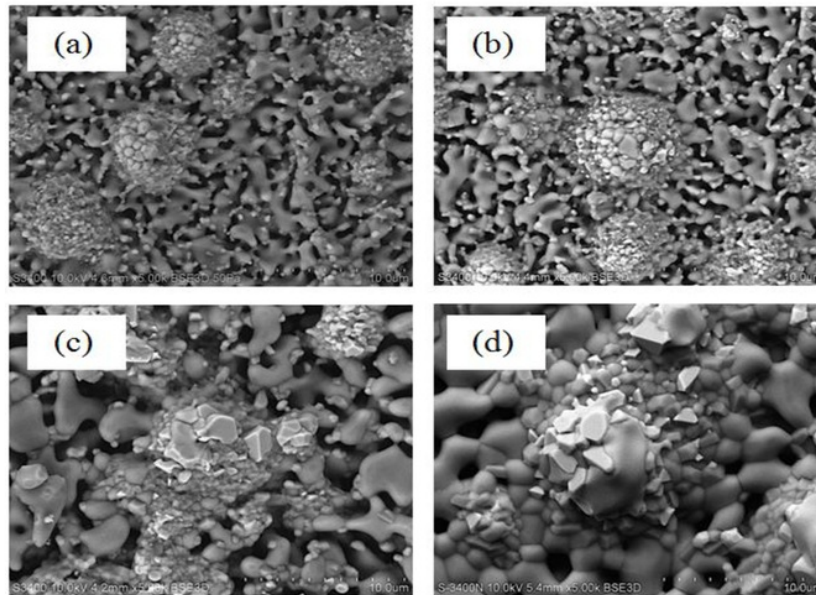


Figure.2 SEM morphology of sintered part for 61.0 vol.% at sintering temperature (a) 1000 °C, (b) 1100 °C, (c) 1200 °C and (d) 1300 °C.

The highest sintered density is 95.61% (4.33 g/cm³) of the theoretical density of SS316L/HA composite, which was sintered at 1300 °C for 1 h while the lowest density is 81.24% (3.68 g/cm³) which was sintered at 1000 °C for 5 h. The sintered density decreased when the sintering time increased due to the decomposition of HA into the other phase which is TCP and TTCP phase [16]. On the other research, the decreasing density was caused by the coarsening of the sintered body [17].

Hardness of the sintered part for the SS316L/HA composite were compared in terms of sintering temperature and time. Hardness value increased with the increasing sintering temperature. The highest value obtained when sintered at 1300 °C for 5 h which is 150.45 HV while the lowest hardness was 123.51 HV when sintered at 1000 °C for 1 h. This is because the sintered parts became denser when the sintering temperature and time increased. The reduced of the porous structure also contribute the increasing of hardness. All the hardness values obtained have a higher value than the hardness of human bone such as cortical and cancellous bone where 40.38 HV and 35.18 HV respectively [18].

Young's modulus was plotted on the graph based on the sintering temperature and time as shown in Fig. 3. The value of Young's modulus shows increasing with the increasing of the sintering temperature. In contrast, The Young's modulus decreased when the sintering time increase from 1 h to 5 h. The highest value obtained were 33.68-52.61 GPa when sintered at 1 h for all sintering temperatures. All Young's modulus values obtained is above the minimum value of the human bones which is 10-30 GPa [19]. Sintering time is taken into account in this study in order to obtain the optimum time for sintering S316L/HA composite at the highest Young's modulus value. As shown in Fig. 3, the Young's modulus values decreased with increasing sintering time. This happened due to the decomposition of HA into β -TCP and TTCP when the sintering time is increased which can lead to low mechanical properties of the sintered part [20].

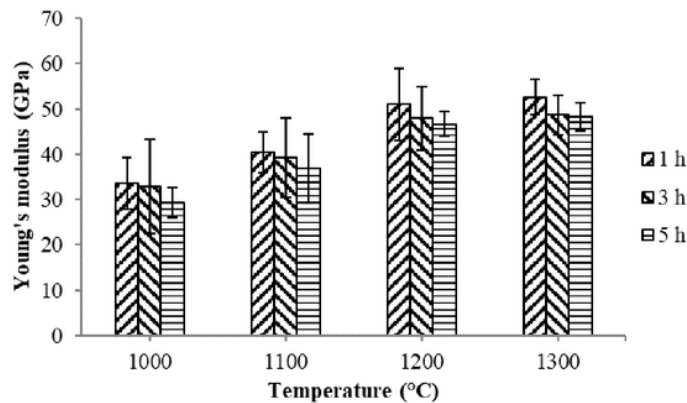


Figure 3. Young's modulus of 61.0 vol.% for sintered part at different temperatures and time

The percentage of carbon content of the sintered part for 61.0 vol.% SS316L/HA composite sintered at 1000, 1100, 1200 and 1300 °C for 1 h was measured. The results shows were 0.046 wt.% (1000 °C), 0.025 (1100 °C), 0.023 wt.% (1200 °C) and 0.022 wt.% (1300 °C). The percentage of carbon content decreased when the sintering temperature increased. At 1300 °C, it shows the lowest reading (0.022 wt.%). Based on ASTM A240, the carbon content of SS316L is 0.030 wt.%.

4. Conclusion

The rheological properties of SS316L/HA composite show pseudo-plastic behaviour, which is suitable for the powder injection moulding process. It was found that when sintered at 1000 °C, pores form on the sintered part. The pores decreased with increasing sintering temperature. Grain growth process can be seen clearly and causes the sintered part to become denser with increase of sintering time from 1 h to 5 h. The density increased with increasing sintering temperature and the highest density value obtained was 4.33 g/cm³ which is 95.61% of the theoretical value when sintered at 1300 °C for 1 h. On the other hand, the density decreased with increasing sintering time because the decomposition of HA into TCP and TTCP. The hardness and Young's modulus of the sintered part obtained is more than the hardness of the human bones. The increase in hardness and Young's modulus when the sintering temperature is increased occurred due to the densification process, but when the sintering time is increased, Young's modulus decreased. Sintering at 1300 °C gives the lowest weight percentage of carbon content which is 0.022 wt.%.

5. Acknowledgements

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