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HA/Ti6Al4V Powder with Palm Stearin Binder System - Feedstock Characterization

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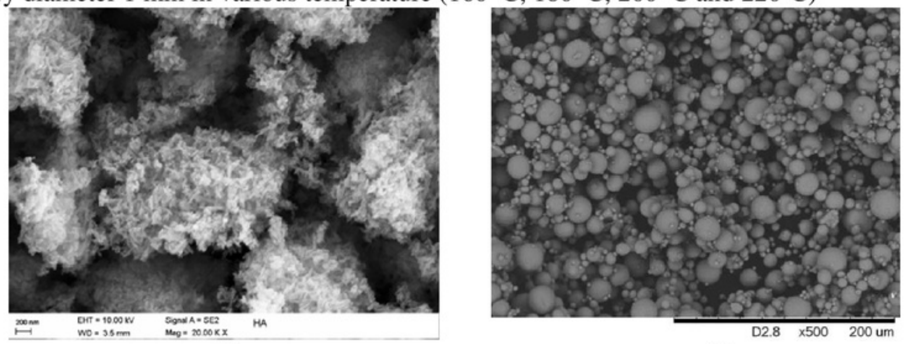
Abstract. Metal Injection Molding (MIM) is widely known as net shape process to mass production for complex shape. In this study polyethylene and palm stearin was used as binder system for composite HA and Ti6Al4V. Ti6Al4V has spherical shape with particle size 25 µm and Hydroxyapatite has particle size 5 µm. Brabender® was used to mix HA and Ti6Al4V powder and binder for making feedstock, rheological properties of feedstock was examined by capillary rheometer. Effects of temperature, viscosity, shear rate and activation energy were discussed in this paper. Binder was removed by thermal debinding. Rheological test showed feedstock of composite HA and Ti6Al4V shows pseudoplastic behavior. Sintering was conducted on 1200°C by argon with holding time 2 hour.

Introduction

Quality of feedstock in MIM process play important role to determine result of final part. Several factors such as, stability, viscosity, homogeneity, flow index behavior, energy of activation and shear stress are factors to evaluate a reliability of feedstock in MIM process to achieve satisfactory results [1]. Hydroxyapatite has similar structure and composition to bone, moreover, it has excellent in biocompatibility and bioactivity and encourage natural tissue ingrowth [2]. Ones of disadvantages of Hydroxyapatite cannot be applied under heavy load application. Combination titanium and hydroxyapatite are potential material that expected to created reliable material in biomedical application [3-5]. Titanium alloy provides in strength of mechanical properties [6] and Hydroxyapatite provides biocompatibility and encourages ingrowth of natural tissue [7]. Palm stearin has fatty acid that is general contained in commercial binder. The objectives of this research are to examine binder system Palm stearin and Polyethylene when used in composite Ti6Al4V/HA for MIM process.

Experimental Procedures

Titanium powder was used in this work using Ti6Al4V type with spherical shape and particle size 25 µm which was kindly supplied by TLS Technik GmbH & Co. Hydroxyapatite with particle size 5 µm in this work was supplied by Sigma-Aldrich, as shown Fig. 1. Powder compositions between Ti6Al4V and Hydroxyapatite are 60 wt % and 40 wt %, respectively. Binder system for both of powder is palm stearin and polyethylene with composition 60 wt % and 40 wt %, respectively. Melting temperature of binders was examined by Differential scanning calorimetry (DSC). Compounding temperature should be higher than melting temperatureofbinders. Before compounding stage was performed, Ti6Al4V and Hydroxyapatite was pre- compounding using Winkworth sigma blade for one hour meanwhile mixing stage which mix powder and binder was conducted in Brabender with mixing temperature 150°C and mixing speed 30 rpm. According to German, optimal powder loading is determined in range 2 vol%-5 vol% before critical solid loading[8]. In This work, Critical powder volume percentage was obtained on 69.5%. Rheological properties of feedstock in this work was measured Shimadzu CFT-500D rheometer with die capillary diameter 1 mm in various temperature (160 °C, 180 °C, 200 °C and 220°C)

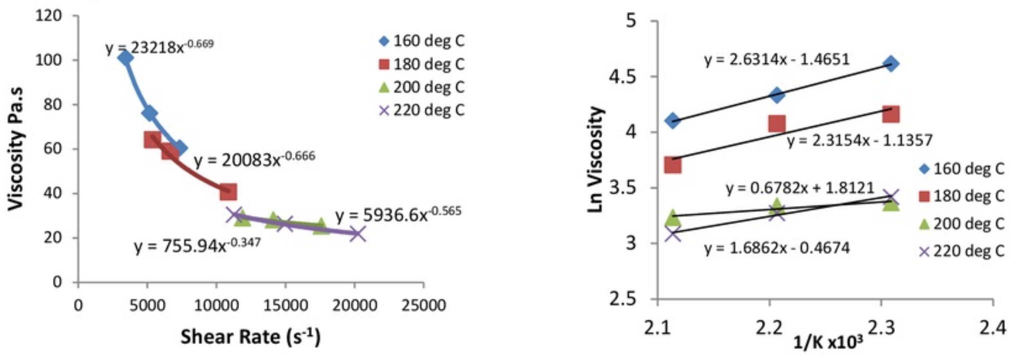


(a) (b)

Fig. 1. Scanning electron micrographs of (a) Ti6Al4V and (b) Hydroxyapatite powders

Result and Discussion

Temperature played important role to determine viscosity of feedstock in metal injection molding. Feedstock viscosity should have a value below 1000 Pa.s for getting optimal result in powder injection molding. Feedstock characterization was necessary to know suitable range in an injection process. Binder was vital component of feedstock that required keeping shape of part after injection stage.



(a) (b)

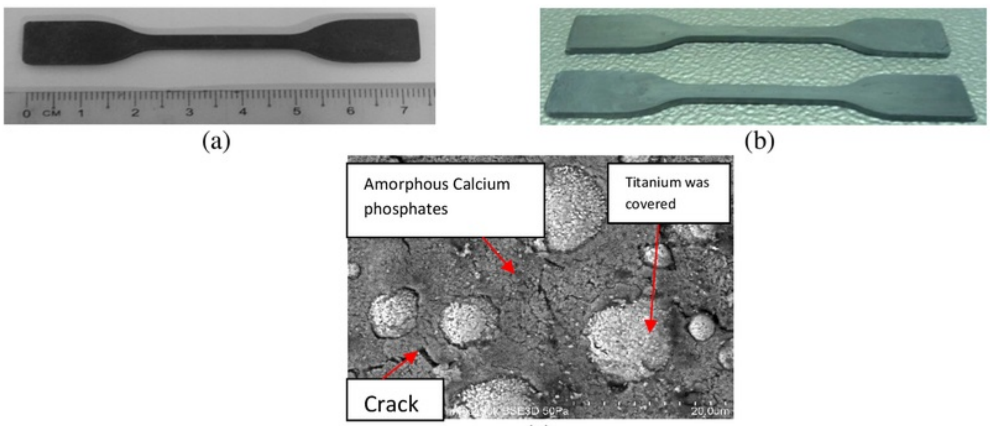
Fig. 2. (a) Viscosity and shear rate under various temperatures with powder loading 67%.
(b) Graph of Temperature dependence Viscosity

Degradation temperature of binder and pseudoplastic flow of feedstock are input factors which considered in determining appropriate temperature for injection molding. Pseudoplastic flow is condition which viscosity of feedstock decrease with increasing value of shear rate. Pseudoplastic condition was achieved if viscosity value less than 1000 Pa.s and has shear rate range between 100and 10000(1/s). Feedstock in this work was considered on pseudoplastic condition as shown on the Figure 4(a). It showed on various load (140, 160 and 180 N) increasing temperature due to has effect in decreasing viscosity and increasing shear rate. In general, viscosity (η) and shear rate $\dot{\gamma}$ can be described using equation (1), n is flow behavior index which determined based on slope *(n-1)* of equation.

$$\eta = K\dot{\gamma}^{n-1} \quad (1)$$

Based on Fig. 2(a), all of feedstock under various parameters was basically pseudo-plastic. Feedstock under temperature 160°C has smallest flow behavior index (n) value (0.331) than all other feedstock (180 °C : 0.334, 200 °C : 0.653 and 220 °C:0.435). The biggest *"n"* was achieved feedstock under temperature 200°C. Feedstock under temperature 160°C was considered having greater pseudo-plastic based on "*n*" value compared with other feedstock. Feedstock with smaller "*n*" has well on rheological stability and higher on pseudo-plasticity properties. Based on Fig.2(b), viscosity of feedstock was sensitive with increasing temperature, at the temperature 160°C, 180 °C and 200 °C there was increasing significantly of viscosity. However, at temperature 220 °C there was no too much change in viscosity of feedstock. It was believed that palm stearin has fully melted. The smallestactivation energy (*E*) of feedstockwas achieved at temperatures 200°C as shown on gradient value on the Fig.2(b). Relationship viscosity, temperature and activation energy generally was described on Arrhenius equation [9].

Properties of feedstock with lower of "*n*" value due to has better on stability of rheological and pseudo-plasticity [9]. Moreover, ideal feedstock for MIM is required low activation energy (*E*) and low "n" value. However, it was difficult to reach condition with low on Flow behavior index (*n*) and activation energy (*E*) in same time. Weir equation about moldability generally is used as reference for selection feedstock [1, 10]. The effect of injection molding parameters process such as; Injection pressure, injection temperature, mold temperature and holding time on the quality of green part[11]. Feedstock composite Ti/HA has been successful injected on pressure 5 bar, mold temperature 150 and melt temperature 100°C as shown on the Fig 3(a).



(a) (b) (c)

Fig.3. (a) before debinding, (b) after debinding and (c) SEM of sintered specimen

Palm stearin and polyethylene was removed by thermal debinding that consist two stage holding temperature, 320°C and 500°C.First stage for removing palm stearin due to has lower decomposition temperature than polyethylene. 3°C/min, 5°C/min were used as heating rate for removing palm stearin and polyethylene, respectively. Holding time for each stage is 1.5 hour. Low heating rate was required on early stage to remove palm stearin, high heating rate was avoided because resulting crack on the specimen due to gaseous burn-out product through small pores [12].Fig.3 (b) shows specimen after debinding, Generally after debinding process, green body tends to fragile due to most of binder has been removed.

On high temperature Hydroxyapatite tend to change become secondary phase such as TCP or TTCP. Generally to determined phase formed using stoichiometry (Ca:P ratio). EDX result of specimen after Debinding has ratio Ca: P = 1.67, it means there were not a phase change on the specimen.Specimen was sintered in Argon gas with temperature 1200°C, heating rate 7°C/min and holding time 2 hour. Fig. 3(c). Show several secondary phases occurs on sintered specimen, secondary phase occur due to high temperature of sintering. Hydroxyapatite filled gabs between of titanium particles tend to have amorphous phase. Cracks also occur on this sintered specimen. Appearance ofcracksbelieveddue to differences inthermalcoefficient oftwodifferent materialsgiving rise toresidualstress.

Summary

Palm stearin andPolyethylenebindersystemhave beensuccessfullyused as binder intitaniumpowderandHydroxyapatite. Binder system has beensuccessful inestablishinga bondbetween theparticlesandalsokeeps the shapeof thegreenbody.Viscosity of feedstock has showed a pseudoplastic in range 10 -1000 Pa.s in and shear rate in range 10^2 s^{-1} – 10^5 s^{-1} various temperatures.

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