FEEDSTOCK CHARACTERISATION OF TITANIUM ALLOY MIX WITH PALM STEARIN BINDER SYSTEM FOR METAL INJECTION MOULDING

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ABSTRACT

This paper presents the characterization of MIM feedstock consisting 90wt% of titanium alloy (Ti-6Al-4V) powder mix with binder 60wt% palm stearin and 40wt% polyethylene. The characterization of Ti-6Al-4V alloy powder, binders and feedstock includes scanning electron micrograph (SEM), thermogravimetric analysis (TGA), differential scanning calorimeter (DCS) and rheological test were established. Rheological results exhibited pseudoplastic or shear thinning flow behaviour, where its viscosity decreased with increasing shear rate. The feedstock viscosity also decreased with increasing temperature and was found to be suitable for moulding.

KEYWORDS: Metal injection Moulding, palm stearin, feedstock, rheological

INTRODUCTION

MIM products are various and range from consumer products, office equipment, medical instruments, and automotive components to industrial processing equipment. The MIM process consists of four steps such as mixing, injection moulding, debinding and sintering. Small sized metallic powder is initially mixed with a wax-polymer binder to form a homogeneous feedstock and the feedstock is shaped a mould. After removal the resulting green part from the mould, the wax polymer binder is removed by heating and sintered in a controlled atmosphere furnace (Suri *et.al.* 2003).

Because of MIM process consists of many steps before the final part could be produced, so there is ample opportunity for defects to be created. Thus, it is necessary to characterize the material being processed, from initial powders and binders to the final component (Iacocca 1999). Characterization of metal powder and binder components are the most important steps in understanding the whole process in MIM (German 1990, German & Bose 1997, Iacocca 1999). The initial properties of MIM feedstock will dictate the final properties of the sintered part; thus key parameters must be identified that can predict the success of the end product (Iacocca 1999 ,Ismail *et. al*. 2005).

The other issue in MIM process is the selection of the binder. The binder should permit feedstock with high powder loading to be mixed and injection moulded. In addition, debinding process should ideally occur in a short time without producing defects (Anwar & Davies 2007). Palm stearin (PS) is a potential binder system that can be used since it is an available in resource in Malaysia. It is believed that palm stearin has possibility as a binder component since it is consists of fatty acid which is used as a surface active agent for many binder used. A primary advantage of palm stearin is that their chemistry and rheological properties can be modified to meet the specific requirements of MIM (Ismail et. al. 2007).

This paper covers the initial step of preparation of Titanium alloy (Ti-6Al-4V) feedstock including characterization of metal powder, binders and feedstocks. Different percentages of volume are used in this paper to study the flow characteristic via rheological experimental studies.

METHODOLOGY

Characterization of MIM powders

Water atomized titanium alloy (Ti-6Al-4V) powders were used in the present research. The particle-size distribution for elemental Ti-6Al-4V powder was determined using a Hydro 2000MU(A). The morphology of the powders was characterized by scanning electron microscopy (SEM). The powder also has been examined their critical powder volume percentage (CPVP) using Haake Rheomix 600p machine. CPVP or critical solids loading is the composition where the particles are packed as tightly as possible without external pressure and all space between the particles is filled with binder. With more powder (less binder) it can prevent voids which lead to difficult moulding and excess of binder will separates the powder in molding, leading to flashing or inhomogeneous in the moulded component (German & Bose 1997)

Preparation of Ti-6Al-4V feedstock

After CPVP was tested, the feedstock were prepared into optimum value of powder loading which the range is between 2% to 5% less than CPVP value. The binder used in this paper was fixed at the composition of 60wt% of palm stearin and 40wt% polyethylene. The mixing temperature was set at 150^{o}C. The total mixing time was 2 hours and the rotational speed of sigma blade was 50 rpm.

Thermal Analysis of the feedstock

Since the MIM feedstock is subjected to a binder removal process, it is important to understand its thermal behaviour. It is common that the feedstock is characterized by thermoravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA can be used to determine the decomposition of the binder and is very useful, especially for the design of the thermal pyrolysis cycle for debinding. Feedstock samples of mass approximately 10g were heated up to 600^{o}C at a heating rate of 5^{o}C/min under flowing, dry argon gas.

Rheological Analysis

After mixing, the feedstock was granulated in pellet form in order to be fed easily during injection process. The rheological characteristics of the feedstock was studied using Capillary Rheometer Shimadzu CFT-500D.

RESULT AND DISCUSSION

Characterization of MIM powders

Generally, the most appropriate powder size for the MIM process is in the range 4 to 20μm (German & Bose, 1997) although some literature indicates preference for a very fine particle size in the range of 2 to 8 μm (Gerling *et. al*. 2006). The Ti-6Al-4V powders were largely spherical in shape as shown in Figure 1, which is promoting excellent mouldability in the MIM process because of a low inter-particle friction (Gerling *et. al*. 2006).

The surface of titanium powder is significantly smoother than for the nickel powder. Finer particle sizes have a tendency to agglomerate into effectively larger particles. It is evident that no agglomeration occurs in almost all regions observed for the Ti-6Al-4V powder.The powder characteristics are given in Table 1. The CPVC value of titanium was 72.78 vol%. As the optimum powder loading for MIM is in the range of 2vol% to 5vol% less than critical powder loading (German & Bose 1997), so the selection of the powder volume fraction are 63, 65, 67 and 69vol%.

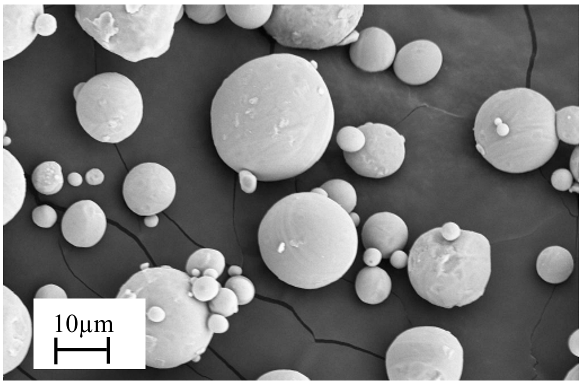

Figure 1: Scanning electron microscope of Ti-6Al-4V powder

Table 1 : Powder characteristics of Ti-6Al-4V

D_{10}(μm)	D_{50}(μm)	D_{90}(μm)	Width of distribution (S_w)*	Specific surface area (m^2/g)	Apparent density (g/cm^3)	Tap density (g/cm^3)	Pycnometer density (g/cm^3)
11.19	18.76	30.52	5.87	0.35	1.91	2.35	4.38

*$Sw = 2.56/\log (D_{90}/D_{10})$

Preparation of Ti-6Al-4V feedstock

Figure 2 shows SEM of fracture surface of the feedstocks at different powder loading. A properly mixed feedstock consists of homogeneous powder dispersion in a matrix of binder, with no agglomeration and no internal porosity and the binder filling the spaces between the particle powders and coating each powder. It is clear that all feedstocks show evidence of binder covering the particle powder. Feedstock 67vol% and 69vol% shows that some of powder separates from matrix of binder to form voids.

Thermal Analysis of the feedstock

Table 2 shows the melting point from Differential Scanning Calorimeter (DCS) test and decomposition temperature from Thermogravimetric Analysis (TGA) test which is useful to predict the temperature during injection and debinding process. Melting point for PS is 61^{o}C and PE is 127^{o}C. PS will start decompose at 288^{o}C and finish at 463^{o}C while PE start at 390^{o}C and finish at 502^{o}C.

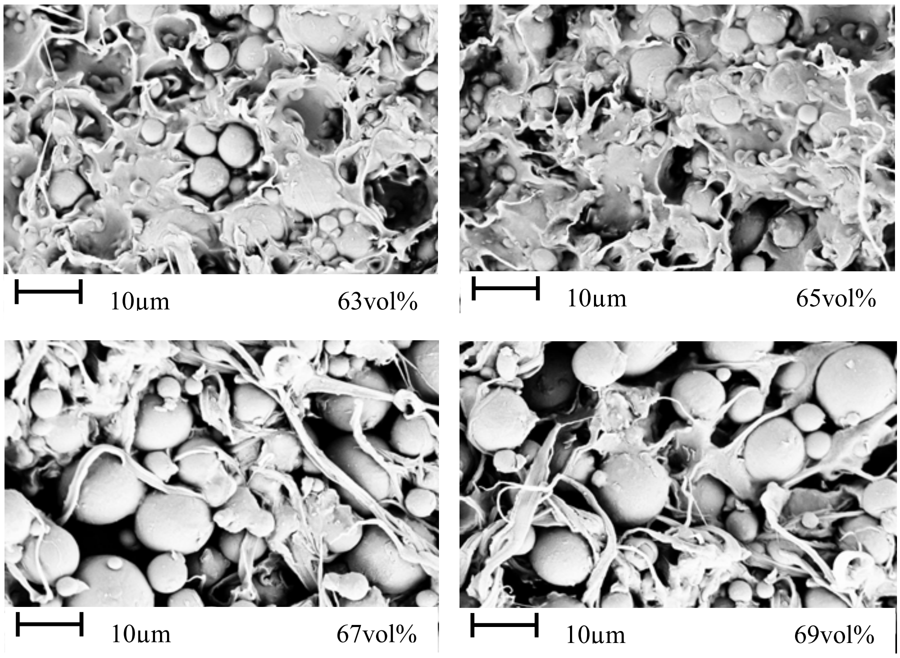

Figure 2: SEM of fracture surface of the feedstock

Table 2: DSC and TGA result for binder PS and PE

Test		Temperature (^{o}C)	
		PS	PE
DCS		61	127
TGA	(start)	288	390
	(finish)	463	502

Rheological Analysis

In MIM process, the viscosity during moulding usually ranges between 10 and 1000 Pa.s. Table 3 shows the relation of the viscosity of the feedstock at different temperature and different pressure, which indicate the flowability of MIM feedstock. From the observation, it showed that almost all the feedstocks have relatively pseudoplastic behaviour where all the viscosity were in the range between 10 Pa.s and 1000 Pa.s, which desirable for injection moulding purposes.

A MIM feedstock is also generally considered to be a shear thinning or pseudoplastic fluid, which indicate a decreasing of viscosity with increase shear rate and temperature. For pseudoplastic fluid, it can be expresses by the equation below:

$$\tau = k\gamma^{n-1}$$

where τ is shear stress, γ is shear rate, k is coefficient and n is shear strain sensitive exponent which is less than 1.

TABLE 3: Viscosity of MIM feedstock (Pa.s) at different solid loading

Solid loading (%vol)	Applied Pressure (MPa)	Temperature (°C)				
		130	135	140	145	150
63	0.07355	108.99	80.95	65.52	55.96	45.32
	0.09807	66.75	45.45	36.44	30.36	25.45
	0.1226	46.56	29.99	24.67	21.23	17.33
65	0.07355	138.79	95.74	79.46	68.63	47.66
	0.09807	84.94	53.20	44.29	37.06	29.89
	0.122	59.20	35.08	28.69	24.57	20.28
67	0.07355	214.91	152.86	52.10	39.10	29.71
	0.09807	127.31	89.19	73.50	65.81	43.25
	0.122	92.33	58.94	130.46	116.77	73.98
69	0.07355	84.16	86.88	98.22	115.22	46.61
	0.09807	107.63	125.19	108.78	80.42	59.54
	0.122	99.14	91.31	125.98	67.58	49.84

The value of n indicates the degree of shear sensitivity. The higher the value of n, the longer the viscosity changes with shear rate. So, it is recommended to select the MIM feedstock, which possess a higher value of n to ensure the viscosity decreases slowly with increasing hear rate during injection process.

The relation between viscosity and shear rate is shown in Figure 4. From the figure, it is clearly show that feedstock 63vol% and 65vol% have pseudoplastic due to the viscosity is increase slowly with decrease of shear rate at all temperature. The value of n for both feedstocks are from 0.472 to 0.52 which indicates that feedstock 65vol% has better rheological stability due to the higher value of n. For feedstock 67vol%, at temperature 130°C to 140°C, a dilatants behaviour was observed. PS

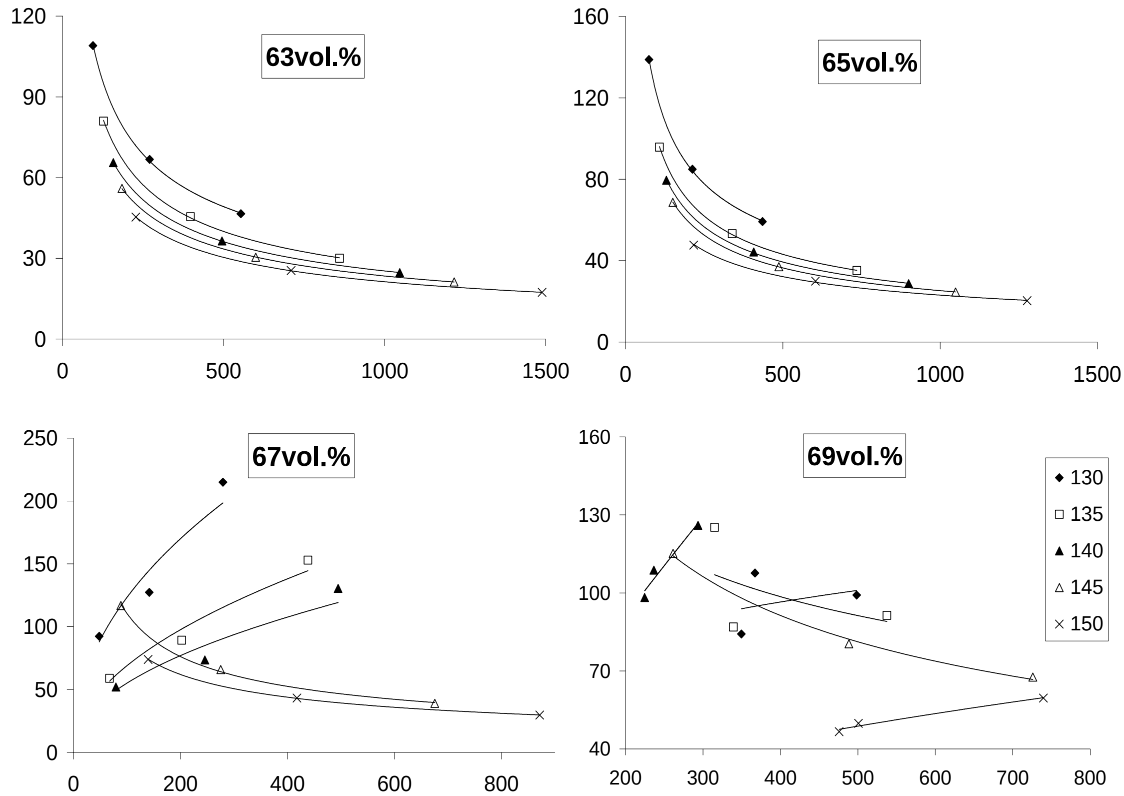

Figure 4: The relation between viscosity and shear rate at different feedstock

binder with low melting temperature was observed to flow leaving powder and binder of PE which is can lead the jetting phenomenon during actual moulding.

After increase the temperature to 145°C, the feedstock tends to change its behaviour, instead of dilatants to pseudoplastic flow. Meanwhile in powder loading of 69vol%, an inconsistent flow was observed at a wide temperature range (130°C – 150°C). Mixed-up flow behaviour (pseudoplastic and dilatant) was observed as can be seen in Figure 4. Feedstock of 69vol% is not suitable for further injection moulding process due to inconsistency of the viscosity.

CONCLUSION

Characterization data would give some insight on the scope of parameter condition to be applied during injection moulding practice. All the steps involve in characterization of MIM are needed to be monitored since they will affect the final product. CPVP test conducted at early stage has shown that the optimum powder loading of Ti-6Al-4V in the range of 2% to 5vol% less than critical powder loading (72.78vol%), but after rheological study, feedstock of 69vol% is not suitable to be injected. Feedstock of 63vol% and 65vol% are possible to be injected as the flow behaviour index indicates pseudoplastic behaviour at all temperature (130-150^{o}C). Feedstock of 67vol% suitable to be injected at temperature above 140^{o}C.

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