The International Conference on Advances in Materials and Processing Technologies, 26-28
October 2009
Kuala Lumpur, Malaysia

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

N. H. Mohamad Nor[1,2], N. Muhamad[2], K. R. Jamaludin[3], S. Ahmad[4], and M. H. I. Ibrahim[4]

1. Faculty of Mechanical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia; email: norhafiez@yahoo.com.

2. Precision Process Research Group, Dept. of Mechanical and Material Engineering, Faculty of Engineering, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia; email: hamidi@eng.ukm.my

3. Department of Mechanical Engineering, College of Science and Technology, Universiti Teknologi Malaysia, City Campus, 54100 Kuala Lumpur, Malaysia; email: khairurrijal@gmail.com

4. Department of Mechanical and Manufacturing, Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia, email: sufizar@uthm.edu.my, mdhalim@uthm.edu.my

ABSTRACT

Metal injection moulding (MIM) is a cost-effective technique for producing small, complex, precision parts in high volume. Each step in MIM process plays a vital role in order to achieve high quality final product. To have a good understanding of the MIM process and successful in manufacturing, characterisation of the material feedstock is essential. This paper presents the characterization of MIM feedstock consisting titanium alloy (Ti-6Al-4V) powder mix with binder 60wt% of palm stearin and 40wt% polyethylene. The characterisation of Ti-6Al-4V alloy powder, binders and feedstock includes scanning electron micrograph (SEM), thermo gravimetric 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

1. INTRODUCTION

Metal Injection Moulding (MIM) is a net-shaped process to produce relative small solid metal parts with high complex geometries. 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 [1].

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 characterise the material being processed, from initial powders and binders to the final component [2]. Characterisation of metal powder and binder components are the most important steps in understanding the whole process in MIM [2,3,4]. 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[2,5].

The other issue in MIM process is the selection of the binder. The binder acts as temporary vehicle for homogeneously packing a powder into desired shape and holding the particles in that shape until the beginning of the sintering. 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 [6]. 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 [7].

This paper covers the initial step of preparation of MIM feedstock including characterisation of metal powder, binders and feedstocks. Different percentages of volume were used in this paper to study the flow characteristic via rheological experimental studies. Some insight on the scope of the parameter condition o be applied during injection process could be obtained especially the effect of injected part due to changing control parameter conditions, during the injection process.

2. METHODOLOGY

2.1 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 characterised by scanning electron microscopy (SEM). The powder has been also examined for 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.

2.2 Preparation of Ti-6Al-4V feedstock

After CPVP was tested, the feedstock was prepared into optimum value of powder loading where the range is between 2vol% to 5vol% 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 50rpm.

2.3 Thermal Analysis of the feedstock

Thermal properties of binders were analysed in order to obtain the result evaporation temperature via thermo gravimetric analysis (TGA) and melting point via differential scanning calorimeter (DSC). Both properties are important especially in mixing, injection moulding and debinding to avoid any difficulties in that processes. On the other hand, these results might give an idea on how to design an appropriate parameter of injection process, and debinding cycles so that the temperature that is applied during the above processes is within the range.

2.4 Rheological Analysis

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

3. RESULT AND DISCUSSION

3.1 Characterization of MIM powders

Generally, the most appropriate powder size for the MIM process is in the range of 4 to 20µm although some literature indicates preference for a very fine particle size in the range of 2 and 8µm [8]. The Ti-6Al-4V powders are 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 [9]. 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.

A major concern in particle-size distribution analysis is the three points on the distribution designated as D_{10}, D_{50}, and D_{90}. Large values S_w correspond to a narrow particle-size distribution (S_w = 4 to 5 or D_{90}/D_{10} = 3.22 to 4.4) and small values correspond to a broad distribution ($S_w \sim 2$) [8]. The width of particle-size distribution affects mouldability; good mouldability can be obtained using wider particle-size distributions [2].

The CPVC value of titanium was 72.78vol%. As the optimum powder loading for MIM is in the range of 2vol% to 5vol% less than critical powder loading, so the selection of the powder volume fraction is 63vol%, 65vol%, 67vol%, and 69vol%.

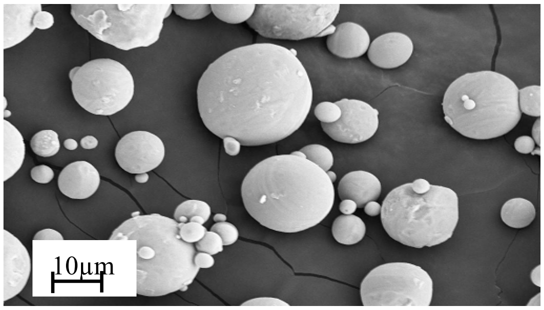

Figure 1: Scanning electron microscope of Ti-6Al-4V powder

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})

3.2 Preparation of Ti-6Al-4V feedstock

Figure 2 shows SEM of facture 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 the 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.

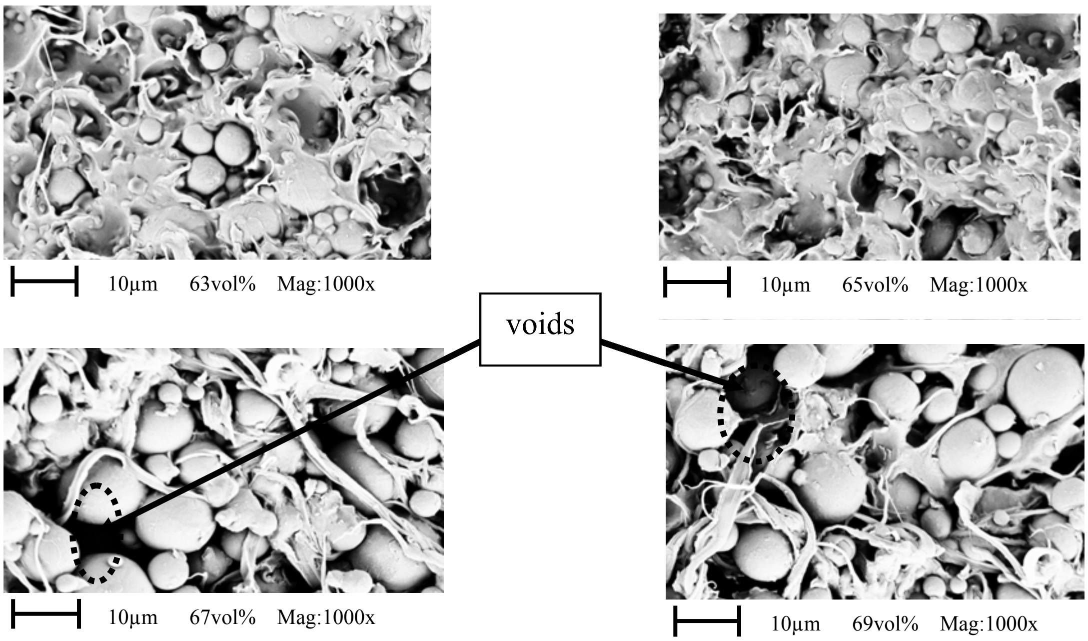

Figure 2: SEM of fracture surface of the feedstock

3.3 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. From the result obtained, debinding schedule can be designed in order to remove all the binder completely from green compact with free defects on the component.

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

3.4 Rheological Analysis

In MIM process, the viscosity during moulding usually ranges between 10 and 1000Pa.s. Table 3 shows the relation of the viscosity of the feedstock at different temperature and different pressure, which indicates 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 10Pa.s and 1000Pa.s, which is 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 expressed 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. 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 possesses a higher value of n to ensure the viscosity decreases slowly with increasing shear rate during injection process. The relation between viscosity and shear rate is shown in Figure 2.

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

Solid loading (%vol)	Applied Pressure (MPa)	Temperature (^{o}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

From the figure above, it is clearly shown that feedstock 63vol% and 65vol% have pseudoplastic due to the viscosity is rising 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 of 65vol% has better rheological stability due to the higher value of *n*. For feedstock of 67vol%, at temperature 130^{o}C to 140^{o}C, a dilatants behaviour was observed. PS binder with low melting temperature was observed to flow leaving powder and binder of PE which is can lead to the jetting phenomenon during actual moulding. After increasing the temperature to 145^{o}C instead of dilatants, the feedstock tends to change its behaviour to pseudoplastic flow. Meanwhile in powder loading of 69vol%, an inconsistent flow was observed at a wide temperature range (130^{o}C – 150^{o}C). Mixed-up flow behaviour (pseudoplastic and dilatant) was observed as can be seen in Figure 4. It can be concluded this powder loading is not suitable for further injection moulding process due to inconsistency of the viscosity.

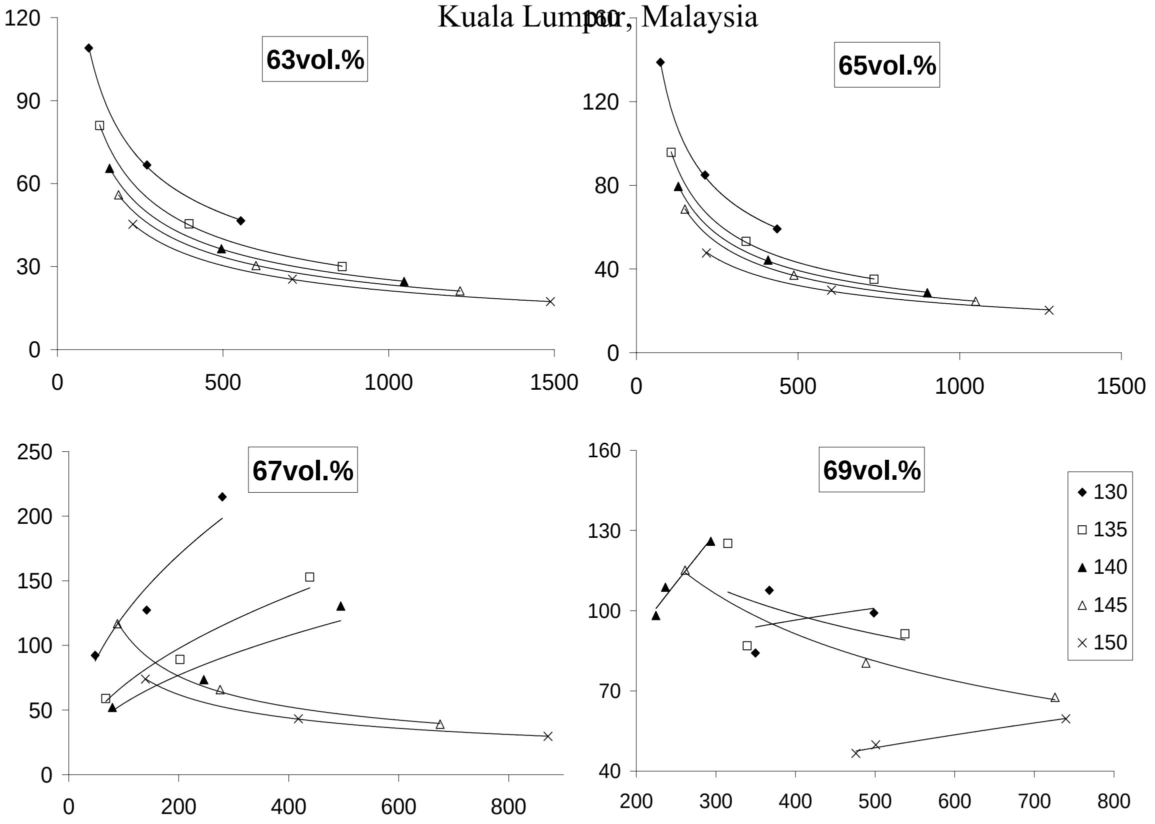

Figure 4: The relation between viscosity and shear rate at different feedstock

4. CONCLUSION

Characterisation data would give some insight on the scope of parameter condition to be applied during injection moulding practice. All the steps involved in characterisation of MIM are needed to be monitored since they will affect the feedstock and the final product. CPVP test conducted at early stage has shown that the optimum powder loading of Ti-6Al-4V is in the range of 2vol% 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°C – 150°C). Feedstock of 67vol% suitable to be injected at temperature above 140°C.

REFERENCES

[1] Z. Y. Liu, N.H Loh, S.B Tor, K. A Khor, Characterization of powder injection molding feedstock, *Int. Jurnal of Materials Characterization,* vol 49 (2003), 313-320.

[2] R. G. Iacocca, A critical assessment of characterization tests needed to support powder injection molding component fabrication, *Review in Particulate Materials*, vol. 2 (1994), 269-313.

[3] German, R. M. *Powder Metallurgy & Particulate Materials Proceessing,* Metal Powder Industries Federation, NJ. (2005).

[4] German, R.M.. *Powder Injection Molding*. Metal Powder Industri (MPIF). New Jersey. (1990).

[5] Muhammad Hussian Ismail, Nor Hafiez Mohamad Nor, Junaidah Jai, Characterization of homogeneous feedstock for metal injection molding Process, *Short Term Research Report*, Universiti Teknologi MARA. (2005).

[6] M. Y. Anwar and H. A. Davies, “A comparative review of various PIM binder system.” *Advances in Powder Metallurgy and Particulate Materials*, vol 4 (2007), 8-18.

[7] F. Ismail, M.A. Omar, I. Subuki, N. Abdullah, E.A.G.E. Ali and N. Hassan, Characterization of the feedstock for metal injection molding using biopolymer binder, *Reg. Conf. On Eng. Math., Mech., Manu., & Arch (EM^{3}ARC).* (2007). 85-92

[8] German, R.M. & Bose, A., *Injection Molding of Metals and Ceramics*. Metal Powder Industri (MPIF), New Jersey. (1997).

[9] L. Liu, N. H. Loh, B. Y. Tay, S. B. Tor, Y. Murakoshi, R. Maeda. Mixing and characterization of 316L stainless steel feedstock for micro powder injection molding. *Int. Jurnal of Materials Characterization,* vol 54. (2005), 230-238.