Journal of Mechanical Engineering *Vol SI 2 (1), 139-149, 2017*

Rheological Properties of Titanium Niobium Based Feedstocks for Metal Injection Moulding

Rozie Nani Ahmad
School of Manufacturing Engineering, Universiti Malaysia Perlis, Kampus Tetap Pauh Putra, Jalan Arau-Changlun, 02600 Arau, Perlis

Norhamidi Muhamad
Abu Bakar Sulong
Azizah Wahi
Faculty of Engineering & Built Environtment, Universiti Kebangsaan Malaysia, 43600, Bangi, Selangor, Malaysia

Farrahshaida Mohd Salleh
Faculty of Mechanical Engineering, Universiti Teknologi Mara, Shah Alam 40450, Malaysia

ABSTRACT

The rheological properties are very important in the metal injection moulding (MIM) process where it can give indication of flow ability of mold feedstock during injection moulding. In this study, the rheological properties of Titanium Niobium based feedstock with polyethylene (PE) and palm stearin (PS) as binder which have different powder loadings were analyzed using a Shimadzu Flow tester CFT-500D capillary rheometer. The binder composition consists of 40% palm stearin and 60% polyethylene based on weight fraction. The palm stearin is selected because of its low cost, low viscosity and high decomposition temperature. Rheology properties such as flow activation energy E, moldability index α_{STV}, and flow behavior index n were determined. Results indicate that the Ti-6Al-7Nb feedstocks have a pseudoplastic behavior and the value of index n is an average of 0.43. All the powder loadings can be injected and the green part density results are obtained.

Keywords: *Metal Injection Moulding, Rheology Properties, Titanium Niobium.*

ISSN 1823-5514, eISSN 2550-164X

Received for review: 2016-05-30
Accepted for publication: 2017-02-01
Published: 2017-05-15

Introduction

Metal Injection Molding (MIM) process has a competency to produce a high-performance and complex shaped metal parts at low cost [1]. Metal injection molding, also known as the alteration, is the common injection molding process for plastic. A small amount volume of plastic was replaced by a fine metal powder. Firstly, a very fine metal powder with a plastic binder was mixed to form a feedstock in the mixing process. The green parts were produced by using the specific feedstock on a conventional thermoplastic molding equipment. After that, the green parts need to remove the binder. Finally, the products need to be sintered with specific temperature. The benefit from this manufacturing technology is it produces more intricate shape, higher product density, better surface finish than the traditional powder metallurgy product and has higher mechanical properties [2].

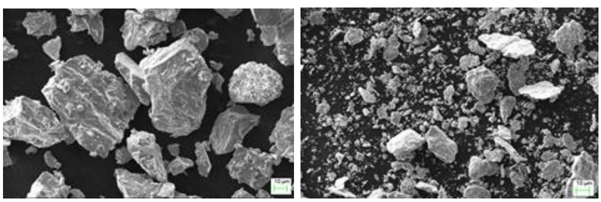
The major key parameter in MIM process requires a good rheological feedstock characteristic of the flow properties of an injection moulding process. Optimal powder loading is known to incorporate a maximum amount of powder, without compromising good rheological properties. As a conclusion, the MIM feedstock with the optimal powder loading will have good rheological properties [3,4]. In the MIM process, mold filling by the feedstock is dependent on the viscous flow of the mixture into the mould cavity. It requires special rheological properties. The most important property is the viscosity, which is extremely sensitive to temperature and shear rate. When the viscosity decreases, the shear rate increases, which is beneficial to the shape forming [5].

The most important issue in MIM process is the selection of the binder. Recently, palm stearin (PS) has been developed as a combination binder which was developed by a research student from Universiti Kebangsaan Malaysia, Iriany. Iriany successfully prepared a homogeneous feedstock by mixing the palm stearin with stainless steel powder. Lately, because of very cheap material and highly available in Malaysia, PS is a good binder system that can be used until today. It is believed that PS has a possibility as a binder component since it consists of fatty acid which provided sufficient lubricity for moulding process [6]. The ability of palm stearin is to produce a capillary way of remaining binder removed in the later stage of debinding prior to sintering [7]. This paper attempts to prove the possibility of using palm stearin as a binder system through the titanium niobium powder and injection moulding process. In order to evaluate the palm stearin as a binder system, it is intended to evaluate the rheological behaviour of the feedstock. During the moulding process, rheological analysis can be identified to quantify the stability of the feedstock.

Methodology

Materials

Figure 1 (a) shows that the titanium niobium powder of average size 50 µm was irregular in shape. This material was purchased from MHC Industrial Co. Ltd (China). This powder has been milled in a planetary ball mill (Fritsh pulverisette-6) to reduce the particle size to 24 µm. Fig. 1(b) shows the scanning electron microscope of titanium niobium powder after ball milling process. To prevent contamination, the hardmetal ball and bowl were used during process, while to avoid agglomeration and to minimize temperature rise and oxidation, the ethanol was used as a milling medium. After wet milling, the powder was dried in vacuum oven at 100 °C [8].



(a) (b)

Figure 1: (a) Scanning electron microscope of Ti-6Al-7Nb powder and (b) Scanning electron microscope of Ti-6Al-7Nb powder after ball milling process

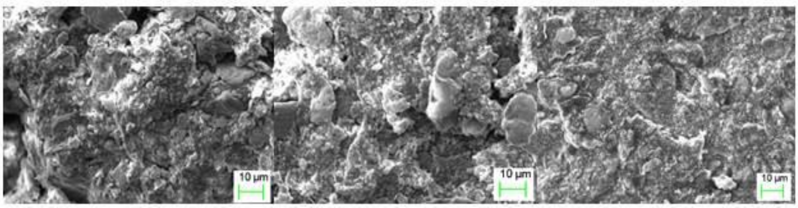
The palm stearin supplied by Intercontinental Fats Sdn. Bhd had a function as a filler phase components in this research. The back bone polymer, which is low-density polyethylene (LDPE), was purchased from Titan Chemicals Sdn. Bhd. The characteristic of binder system is shown in Table 1.

Table 1: Binder Properties

Binder	**Weigth (%)**	**Melting Temperature (°C)**	**Density (gcm^{-3})**
Palm Stearin (PS)	40	61	0.891
Polyethylene (PE)	60	127	0.950

Feedstock preparation

A balance mixture of powder and binder represents a feedstock. The success and failure in the next subsequence process were determined by proportion of binder and powder. Nevertheless, the particles are filled as tightly as possible without external pressure and all spaces between the particles are filled with binder also known as the critical powder loading [9]. In the current work, the mixing process and critical powder volume percentage (CPVP) were implemented by using the Brabender measuring mixture (W50 ETH). The titanium niobium powder was mixed with 60 wt. % and 40 wt. % LDPE and Palm Stearin, respectively at 150 °C for 90 min. in 66, 67 and 68 vol. % powder loading. Figure 2 (a-c) shows the Scanning electron microscope (SEM) micrograph of the feedstock at different powder loading. The micrographs show that the powder particles were bounded by binder components; this means that the powder-binder mixture is a homogeneous. The mixing temperature was selected depending on the melting point of binder system to obtain homogeneous feedstock[8)].



(a) (b) (c)

Figure 2: SEM micrograph of the feedstock
(a) 66vol. %, (b) 67vol. %, (c) 68 vol.%

Rheology measurement

The rheology measurement testing was conducted by CFD-500D Shimadzu capillary rheometer. Meanwhile, L/D=10 die is attached to the bottom of the extruder barrel to allow the flow through the die. The pressure drop across the die was recorded in order to calculate the shear rate at die wall. The constant capillary temperature was set at various temperatures (150°C, 160°C and 180°C) and also the load applied to the tester was from 80-120kgf. The feedstocks were charged in cavity of the rheometer with 200s heating time to maintain the thermal equilibrium.

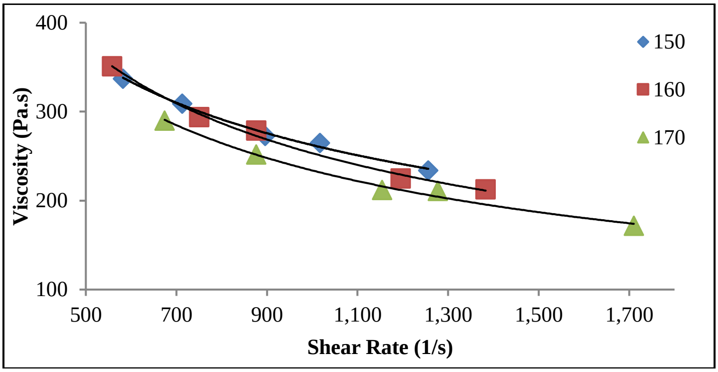
Injection moulding

Injection moulding process is very important to make sure the green parts should be released without difficulty through the die and be hard enough to be ejected without failure [9]. The feedstock was then ejected by using a DSM Xplore plunger type semi-automatic injection moulding machine and the chamber of injection molding machine can be filled with about 10 cm^3 and heated up to 350 ^{0}C. The green parts must be defect free. The density of the green parts needs to be measured by electronic balance (Mettler Toledo) by using distilled water as a medium.

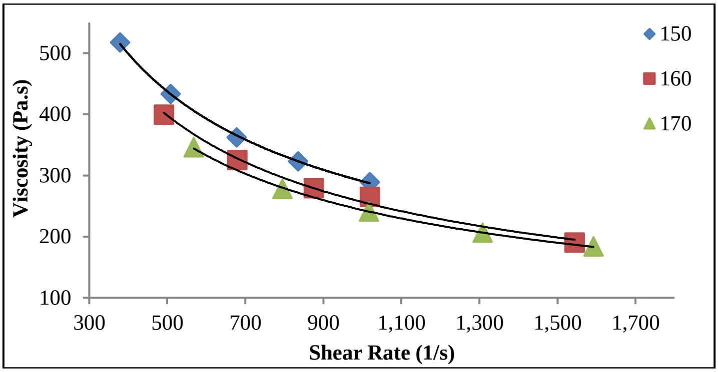
Result and Discussion

One of the characteristics of an ideal binder system for metal injection moulding is powder interaction. The powder interaction includes desirable of the low contact angle, good adhesion with powder and capillary attraction of particle[14)]. As far as particle shape is concerned, it was observed that irregular particles as shown in Fig. 1 and feedstock as shown in Fig. 2 have good in wetting of binder to the powder surface, which will assist the mixing and molding process, and this is called the low contact angle. Besides, the feedstocks have good adhesion with "interlock" situation with one another because from the feedstock result, all the particles have contact with the binder[13)].

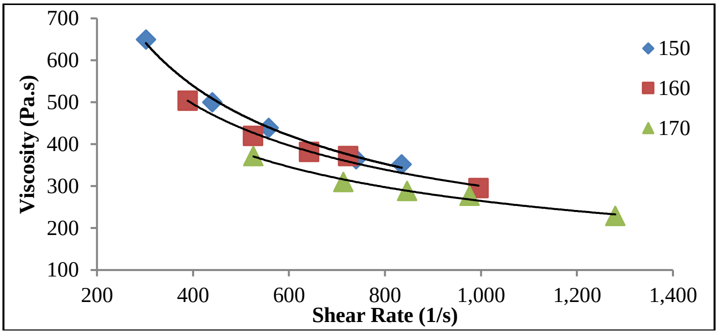
Figure 3 shows their rheological properties in all various powder loading viscosities against shear rates. The feedstock consists of pseudo-plastic behavior that decreases in viscosity with an increase in shear rate which can help to maintain the form of the precast sample, minimize jetting and ease mold filling [10]. From the graph, all the shear rate data varies between 100 and 100,000 1/s, and this is for good moldability. In this shear rate range, viscosity of a required feedstock at molding temperature should be lower than 1000 Pa.s [1] and the result showed for 66 vol.%, 67 vol.% and 68vol.% powder loading was below than 700 Pa.s. When decreasing the feedstock viscosity, the temperature also increases. This happens when heat was introduced with the decreasing of the powder volume and because of the larger extension of the binder. Second, when more heat is distributed to fluctuate the random molecular structure, the disentanglement of the molecular chain happens [1].



(a)



(b)



(c)

Figure 3: Correlation of viscosity and shear rate at (a) 66 vol. % (b) 67 vol. % (c) 68 vol. % powder loading

The purpose of power-law equation is to determine rheological behavior of a pseudo plastic liquid as defined by:

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

where η is the vicosity, γ is the shear rate, K is a constant and n is the power-law exponent which is defined as a flow behavior index of a fluid. In addition, n<1 is defined as a pseudo plastic fluid [1].

Table 2 shows the value n of the experiment. It indicated that the n values for all powder loadings at 150°C, 160°C and 170°C are in range 0.37< x< 0.53. At 66 vol. % powder loading, the temperature increases and the value of n decreases. During injection molding process, lower value of n contributes to a higher shear sensitivity and hence faster the viscosity to decrease with increasing shear rate[3)].

Table 2: Flow behavior index of examined Ti-6Al-7Nb feedstock at different temperatures

Solid Loading (%)	Temperature (°C)	Flow Behavior Index,n
66	150	0.53
	160	0.44
	170	0.45
67	150	0.41
	160	0.37
	170	0.39
68	150	0.39
	160	0.45
	170	0.48
Avarage		0.43

The comparison value of *n* from other researcher is shown in Table 3. By using the same binder system (PA and PE), the same approximated result was obtained. From this comparison, the significant result and the feedstock in this research were suitable for MIM process. Other than that, the differences between the comparisons are the shape, size of particle and composition of binder system.

Table 3: The comparison of flow behaviour index

Researcher	Binder components	n value
Iriany (2002)	92.5 PW - 30 PS- 60 PP -10 PE	0.36
Istikamah (2010)	SS 316L & PSPE 60	0.33
Nor Hafiez (2012)	Titanium & PS PE	0.52
Amir (2014)	Titanium & HA & PS PE	0.33
Sri Yulis (2015)	WC-Co & PS PE	0.62
This research	Titanium Niobium & PS PE	0.43

The influence of temperature on the viscosity of the feedstock is another factor to define fluid characteristics. The correlation of viscosity and temperature can be calculated by Arrhenius equation:

$$\eta = \eta_o \exp\left(\frac{E}{RT}\right) \tag{2}$$

where η_oa viscosity at reference temperature, T is the temperature (K), R is the universal gas constant and E is flow activation energy. The lower sensitivity of viscosity to temperature change refers to the small value of η.

The slope of ln η versus 1/T graph is identical to flow activation energy where temperature reliance of viscosity has a good contract with straight line [9]. Referring to Figure 4, 66 vol. % powder loading graph indicates that it is approaching a straight line as compared to two other powder loadings. By derivation flow activation energies in different shear rates, the $E_{66\%}$=7.21, $E_{67\%}$= 13.68 and $E_{68\%}$=13.02 kJ/mol have been calculated. It indicated that 66 vol. % powder loading is the lowest and the best value for activation energies because the viscosity is not so sensitive to temperature variation when the value E is low. In molded part, the undue stress concentrations can happen due to a sudden viscosity change which may lead to cracking and distortion defects.

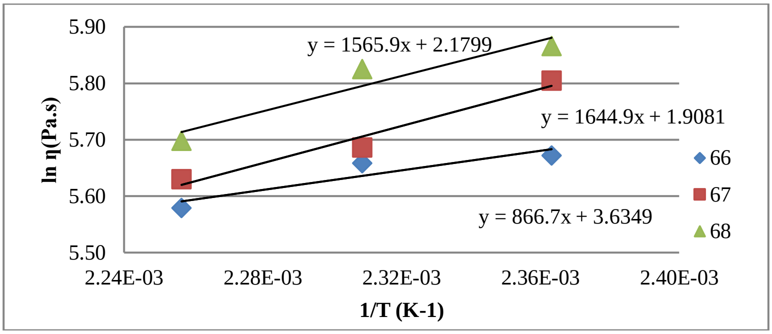


Figure 4: Correlation of viscosity and temperature for feedstock with different powder loading

The mold ability index is the third factor that affects the rheological properties for MIM feedstock. Mold ability index is well known as Weir model, simplifying the above equation:

$$\alpha_{STV} = \frac{1}{\eta_0}\frac{|n-1|}{E/R} \tag{3}$$

where, η_0 is a reference viscosity, E is the activation energy, R is the universal gas constant and n is the power law index [11]. Result obtained from Weir equation is tabulated in Table 3. In this situation, the shear rate reference is at 800 Pa.s and a temperature of 150°C was obtained. According to this Table 3, 66 vol. % powder loading feedstock gives the highest value for rheological index. The higher the value of α_{STV}, the better the rheological properties [11]. The feedstock is considered suitable for injection molding.

Table 3: Comparison of Rheological index at 800 Pa.s shear rate

Solid Loading %	Rheological index, α_{stv}
66	7.305×10^{-5}
67	5.718×10^{-5}
68	1.031×10^{-5}

As shown in Figure 5, injection moulding process was caried out with successfull injection-molded part. The green part with tensile shape design is in good condition and free from injection molding defects such as flashes at the parting line surface, binder separation and short shot [12]. Finally, all the feedstocks are well injected. Table 4 shows the green density result measurement for 66 vol. %, 67 vol. % and 68 vol. % powder loading. From the green density measurement, it indicates that the highest value for green density is 3.322 ± 0.004 g/cm^3 for 68 vol. % powder loading. Meanwhile, the values for 66 vol. % and 67 vol. % powder loadings are 3.275 ± 0.006 and 3.306 ± 0.008 respectively.

Figure 5: Injected feedstock with tensile shape design (green part) without physical defects

Conclusion

The study for the reological behavior of titanium niobium alloy powder mixed with palm stearin and polyethylene binder was succesfully investigated. Feedstock of 66 vol. %, 67 vol. % and 68 vol. % powder loadings indicated pseudo plastic behavior at all temperatures and can be injected successfully through injection molding process.

From the stand point of influence of temperature on rheological properties and the highest rheological index, 66 vol % powder loading feedstock has the highest value of rheological index and the best value for activation energy. Somehow, all the feedstocks have very similar result and can give a worthy result for injection molding. This is because the binder is the main impact on the activation energy, and all the feedstocks used the same binder [11]. Besides, all the green part density results have been measured and it has been proven that the 66 vol. %, 67 vol. % and 68 vol. % powder loadings were successful injected and defect free.

Acknowledgement

Appreciation goes to Universiti Kebangsaan Malaysia for providing the research grant, DIP-2012-29 and ICONIC-2013-003, the Ministry of Higher Education of Malaysia and Universiti Malaysia Perlis for granting the PhD fellowship.

References

[1] B. Randall, M. German, "Injection Moulding of Metal and Ceramics", Metal Powder Industries Federation, Princeton, N.J (1997).

[2] S. L. Baiyun Huang, Xuanhui Qu, "The Rheology of Metal Injection Molding", Journal of Materials Processing Technology, (2003) 132-137.

[3] G. Aggarwal, S. J. Park, I. Smid , "Powder Injection Molding of Niobium", International Journal of Refractory Metals & Hard Materials", 24 (2006) 253–262.

[4] L. L. Yimin Li, K.A. Khalil , "Effect of Powder Loading on Metal Injection Molding Stainless Steels", Journal of Materials Processing Technology", 183 (2007) 432-439.

[5] J.-h. Wang, Q.-n. Shi, C.-l. Wu, J. Xi , "Rheological Characteristics of Injection Molded Titanium Alloys Powder", Transactions of Nonferrous Metals Society of China", 23 (9) (2013) 2605-2610.

[6] M.R. Harun, N. Muhamad, A.B. Sulong, N.H. Mohamad Nor, M.H.I. Ibrahim, "Rheological Investigation of ZK60 Magnesium Alloy Feedstock for Metal Injection Moulding using Palm Stearin Based System", Applied Mechanics and Materials", 44-47 (2011) 4126-4130

[7] F. Ismail, M.A. Omar, I. Subuki, N. Abdullah, E.A.G.E Ali, N. Hassan, "Characterization of the Feedstock for Metal Injection Molding using Bio Polymer Binder", Regional Conference on Engineering Mathematics, Mechanics, Manufacturing and Architecture (EM3ARC), (2007) 85-92.

[8] A. Fayyaz, N. Muhamad, A. B. Sulong, H. S. Yunn, S. Y. M. Amin, J. Rajabi , "Rheological Properties of Cemented Tungsten Carbide Feedstock for Micro Powder Injection Molding", Materials Science Forum, (2014) 827-832.

[9] N. M. H. Abolhasani, "A New Starch-Based Binder for Metal Injection Molding", Journal of Materials Processing Technology, 210 (2010) 961–968.

[10] N. H. L. Z.Y. Liu, S.B. Tor, K.A. Khor , "Characterization of Powder Injection Molding Feedstock", Materials Characterization, 49 (2003) 313-320.

[11] I. Agote, A. Odriozola, M. Gutierrez, A. Santamarıa, J. Quintanilla, P. Coupelle, J. Soares, "Rheological Study of Waste Porcelain Feedstocks for Injection Moulding", Journal of the European Ceramic Society, 21 (2001) 2843–2853.

[12] M. I. Ramli, A. B. Sulong, A. Arifin, A. Muchtar, N. Muhamad , "Powder Injection Molding of SS316L/HA Composite Rheological Properties and Mechanical Properties of the Green Part", Journal of Applied Sciences Research, (2012) 5317-5321.

[13] R. Zauner, D. Heany, J. Piemme, C. Biret, R. M. German, "The Effect of Powder Size on Dimensional Variability in PIM", Advances in Powder Metallurgy and Particulate Materials, (2002) 10-191

[14] D. F. Heaney, "Handbook of Metal Injection Molding", Cambridge, UK: Woodhead Publishing, (2012)