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Original Research Article

Rheological Evaluation of Titanium Alloy Metal Injection Molding (MIM) Feedstock

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Abstract

In PIM process, the molding stage is a critical step for the fabrication of sound parts without cracks and distortions. So, this step requires specific rheological behavior. Rheological analysis can be made to quantify the stability of the PIM feedstock during molding process. In this study, an experimental rheological study has been performed to evaluate the effect of composition binder system on the stability of titanium alloy powder injection molding (MIM) feedstock in term of shear sensitivity and activation energy. The rheology properties of feedstock were measured via capillary rheometer machine. The viscosity of feedstocks was measured at temperature of 120C, 130C, 140C and 150C under different five (5) different constant load at 20N,30N,40N,50N and 60N respectively. The results show that the addition of binder component which possess low molecule weight has reduce flow behavior index value and increase flow activation energy value of feedstock. However, the feedstock produced will get a problem such powder-binder separation and high residue stress which will defect the molded part component. The formulation binder system PEPS 4060 is the best feedstock for titanium alloy MIM process because it has moderate value of flow sensitivity index and flow activation energy value and higher value for mold ability index.

Keywords: Metal injection molding; Rheological behavior; Mold ability index; *titanium alloy (Ti6Al4V)*; *Palm stearin, Paraffin wax, Stearic acid*.

INTRODUCTION

Metal Injection Molding (MIM) is a cost-effective technique for producing small, complex, precision parts in high volumes. The MIM process developed from the traditional shape-making capability of plastic injection molding and the materials flexibility of powder metallurgy [1, 2]. The PIM process consists of four main steps: mixing, injection molding, debinding and sintering [3]. In the mixing step, the powder is mixed with a typical binder volume percentage of 35-50 vol. percent, to form a homogeneous feedstock. The binder is key component, which provides the powder with the flow ability and formability necessary for molding [4]. During molding, the feedstock will flow into barrel and fills a cast mold under certain pressure and temperature to form a green part with the desire shape which the particle powder in that shape.

In MIM process, the molding stage is a critical step for the fabrication of sound parts without cracks or distortions defect such as voids, sinks marks, weld line density variation [5, 6]. Non- homogeneous melt flow and powder -binder separation during the injection molding process may defect the compacts, resulting in cracking and war page during debinding and sintering and ultimately poor physical and mechanical properties of the final parts [7]. For ensure successful injection process the specific rheology behavior is required, so that the rheological characteristics of PIM feedstock are of crucial importance. Rheological analysis can be made to quantify the stability of the feedstock for achieving a successful manufacturing process.

The most important a rheological property in MIM is viscosity, which is relates a shear stress and shear rate. The Viscosity is the single most important predictor of feedstock quality that influences the success of molding stage. High melt viscosity will cause the molding difficulty. The molded part would be obtained when the viscosity is controlled within a narrow range. So, feedstock viscosity with low temperature sensitivity and low sensitivity to shear thinning behavior is desired [8]. Thus, parameters such as flow behavior index, n; activation energy: and mold ability, α; are important in the rheological study.

Most previous study are focusing on investigate the homogeneity and stability of rheological properties MIM feedstock. Huang *et al*., [9] study on the effect of shear rate, solid volume fraction (powder loading) and melt temperature on the rheological behavior of the Fe/Ni MIM feedstock. He concluded that the selected binder system should have stable relationship in both the shear-rate dependence and temperature-dependence of the viscosity. While Karatas *et al*., [10] study on the rheological properties of the ceramic feedstock using polyethylene (PE) and three waxes (carnauba, bees wax and paraffin with steatite powder). The experiment concluded that the feedstock indicated a pseudo-plastic behavior is suitable to be injection molded. Khakbiz *et al*., [8] which studies the influence of TiC addition on the rheological behavior and stability SS 316L MIM feedstock concluded that the rheological behavior of feedstock is highly depend on the blend composition. The instability of feedstock would increase, particularly at higher powder loading. So, by increasing shear rate and temperature, the viscosity decreases, and the instability of the feedstock has improved.

In this study, the palm oil derivative which is palm stearin has been formulated and evaluated as a possible alternative binder system for titanium alloy feedstock. The reason for using palm stearin as binder is due to palm stearin consists of variety fatty acids which can be used as a surface-active agent for powder. Besides that, the primary advantage of palm stearin is their chemistry and rheological properties can be modified to meet the specific requirements of MIM [11].

This paper evaluates the rheological properties in term of their pseudo plastic behavior, index sensitivity, and activation energy and mobility index of titanium alloy feedstock. This involves an investigation to the influence of temperature and shear rate to viscosity of the MIM feedstock.

EXPERIMENTAL PROCEDURES

Starting Material

In this research, gas atomized Ti-6Al-4V powders (Ti-5.9 Al-3.9 V-0.19 Fe-0.12 O-0.01 N-0.004) provided by TLS Technik Spezialpulzer (German) was used. The particle size distribution of the powder was determined using CILAS 1190 Dry equipment while the morphology of powder was observed using scanning electron microscope. The characteristic of the powder is as shown in Table 1; Fig 1 shows SEM micrographs of the particles power.

Table 1: Characteristic of titanium alloy (Ti6Al4V) metal powder

Particle Shape	Spherical
D_{10}	D10=5.27 um
D_{50}	D50=16.11um
D_{90}	D90=28.53 um

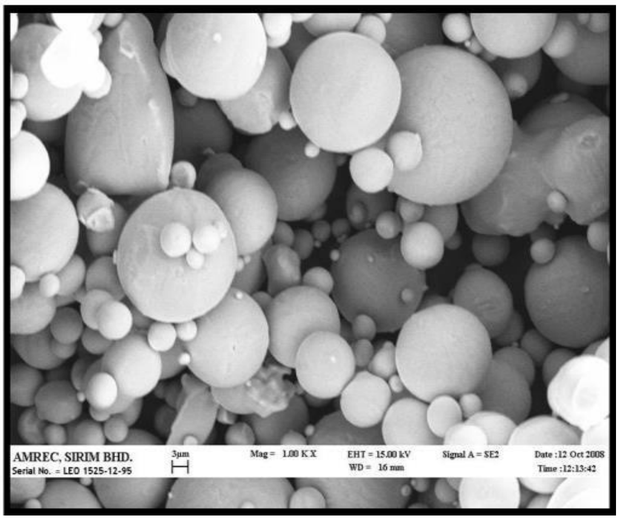


Fig 1: SEM micrograph of gas atomized titanium alloy (Ti6Al4V) powder

Compounding

In order to study the effect of composition binder system to the rheological behavior of the titanium alloy feedstock. Three (3) titanium alloy feedstock which compose with different composition binder system consisting locally binder based palm stearin, commercial paraffin wax and stearic acid; and polyethylene were formulated as shown in Table 2.

Table 2: The composition of binder systems

Abbreviation	Polyethylene (vol.%)	Palm Stearin (vol.%)	Paraffin wax (vol.%)	Stearin wax (vol.%)
PE/PW/SA	35	-	55	10
PE/PS 4060	40	60	-	-
PE/PW/PS	35	10	55	-

The powder and binder were mixed using Z-blade mixer with rotation frequency of 25 rpm. The mixer temperature was set at 160°C. The components of the binder system were added simultaneously to the powder at room temperature for 5 minutes and the mixing process continued until 2 hour. After mixing, the dough is removed from the mixer and cooled to 60°C, before the dough is subsequently fed into a strong crusher in order to produce homogenized granules.

Rheological Properties Measurement

The feedstock viscosity was measured using a CFT-500D Shimadzu Capillary Rheometer. During the Capillary Rheometer test, the feedstock was forcibly extruded through a small cylindrical orifice with a 1.0 mm diameter and 1.0 mm length (L/D=1). The palletized feedstock were placed in the rheometer barrel and allowed to preheat for 300s before initiating testing under constant load at 20N, 30N, 40N, 50N and 60N respectively.

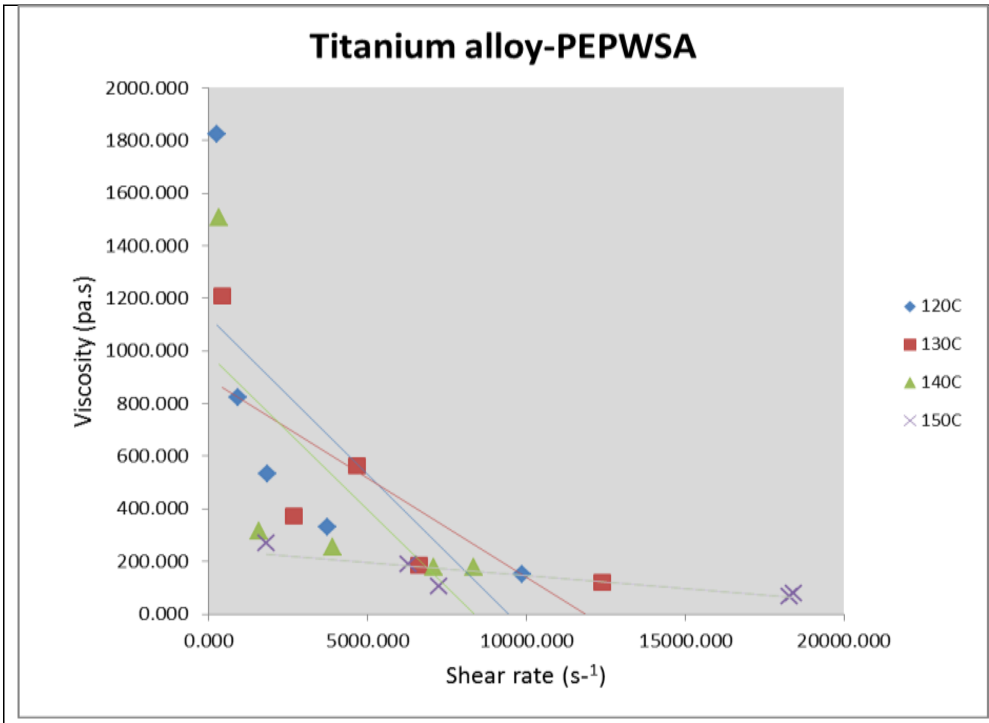
RESULT AND DISCUSSION

1. Rheogical Characteristic

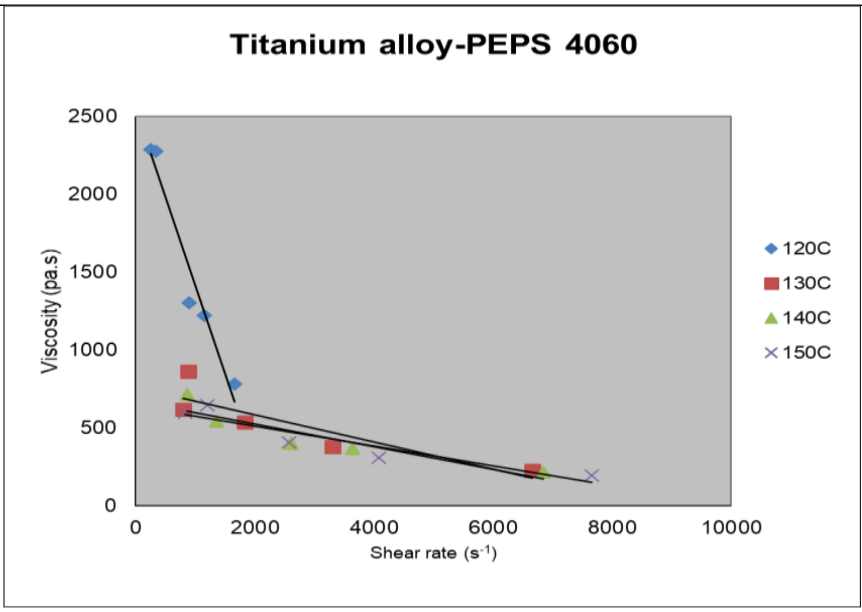
Initially, for successful molding, the feedstock melt viscosity should exhibit a shear thinning or pseudo-plastic behavior since increasing shear rates produce lower viscosities that will assist mold filling. The viscosity of a pseudo-plastic substance decreases as the shear rate increase due to particle agglomerates released together with the binder [12]. In Table 3, the viscosity data for different composition of palm stearin feedstock (Pa.s) at different test load and temperatures are reported.

Table 3: The viscosity data for different composition of palm stearin feedstock (Pa.s)

Feedstock	Temperature (°C)	Applied load (N)				
		20	30	40	50	60
	120	1822.000	824.800	532.033	329.167	149.733
PE/PW/SA	130	1207.333	562.078	370.833	185.367	119.000
	140	1508.333	317.000	253.133	176.000	176.367
	150	267.767	103.993	188.933	67.003	79.557
	120	2290.667	2275.333	1302.600	1222.633	785.000
PE/PS/4060	130	1018.367	864.600	539.933	380.533	227.600
	140	716.467	543.200	400.067	369.400	215.500
	150	598.533	649.030	405.133	308.000	196.500
	120	1940.667	390.400	328.833	125.900	113.267
PE/PW/PS	130	592.267	635.333	271.767	81.580	83.957
	140	375.567	197.000	90.000	70.037	82.127
	150	237.967	167.767	65.710	70.007	82.443



(a)



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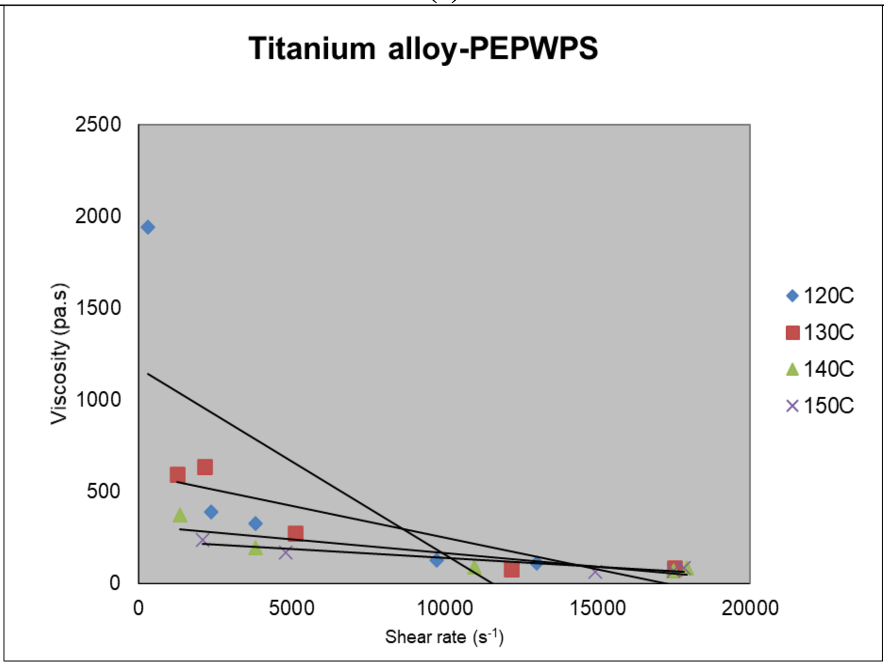

(c)

Fig 2: Viscosity graph for different composition palm stearin (a) PE/PW/SA, (b) PE/PS 4060 and (c) PE/PW/PS

The Fig 2 shows the effect of different composition binder system on viscosity behavior of feedstocks. All the formulation of feedstocks indicates its stability in decreasing viscosity and pseudoplastic behavior when evaluate at temperature 120°C under different constants load at 20N, 30N, 40N and 50N. The viscosity result of all these formulations of feedstocks when evaluated at this parameter were meet requirement MPIF 35 standards and acceptance for MIM process. However, the formulation PE/PW/SA feedstock only show stability for decreasing of viscosity and pseudoplastic behavior at temperature 120°C and 130°C. The viscosity properties of formulation PE/PW/SA feedstock show stability pseudoplastic behavior at temperature 120°C, 130°C and 140°C. While the formulation of PE/PW/PS feedstock indicate fluctuate decreasing viscosity and instability pseudoplastic behavior after evaluated at temperature over than 120°C. This is due to the molecule weight of PS and PW content in formulation feedstock of PE/PW/PS were lower than polyethylene. In addition, the variety fatty acids content in PS with different melting point temperature which acts as surfactant agent for metal powder particle surface has increase mobility of particle powder and consequence powder-binder suspensions. These similar results also have been reported by Tseng. W. J *et al.*, (1999) [13] in his studied that the increasing stearic acid in feedstock formulation will decrease viscosity of feedstock but increasing powder-binder suspensions.

2. The flow behavior index of feedstocks

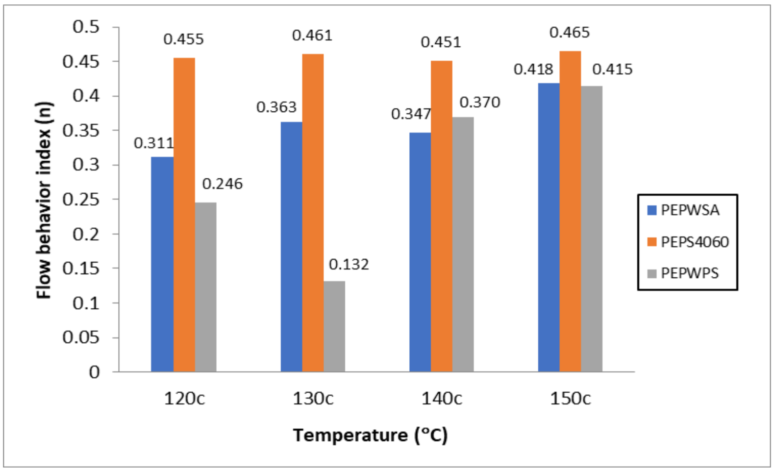

Fig 3: The flow behavior index of feedstocks at 120°C, 130°C, 140°C and 150°C

The calculation of flow behavior index, n value is must be considered prior injection molding process to determine the ability of feedstock to flow. The ability of feedstocks to flow is greatly depend on the shear rate due to the applied load at given temperature. The equation (1) can be used to relate viscosity to shear rate at a given temperature to determine flow behavior index value.

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

Where η is the feedstock viscosity, γ the shear rate, K a constant and n is the flow behavior index. The value of n is very important in the rheological characteristic of MIM feedstock. The value of n indicates the degree of shear sensitivity of feedstock and it must smaller than 1 according requirement standard MPIF 35. Fig 3. show the effect of temperature on the value flow behavior index of the MIM feedstocks. The graph show, all the feedstock exhibits a shear thinning or pseudo plastic behavior, as the flow behavior index is smaller than 1. The lower value of the flow behavior index means the more sensitivity of viscosity on the shear rate. Unfortunately, too lower value of the flow behavior index would produce some defect such as jetting, weld line and imperfection in the final molded part [12]. However, the high shear sensitivity value will provide better stability of feedstock during injection molding because of the viscosity will decreased slowly with increasing shear rate [15]. However, all the formulation of feedstocks possesses moderate value of flow behavior index, n and has fulfills requirement of MPIF 35 standard.

3. Flow activation energy of feedstock

Another important characteristic of MIM feedstock is temperature-dependence of viscosity. Theoretically, pure binder has a viscosity that usually varies exponentially with absolute temperature. Therefore, the influence of temperature on the viscosity of feedstock can be evaluated according to the following Arrhenius equation as below:

$$\eta = K\gamma^{n-1} \exp\left(\frac{E}{RT}\right) \quad \text{………………. (2)}$$

Where E is the flow activation energy, R the gas constant and T the temperature in Kelvin unit. High value of flow activation energy indicates a strong temperature-dependence on the feedstock to the viscosity. Therefore, any small fluctuation of temperature during injection molding result in a sudden viscosity change, resulting defects in molded part, such as cracking and distortion due the stress concentration. While, the small value of the activation energy, E indicates that the viscosity of the feedstock is low sensitivity to temperature, thus any fluctuation of temperature during injection molding process might not results on sudden viscosity change that can cause molding part defect such as stress concentration, crack and distortion in the molded part [12].

The flow activation energy of tested feedstock as function of shear rate is shown in Fig.4. It is visible the formulation feedstock of PE/PS 4060 possess moderate value of flow activation energy at increasing load applied. While the feedstocks of PE/PW/SA and PE/PW/PS indicate possess high values of flow activation energy and fluctuate with increasing load applied. The fluctuate of flow activation energy value may be due to occurrence of powder-binder separation phenomenon during rheology testing [12].

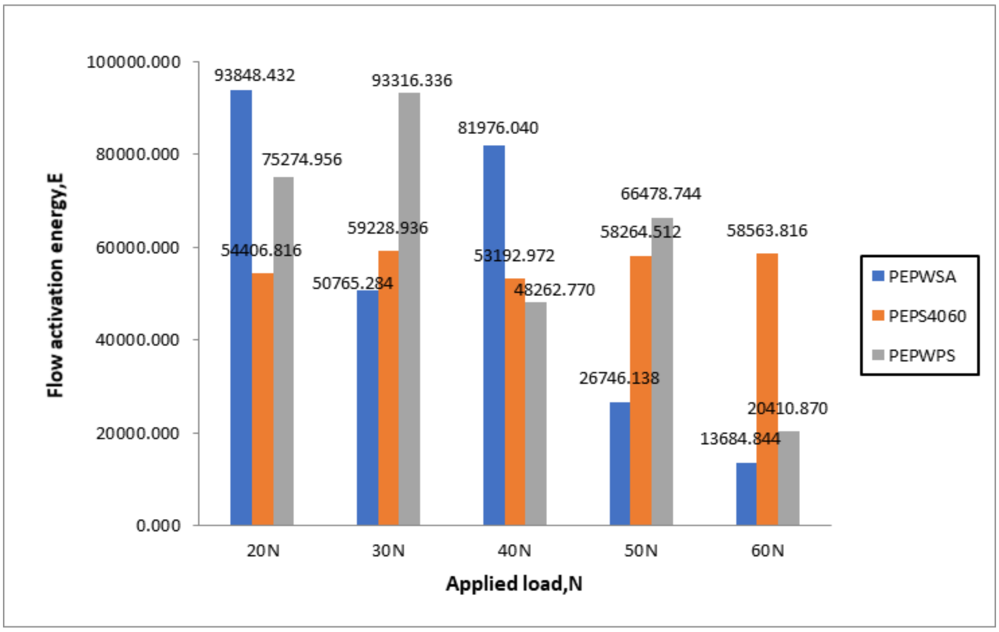

Fig 4: Flow activation energy of feedstock at load 20N,30N,40N,50N and 60N

4. Mold ability Index, α

It is known that the ideal feedstock which possesses low temperature sensitivity to shear rate and high values of flow activation energy is desire in MIM process. However, there is still have a conflict between these two parameters in terms of requirements. Thus, to determine the final decision for the best selection of feedstock, the weir equation was used by previous researchers to determine mold ability index, α value [18, 19]. These equations are combined with both the above parameters and are given in the following equation.

$$\alpha = 10^9 \, (n)/\eta_o \, (E/R) \quad \ldots\ldots\ldots\ldots (3)$$

Where E is activation energy, R constant gas and α is the apparent viscosity at reference shear rate at 100 and 1000s^{-1}. The calculation feedstock mold ability index, α for different formulation of composition binder system at temperature 140°C and applied load 40N is show in Table 4.

Table 4: The feedstock of mold ability index, α for different composition of PS

Feedstock	Moldability Index,α
PE/PW/SA	5091.34
PE/PS 4060	10197.93
PE/PW/PS	9221.03

The results show that the formulation composition of binder system for PE/PS 4060 feedstock show higher mold ability index value. The higher value of the mold ability index is desired able in MIM process since this value indicate the feedstocks has high ability to be molded during injection molding process. So, the feedstock PE/PS 4060 is consider as the best feedstock for titanium alloy MIM process.

CONCLUSION

All the feedstock which using gas atomized titanium alloy (Ti6Al4V) powder in this study show pseudo plastic behavior. The addition of binder component which possess low molecule weight has reduce flow behavior index value and increase flow activation energy value of feedstock. However, the feedstock produced will get a problem such powder-binder separation and high residue stress which will defect the molded part component. The formulation composition binder system of PE/PS 4060 feedstock is considered as the best feedstock for titanium alloy MIM process due to it have moderate value for flow behavior index and flow activation energy; and high moldability index value.

REFERENCE

1. German, R. M. (1997). Injection molding of metals and ceramics. *Metal powder industries federation*, 3-10.
2. Merhar, J. R. (1990). Overview of metal injection moulding. *Metal Powder Report*, *45*(5), 339-342.
3. Liu, Z. Y., Loh, N. H., Tor, S. B., & Khor, K. A. (2002). Characterization of powder injection molding feedstock. *Materials characterization*, *49*(4), 313-320.
4. Chung, C. I., & Rhee, B. O. (1987). Requirements of binder for powder injection moulding, *Compending Metal Injection Moulding*, 2(5), 269-277.
5. Mutsuddy, C., & Ford, R. G. (1995). Ceramics Injection Moulding, Champion Hall, London.
6. German, R. M., & Bose, A. (1997). Injection Moulding of Metals and Ceramics, MPIF, Princeton, New Jersey.
7. Dihoru, L. V., Smith, L. N., & German, R. M. (2000). Experimental analysis and neural network

modelling of the rheological behaviour of powder injection moulding feedstocks formed with bimodal powder mixtures. *Powder metallurgy*, *43*(1), 31-36.

8. Khakbiz, M., Simchi, A., & Bagheri, R. (2005). Analysis of the rheological behavior and stability of 316L stainless steel–TiC powder injection molding feedstock. *Materials Science and Engineering: A*, *407*(1-2), 105-113.
9. Huang, B., Liang, S., & Qu, X., (2003). The rheology of metal injection molding, *Journal of Materials Processing technology*, 1237, 132-137.
10. Karatas, C., Kocer, A., Ünal, H. I., & Saritas, S. (2004). Rheological properties of feedstocks prepared with steatite powder and polyethylene-based thermoplastic binders. *Journal of Materials Processing Technology*, *152*(1), 77-83.
11. Ismail, F., Omar, M. A., Subuki, I., Abdullah, N., Ali, E. A. G. E., & Hassan, N. (2007). Characterization of the feedstock for metal injection molding using biopolymer binder. In *Reg. Conf. On Eng. Math., Mech., Manu., & Arch (EM 3 ARC)* (pp. 85-92).
12. Yulis, S., Amin, M., Khairur Rijal, J., & Norhamidi, M. (2009). Rheological properties of SS316L MIM feedstock prepared with different particle sizes and powder loadings. *Journal The Institution of engineering, Malaysia*, 71(2), 59-63.
13. Tseng, W. J., Liu, D. M., & Hsu, C. K. (1999). Influence of stearic acid on suspension structure and green microstructure of injection-molded zirconia ceramics. *Ceramics International*, *25*(2), 191-195.
14. Iriany. (2002). Rheology properties study on the feedstock contains palm stearin for metal injection molding, Doctor of Philosophy Thesis, National University of Malaysia.
15. Omar, M. A., Subuki, I., Abdullah, N., & Ismail, M. F. (2010). The influence of palm stearin content on the rheological behaviour of 316L stainless steel MIM compact. *Journal of Science and Technology*, *2*(2), 1-14.
16. Tseng, W. J., Liu, D. M., & Hsu, C. K. (1999). Influence of stearic acid on suspension structure and green microstructure of injection-molded zirconia ceramics. *Ceramics International*, *25*(2), 191-195.
17. Yimin, L., Xuanhui, Q., Baiyun, H., & Guanghan, Q. (1997). Rheological properties of metal invection molding binder and feedstock. *Trans Nonferrous Met Soc China*, 7(3).
18. Yulis, S., Amin, M., Khairur Rijal, J., & Norhamidi, M. (2009). Rheological properties of SS316L MIM feedstock prepared with different particle sizes and powder loadings. *Journal- The Institution of Engineers, Malaysia*, 71(2).
19. Abolhasani, H., & Muhamad, N. (2009). Rheological investigation of a starch-based binder and feedstock for metal injection molding. *International Journal of Mechanical and Materials Engineering (IJMME)*, *4*(3), 294-299.