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# Injection Molding of Inconel718 Parts for Aerospace Application Using Novel Binder System Based on Palm Oil Derivatives

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***Abstract*****—**Inconel718 has been widely used as a super alloy in aerospace application due to the high strength at elevated temperatures, satisfactory oxidation resistance and heat corrosion resistance. In this study, the Inconel718 has been fabricated using high technology of Metal Injection Molding (MIM) process due to the cost effective technique for producing small, complex and precision parts in high volume compared with conventional method through machining. Through MIM, the binder system is one of the most important criteria in order to successfully fabricate the Inconel718. Even though, the binder system is a temporary, but failure in the selection and removal of the binder system will affect on the final properties of the sintered parts. Therefore, the binder system based on palm oil derivative which is palm stearin has been formulated and developed to replace the conventional binder system. The rheological studies of the mixture between the powder and binders system have been determined properly in order to be successful during injection into injection molding machine. After molding, the binder holds the particles in place. The binder system has to be removed completely through debinding step. During debinding step, solvent debinding and thermal pyrolysis has been used to remove completely of the binder system. The debound part is then sintered to give the required physical and mechanical properties. The results show that the properties of the final sintered parts fulfill the Standard Metal Powder Industries Federation (MPIF) 35 for MIM parts.

***Keywords***—Binder system, rheological study, metal injection molding, debinding and sintered parts.

## I. Introduction

SUPERALLOYS are a kind of heat-resisting alloys based on nickel-iron, or cobalt, exhibiting a high strength at elevated temperatures, satisfactory oxidation resistance and heat corrosion resistance. These properties make them useful for many applications in the aerospace, automotive, medical, chemical, and petrochemical industries, but their high strength and toughness make them difficult to shape via machining or forging [1].

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Therefore, to overcome the problem of machining or forging, an alternative process called Metal Injection Molding (MIM), which can offers improved cleanliness, better element homogeneity, finer grain size and a more uniform distribution of precipitating phases MIM is known as a cost-effective technique for producing small, complex, precision parts in high volume [2-5]. The MIM process was developed from the traditional shape-making capability of plastic injection molding and materials flexibility of powder metallurgy. The process consists of four main steps: mixing, injection molding, debinding and sintering [6-9].

In this study, the Inconel718 powder will be used in the investigation to prepare and produce the potential aerospace application parts such as wheels, buckets, spacers, and high temperature bolts and fasteners. The novel binder system based palm oil and thermoplastic will be mixed to prepare the feedstock. Before mixing, the solid loading between the powder and the binder system has to be determined to measure the optimum solid loading for the mixture. The rheological study in term of compatibility and homogeneity of the mixture will be measured using capillary rheometer [10-17]. After molding, the binder holds the particle in place. The binder will be then removed in the debinding step through solvent extraction. The debound part will be then sintered at different temperature ranging from 1100°C to 1300°C under vacuum atmosphere to give the required physical and mechanical properties [18-19].

## II. Experimental Procedure

The Inconel718 powder was supplied by Epson Atmix Corporation with a mean particle size of 8 μm was used in the investigation. Chemical composition of the powder is listed in Table I. The morphology of the Inconel718 powder is shown in Fig. 1 was determined by Scanning Electron Microscopy (SEM). It shows that the particle shape of the powder is irregular shape. This leads to an optimum solid loading in the feedstock between 45 to 50 volume percent was determined by Brabender Plasticorder and it is possible to make a good moldings.

TABLE I
Chemical Composition of the Inconel718 Powder

| Element | Ni | Cr | Fe | Nb | Mo | Ti | Al | Cu | C |
|---|---|---|---|---|---|---|---|---|---|
| Content | 52.60 | 19.17 | Bal. | 5.27 | 2.96 | 0.71 | 0.18 | 0.01 | 0.035 |

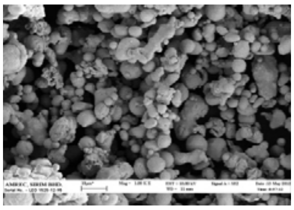


Fig. 1 The particle morphology of the Inconel718 powder

Combination of polyethylene and palm oil which is palm stearin was used as the binder system. The binder system comprised of 40% of polyethylene and 60% of palm stearin (percentage by weight) as shown in Table II.

TABLE II
DIFFERENT COMPOSITION OF BINDER SYSTEMS

| Binder | System A (vol. %) |
|---|---|
| Polyethylene | 40 |
| Palm Stearin | 60 |

The melting temperature of each of the binders system was measured using Differential Scanning Calorimetric (DSC) is shown in Table III.

TABLE III
MELTING TEMPERATURE OF EACH BINDER SYSTEM

| Binder | Melting Temperature (°C) |
|---|---|
| Polyethylene | 125.86 |
| Palm Stearin | 54.22 |

Determination of critical and optimum solid loading of the mixture powder/binder was measured using Brabender Plasti-Corder. The weight of Inconel718 powder of 245.5 gram was poured into mixer bowl at different volume percent of oleic acids. The powder then was mixed with the binder system at the temperature of 130 to 160°C for 2 h using Z-Blade Mixer. The feedstock then was studied in term of rheological which is the viscosity, shear rate, shear sensitivity and activation energy was measured using Capillary Rheometer. The Vertical Injection Molding Machine was adopted to mold into the tensile test specimen. The green molded specimens were subjected to a solvent extraction step where two third of the binder system was removed. The green molded specimens then were immersed into the heptane for 6 h at the temperature of 60°C. The specimen then was continued heated at rate between 0.25 to 7.14°C/min up to 1100°C and 1200°C under vacuum atmosphere with holding time of 8 h. Fig. 2 shows the schematic process of Metal Injection Molding (MIM) technique.

The density of each of the sintered specimen was measured using Densitometer ED-120T. The porosity of the each sintered specimen was measured using Image Analyzer. The weight loss and shrinkage of the sintered specimen also was measured. Optical micrograph was used to observe the microstructure of sintered specimen.

## III. RESULTS AND DISCUSSION

### *A. Optimum Solid Loading and Rheological Study*

Fig. 3 show torque value versus solid loading of the mixture between powder and binder. From the results, the critical and optimum solid loading of the powder is 54.97% and 50.00% respectively [5].

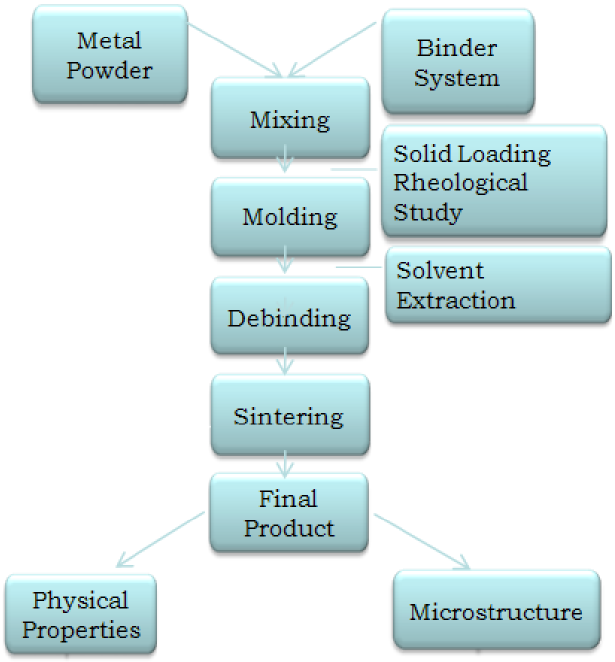



Fig. 2 Show the schematic process of Metal Injection Molding (MIM) technique

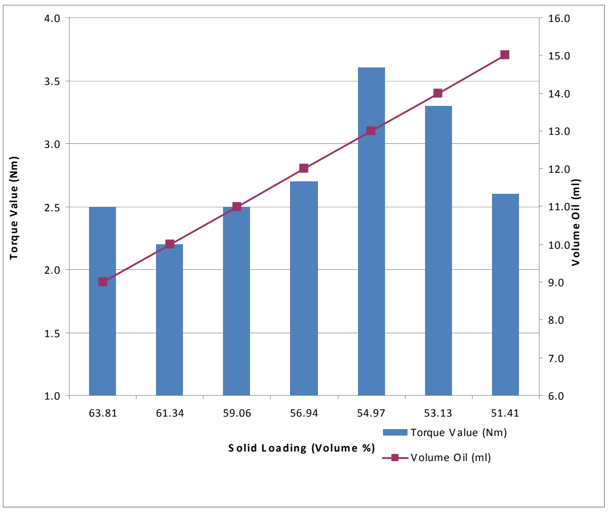



Fig. 3 The torque value versus solid loading of the mixture

Fig. 4 shows the viscosity versus shear rate at different temperature of Inconel718 feedstock. The graph indicated clearly that as the viscosity decrease the shear rate increase relatively to the increasing of the temperature. The results conclude that the viscosity of the feedstock exhibit pseudo-plastic behavior, which is common for MIM feedstock. The variation of viscosity versus shear rate in log-log scale graph is almost linear, which is an indicator of feedstock stability. In order to inject the feedstock into injection molding machine, the shear rate and viscosity should be in the range of 100 $s^{-1}$ to 10000 $s^{-1}$ and 1000 Pa s respectively. The recommended temperature range that can be injected for the Inconel718 feedstock is 106°C to 114°C. Pseudo-plastic fluid of the feedstock also indicates the decreasing of viscosity with increase shear rate and temperature. At low temperature the feedstock viscosity is too high for standard molding conditions, while at high temperature results in binder separation that leads to defect of the injected parts.

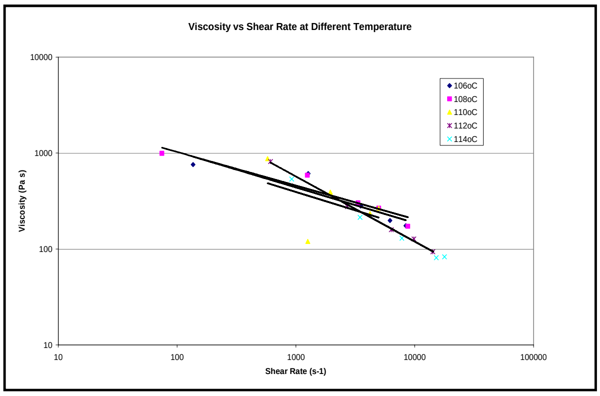



Fig. 4 The viscosity versus shear rate at different temperature of Inconel718

The feedstock that exhibit as a pseudo-plastic behavior is a common for MIM feedstock. This could be due to particle orientation and ordering with flow as well as breakage of particle agglomerates with release of fluid binder [3]. For pseudo plastic fluid, it can be expressed by the following equation;

$$\tau = K\gamma^{n} \qquad (1)$$

Where $\tau$ is the shear stress, $\dot{\gamma}$ is the shear rate, *n* the flow behavior index and K a constant. In (1) has been widely used to correlate the data of shear stress to shear rate for pseudo plastic and dilatants fluids which known as the power-law equation. The exponent *n* of the power-law index indicates the shear sensitivity. Smaller *n* of feedstock indicates higher shear sensitivity and more pseudo plastic behavior of the feedstock while *n* >1 indicated a dilatants material where the metal powder and binder would separate under high shear rate [10, 11]. The mathematical significance of *n* can be more clearly obtained from the logarithmic form of the above power-law (2).

$$log_{10}\,\tau = log_{10}\,k + n\ log_{10}\,\gamma \qquad (2)$$

The most important rheological property for a metal powder feedstock is viscosity (η) which is defined by (3):

$$n = \frac{\tau}{\gamma} \qquad (3)$$

If we consider the relationship between the apparent viscosity η and power-law index *n* as show in (4) in logarithm form, we can find that value | *n* -1| also represent the sensitivity of viscosity to shear rate.

$$n = \frac{\tau}{\gamma} = K\frac{\gamma^{n}}{\gamma} = k\,\gamma^{n-1} \qquad (4)$$

The exponent *n* which represent the shear sensitivity, which is can calculated from the slope of the curve in Fig.5 using (4). The Table III show the value of *n* of feedstock which is smaller than 1 and fall between 0.011 and 0.444 for all the working temperatures, meant the flow behavior of the feedstock is obey the pseudo plastic fluid, and the *n* value is consistent with small differential meant this feedstock is homogeny. Refer to viscosity of feedstock when temperature at 106°C, 108°C, 110°C, 112°C and 114°C was applied respectively.

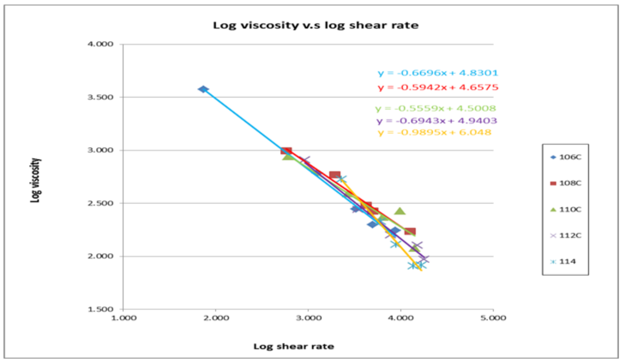



Fig. 5 The log viscosity versus log shear rate at different temperature of Inconel718

The viscosity at temperature 106°C, 108°C, 110°C and 112°C were fall progressively and not much different their value between each other. From analysis data we can conclude that the formulation gradient of the feedstock is fulfill requirement of MIM standard which MIM standard have specific shear sensitivity value must around 0< *n*< 1 (Table IV).

TABLE IV
SHEAR SENSITIVITY (*N*) OF THE INCONEL718 FEEDSTOCK

| Temperature (°C) | Shear sensitivity ( *n*) |
|---|---|
| 106 | 0.330 |
| 108 | 0.406 |
| 110 | 0.444 |
| 112 | 0.306 |
| 114 | 0.011 |

The second important aspects of the rheological property for a MIM feedstock are the temperature-dependence of viscosity. Normally, as a good approximation, the Arrhenius (5) can be used as followed:

$$n = n_o \exp\left(\frac{E}{RT}\right) \qquad (5)$$

Where *E* is the flow activation energy, *R* is the gas constant and *T* is the temperature in Kelvin. According to the (5), a graph plot as shown in Fig 6, ln (viscosity) vs. 1/T at a certain shear rate are obtained. The graph is fit into straight line.

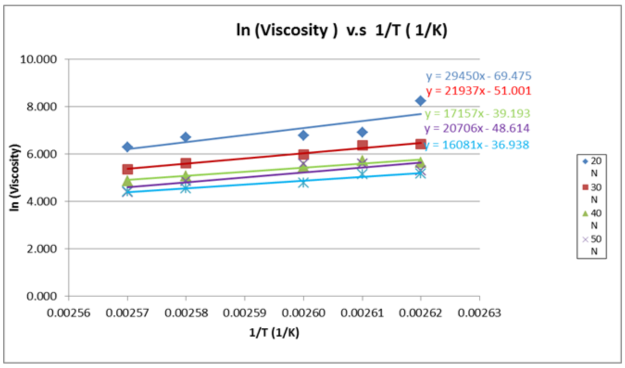



Fig. 6 ln (viscosity) versus 1/T at different loading

The slope of the graphs indicates the temperature-dependence of viscosity, which should be as small as possible to avoid sharp viscosity changes that reduce the flow ability of the feedstock and cause stress concentrations, cracking and distortion on the molded parts.

The Table V was shown activation energy value of feedstock which analyses by graph in Fig 5. From the Table V, the flow activation energy value of the feedstock is moderate, which indicates that the viscosity of feedstock is not sensitive to temperature. So, this feedstock can be injected into mold in a relatively wide temperature range without any problem although fluctuation temperatures of mold occur.

TABLE V
FLOW ACTIVATION ENERGY; E DATA (JOULE/MOL)

| Load (n) | Temperature (1/T) | Activation Energy (joule) |
|---|---|---|
| 20<br>30<br>40<br>50<br>60 | 0.00257 | 244847.2706 |
| 20<br>30<br>40<br>50<br>60 | 0.00258 | 182384.1961 |
| 20<br>30<br>40<br>50<br>60 | 0.00260 | 142643.2808 |
| 20<br>30<br>40<br>50<br>60 | 0.00261 | 172149.6633 |
| 20<br>30<br>40<br>50<br>60 | 0.00262 | 133697.4179 |

*B. Physical Properties and Microstructure*

The physical properties of the sintered Inconel718 specimen were shown in Table VI. The results show that the density of sintered Inconel718 specimen at the temperature of 1100°C and 1200°C is roughly 6.46 g/cm$^3$ and 7.51 g/cm$^3$ respectively.

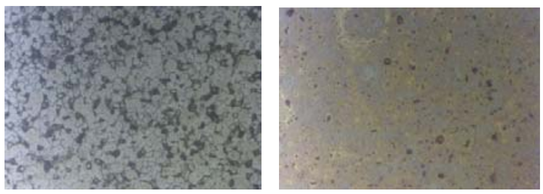
The density has achieved only 78.99% and 91.69% compared to theoretical density of Inconel718 which is 8.19 g/cm$^3$. The porosity of sintered Inconel718 specimen at the temperature of 1100°C and 1200°C is 21.04% and 8.30% respectively. It is shows that as the density increased, the porosity decreased.

The results of weight loss of sintered Inconel718 specimen at the temperature of 1100°C and 1200°C is 11.20% and 11.91% respectively. Meanwhile the shrinkage of sintered Inconel718 specimen at the temperature of 1100°C and 1200°C is 15.88% and 25.46% respectively. It is shows that as the temperature increased, the shrinkage is also increased proportionally.

Microstructure observation using optical micrograph clearly shows that at the temperature of 1100°C, the porosity is highest and many irregular pores can been seen clearly in the Fig. 7 (a). At the temperature of 1200°C (Fig. 7 (b)), the porosity is significantly decreased and the microstructure with small and spherical pores can be seen clearly.

TABLE VI
THE PHYSICAL PROPERTIES OF THE SINTERED INCONEL718 SPECIMEN

| Properties | Sintered Inconel718 Specimen at 1100°C | Sintered Inconel718 Specimen at 1200°C |
|---|---|---|
| Density (g/cm$^3$) | 6.47 (78.99%) | 7.51 (91.69%) |
| Porosity (%) | 21.04 | 8.30 |
| Shrinkage (%) | 15.88 | 25.46 |
| Weight Loss (%) | 11.20 | 11.91 |



(a) (b)

Fig. 7 The micrograph observation at the temperature of (a) 1100°C (b) 1200°C of the sintered Inconel718 Specimen

## IV. CONCLUSION

It can be concluded that the viscosity plays a big role in predicting the injected parts by considering the shear sensitivity, *n* and flow activation energy, *E*. From the experiments carried out, the Inconel718 feedstock based on the palm oil binder was shown a pseudo plastic flow behavior. The shear sensitivity value of feedstock is below than 0.5 which is mean the viscosity of feedstock is sensitive with shear rate. While the activation energy of feedstock is moderate which it means the feedstock is not sensitive to temperature. So, the feedstock can be injected into mold in a relatively wide temperature range without any problem although fluctuation temperatures of mold occur. The properties of the sintered Inconel718 specimen do not achieve the minimum requirement for sintered MIM parts.

## ACKNOWLEDGMENT

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**Rosdi Ibrahim, PhD.,** completed his first degree in Physic Nuclear from Universiti Kebangsaan Malaysia in 1993. He pursued his M.Sc. in Nuclear Physics at Department of Physics, Universiti Putra Malaysia in 1994. Dr. Rosdi started his career as a research officer at Malaysia Institute for Nuclear Technology (MINT), now known as Agency Nuclear Malaysia from December 1994 until Mac 1995. Dr. Rosdi started his career as a research officer at Ceramic Technology Center, SIRIM in middle of Mac 1995 and joint Advanced Materials Research Centre (AMREC), SIRIM Berhad in a year after. He worked on the project entitle “Development of High Performance Near Net Shape Automotive Components Through Powder Metallurgy Technique” and “Development of High Performance Neodymium Iron Boron Permanent Magnet through Powder Metallurgy Technique”. After 3 years of attachment with AMREC, Dr. Rosdi left for Universiti Putra Malaysia in end of 1998 to pursue his doctorate studies in Materials Science majoring in Metal Injection Molding of Stainless Steel. Dr. Rosdi was successfully managed to finish his research work within 3 years. His Ph.D. work inspires him to initiate some research focusing on developing an alternative novel binder system based on palm oil derivatives, which was demonstrated in 4 research projects awarded by Internal Seed Funding SIRIM Berhad and Ministry of Science, Technology and Innovation (Seed Fund, RM80,000.00), Science Fund SF0004 (MOSTI, RM185,716.00), Science Fund SF0093 (MOSTI, RM271,600.00) and Techno Fund TF0206D124 (MOSTI, RM3,658,583.00).

The Internal Seed Funding and Science Fund SF0004 projects concentrated on the developing an alternative binder system based on palm oil derivatives and the use of developed novel binder system for orthopaedic and orthodontic application. His effort has resulted in a 2 patents on the title “An Improved Binder-System for use in Injection Moulding of Metal Powders” - patent pending number is PI 20064276 and “A Method for Removal Binder Materials from Derivatives of Palm Oil in Powder Injection Molding” - patent pending number is PI20072243 which was claimed to be more environmental friendly and very economical. This invention has also won him a gold medal in MTE 2006, ITEX 2007, MTE2008, BIO INNO Awards 2011, MTE 2012, and ITEX 2012, silver medal in MTE 2007 and ITEX 2008 and a bronze medal in ITEX 2006 and BIO INNO AWARDS 2008. Several publications in conferences and journals have also been made pertaining to this work. Following the success of this internal seed funding and Science Fund MOSTI project, Dr. Rosdi ventured into a more challenging pre-commercialization research work title “Up-Scaling of Prototype Titanium Alloy Implant for Dental and Craniofacial Application by Powder Injection Moulding Technique using Palm Oil Based Binder System”. This work is also fully supported by the Ministry of Science, Technology and Innovation, a project which runs in 3 years duration. This project has shown many successes in design and biomechanical analyses for the expanding the implant inventory in-line with clinical needs and application; manufacturing process of titanium alloy implants prototype using powder injection moulding technique; regulatory and standards compliance on the biocompatibility and regulatory and standards compliance of prototype produced through prospective clinical trial for marketing purposes. This project also successes come out with a prototyping set of medical box including instruments, plates and screw for surgery purposes.

In December 2007, Dr. Rosdi successfully managed to collaborate with Advanced Manufacturing Research Institute of National Institute of Advanced Industrial Science and Technology, Japan under Memorandum of Understanding title “Powder Metallurgy and Processing Techniques” with exclusively for 5 years duration. Therefore, in February 2009 and October 2009, Dr. Rosdi enrolled himself in a professional research attachment on “Sintering and Super Critical $CO_2$ Debinding Technique of Titanium Alloy and Stainless Steel 316L using the Powder Injection Molding (PIM) Technique, at Low Formability Materials Processing Group, Advanced Manufacturing Research Institute (AMRI), AIST, Japan. Through the research attachment, Dr. Rosdi had an experienced and obtained indeed invaluable sharing knowledge among the Japanese scientists. Besides, Dr. Rosdi also participate and promote Malaysian medical product implant which is craniofacial parts in-line with techno fund project at the 40th World International Trade Fair with Congress World Forum for Medicine (MEDICA 2008) that held in the Dusseldorf, Germany on the 17th until 23rd November 2008.

Still in the research area, Dr. Rosdi also involved as the review panel for MOSTI’s Science Fund Application and Science Fund Progress evaluation. Besides publishing various technical papers in journals and proceedings, Dr. Rosdi is also has been appointed as an editor for American Institute of Physics Conference Proceedings under subseries of Materials Physics and Application, vol. 1217, ISBN:978-0-7354-0760-2. Last but not least, he is a registered life membership for Electron Microscopy of Malaysia.