

# Solvent Debinding of MIM Parts in a Polystyrene-Palm Oil Based Binder System

R. Asmawi<sup>1</sup>, M.H.I Ibrahim<sup>1</sup>, A. M. Amin<sup>1</sup>, N. Mustafa<sup>1</sup>

<sup>1</sup> Advanced Manufacturing and Materials Center (AMMC), Universiti Tun Hussein Onn Malaysia (UTHM), 86400 Parit Raja, Batu Pahat, Johor, Malaysia

E-mail: roslias@uthm.edu.my

**Abstract.** The influence of the leaching temperatures on the solvent debinding process of SS316L stainless steel alloy compact has been investigated. Solvent debinding process of injection moulded SS316L feedstock was conducted to eliminate palm kernel oil by using n-heptane solution at various temperatures of 40, 50 and 60°C and at different time duration up to 9 hours. The samples were prepared by injection molding a mixture of stainless steel SS316L alloy with waste polystyrene polymer and palm kernel oil at powder loading 60% vol. The weight loss percentages of palm kernel were calculated and the pore structure evolution was analyzed by scanning electron micrograph to observe the pores created after solvent extraction process. Results show that a complete diffusion of the palm kernel molecules out of the compact part best at 60°C within 6 hours of extraction time

## 1. Introduction

Metal injection Molding (MIM) is an advanced technology for the large scale production of high standard part, which combines the geometrical complexity of plastic injection molding with the powder metallurgy technique of sintering. In MIM, the process comprises of several processing stage which are mixing, injection molding, debinding and sintering [1]. The removal of organic binder is a procedure that is generally called as debinding. It is a critical process in obtaining a green part with good-desired shaped [2].

Solvent debinding process was introduced to the powder injection molding (PIM) industries during the 1980s [3]. During solvent debinding, one of the binder components is dissolved by immersing the compact parts into solution, e.g. water, hexane and heptanes, thus producing open pores. Yang et al [4] indicated that, solvent debinding process comprised of four stages. It is started with solvent molecules penetrate into the binder and producing swollen gels. The gels then gradually disintegrate into true solution. The solution then diffuses toward the surface, and finally the solution is removed from the specimen. The main purpose of this process is to extract soluble binder components by creating pores that allows the degraded products to diffuse to the surface easily. Therefore the main goal in debinding is to remove the binder in the shortest time with the minimum effect on the compact and it is inversely proportional with the thickness of the compact [5]. It is in agreement with a study done by Iriany et. al [6] which also found that the sample thickness and debinding temperature have significant impact on the debinding rate as it increases with decreasing sample thickness and increasing debinding temperature. Furthermore, White and German [7] found that a higher powder loading needs a longer solvent debinding time to remove the soluble binder. The long debinding time relates to the difficulties of diffusion through the smaller pores. On the other hand, a study by Tsai and Chern [8] found that by increasing solvent temperature will also result in greater binder removal rate.

In this study, injected SS316L feedstock that consists of waste polystyrene and Palm kernel oil (PKO) as the binder were used to investigate the solvent debinding behavior during removal of soluble binders via SEM observation to monitor the pore structure and distribution of the binder at



different temperature and extraction times. Palm oil based binder includes Palm Kernel and Palm Stearin. It is an alternative substitution with multi-components conventional binder system to overcome long debinding times drawbacks.

## 2. Experimental Procedures

### 2.1 Feedstock preparation

Water atomized stainless steel 316L powder having irregular shape was used in this study as the metal powder. Figure-1 confirms the irregular shape of the metal powder. The characteristics and chemical composition of the metal powder are shown in Table 1 and Table 2, while Table 3 shows the binder system properties. The powder was mixed with 60wt% of waste PS and 40wt% of palm kernel oil (PKO). The mixing process was done by using a rotary mixer (Brabender Plastograph EC) with rotational speed of 30 rpm. The mixing temperature was set at 190 °C, which is within the highest melting temperature (185.4 °C) and the lowest degradation temperature of the binder system (324.3 °C). The feedstock was then injected using Nissei NP7 Real Mini injection molding machine. The homogenous paste of the prepared feedstock was then granulized using a crusher to form a small granule as shown in Figure-2.

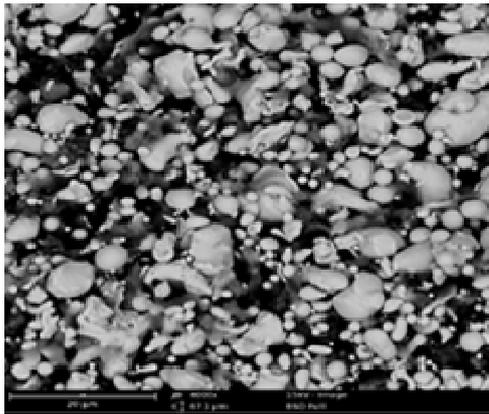


Figure 1. SEM Micrograph of SS316L at 4000x

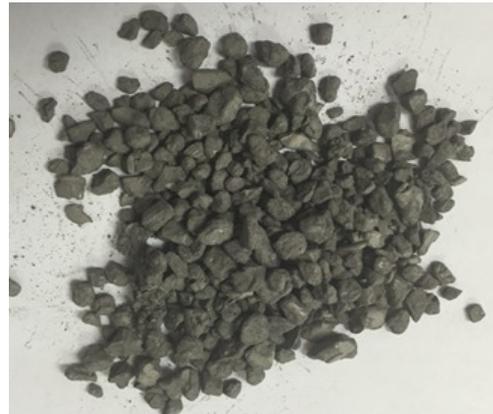


Figure 2. Granulized Feedstock

Table 1. Stainless Steel (316L) Powder Characteristics (Epson Atmix Japan)

Specification	Value
Density	8.0471 g/cm <sup>3</sup>
Particle Size	D <sub>10</sub> = 2.87μm D <sub>50</sub> = 5.96μm D <sub>90</sub> = 10.65μm

Table 2. Chemical Composition Stainless Steel 316L (Epson Atmix Japan)

Element	wt%
C	0.027
Si	0.84
Mn	0.19
P	0.016
S	0.012
Ni	12.20
Cr	16.40
Mo	2.10
Cu	0.03

Table 3. Binder Properties

Binder	Density (gcm <sup>-3</sup> )	Melting Temperature (°C)	Binder Ratio (weight %)
Waste PS	0.906	185.4	60
Palm kernel oil(PKO)	0.9087	30	40

### 3. Results and Discussion

#### 3.1 Injection Molding

The injection molding parameters were adjusted during injection process until the best condition was obtained. The parameters adjusted were injection pressure, speed, and temperature together with mold temperature. Lastly, after several tests, the feedstock was successfully injection molded at a temperature of 180 °C with the injection pressure of 85 MPa as in figure 3 below.



Figure-3. Injected Sample for Solvent Debinding

#### 3.2 Solvent Debinding Process

The palm kernel oil removal was studied as a function of time and temperature in heptane solution. The removal process have to consider the types of solvent used and the heating temperature of the stainless steel (SS316L) powder and the binders. Solvent debinding phase was conducted by immersing the injected part into beaker that is filled with heptane and heated with three various temperatures of 40, 50 and 60°C, because the PKO starts to soften at 30°C. The soften effect of PKO

is essential for the heptane solution to diffuse into the MIM compact. The specimens were then removed from the beaker and dried at 50°C for 1 hour to evaporate the solvent from the pores before weighing. The total weight of the compact part was typically around 9.76 g, containing approximately 8.34 g of Waste PS and 5.56 g of PKO. Since the weight fraction of the binder is 60/40 of PS/PKO, the mass loss percent of PS binder can be calculated with the following formula [1]

$$W_p = \frac{\rho_p \phi}{\rho_p \phi + \rho_B (1 - \phi)} \quad (1)$$

$$W_B = 1 - W_P \quad (2)$$

where  $\phi$  is the powder loading,  $\rho_p$  and  $\rho_B$  are the powder and binder theoretical density, respectively.  $W_p$  is the powder weight and  $W_B$  is the binder system weight.

During immersion, the PKO binder begins to dissolve in the heptane producing fine pore channels. The weight losses of the specimens were recorded at every hour until the total debinding time achieved 540 minutes (9 hours). Figure 4 shows the relation between PKO removed and various immersion times for different leaching temperatures during solvent debinding. The finding shows that during the first 2 hours the weight loss was dramatically changes proportional to time. It is because the diffusion distance for the heptanes and PKO is short in the early stage. As the time increased and debinding process continues, the weight loss of PKO also increased. In the meantime, pores channel enlarged and extend to the inner region of the compacts, thus slows down the debinding rate.

The results clearly demonstrate that at temperature 60 °C PKO binder had completely removed after 360 minutes, compared to 50 °C and 40 °C which each temperature has taken 420 minutes and 480 minutes respectively. Thus, it is considered that a complete diffusion of PKO molecules out of the compact part best at 60 °C within 6 hours of extraction time. The surfaces of the brown part were flat, indicating good shape retention during debinding and no visual defect was observed on the brown part. In order to observe the evolution of the pore structure during solvent extraction at different length of time, scanning electron micrographs were taken and then analyzed. Figure 5 (a-d) presents the cross section SEM observation of the brown part after leaching at 60 °C at various temperatures which shows open-pore channels evolution formed by the removal of PKO. Based on the SEM image, it is observed that the pore size increases with debinding time. This indicates the success of n-heptane as a solvent for debinding of the soluble binder (PKO).

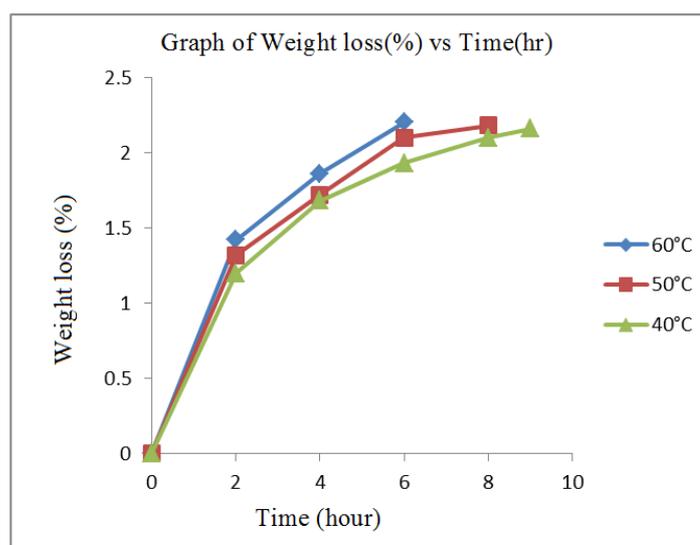


Figure 4. Percentage Of PKO Removal as a Function of Temperature, at Different Extraction Time

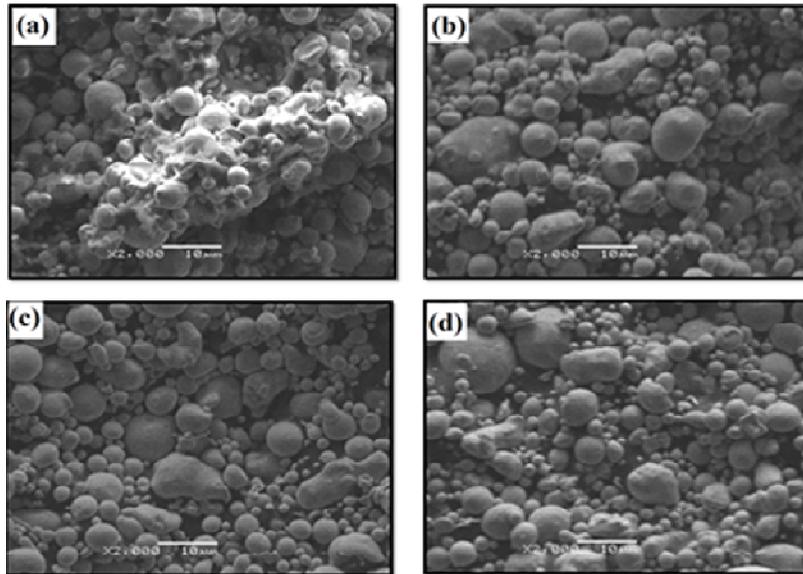


Figure 5. SEM Crosssection of Solvent Debound Part at at 2000x for Different Immersion Time; (a) Before Solvent, (b) 2 Hour Solvent, (c) 4 Hour Solvent, (d) 6 Hour Solvent

### Conclusion

The effects of 0 on the solvent debinding of palm kernel oil in stainless steel MIM compact parts were investigated. The study has shown clearly that leaching temperatures play an important role in debinding rate of PKO. Since the PKO is completely removed in solvent debinding, the backbone binder (waste PS) will be removed by the subsequent thermal debinding through the formation of open-pore channels created by the removal of soluble binder(PKO). SEM observations show no PKO binder and creation of a large amount of pores is observed after 6 hours immersion at 60 °C. Therefore this will facilitate the subsequent thermal debinding without endangering the integrity of the green body.

### References

- [1] R.M. German, and A. Bose: Injection Molding of Metals and Ceramics; Metal Powder Industry (MPIF),New Jersey (1997)
- [2] D. M. LIU and W. J . TSENG, *Ceram. Intern.* **24** (1998) 471
- [3] Lin, H. K. and Hwang, K.S. "In Situ Dimensional Changes of Powder Injection-Molded Compacts during Solvent Debinding", *Acta Metallurgica.*, Vol. 46, No.12, 1998, pp.4303-4309.
- [4] Yang, W. W., Yang, K. Y., Wang, M. C. and Hon M. H., "Solvent Debinding Mechanism for Alumina Injection Molded Compacts with Water-soluble Binders", *Ceramics International*, Vol. 29, 2003, pp.745-756.
- [5] R. M. GERMAN and A. BOSE, "Injection Molding of Metals and Ceramics" (Metal Powder Industries Federation, Princeton, New Jersey, 1997) p. 23, 33, 175, 197.
- [6] Iriany, Muhamad, N., Ariffin, A. K. and Sahari, J., "Effect of Temperature and Thickness on Solvent Debinding Rate of Metal Injection Molding Compacts", 2nd World Engineering Congress, 2002, pp. 233-236.
- [7] White, G. R.and German, R. M. "Dimensional Control of Powder Injection Molded 316L Stainless Steel using in-situ Molding Condition", *Metallurgy and Particulate Materials*, Vol. 5, 1993, pp. 121-132.
- [8] Tsai, D. S. and Chen, W.W. "Solvent Debinding Kinetics of Alumina Green Bodies by Powder Injection Molding", *Ceramics International*, Vol. 21, 1995, pp. 257-264.