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Solvent Debinding Process for ZK60 Magnesium Alloy Mim Compact

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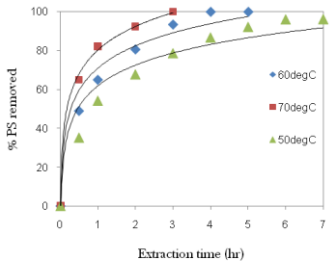
Graphical abstract



Abstract

The influence of the leaching temperatures on the solvent debinding process of ZK60 magnesium alloy compact has been investigated. In this experiment, the soluble binder, palm stearin molecules were extracted out from the compact parts by immersing the compact parts in an n-heptanes solution. The solvent debinding rate was then investigated under the conditions of different leaching temperatures. The weight loss percentages of palm stearin were calculated and the pore structure evolution was analyzed by scanning electron micrograph. Results show that a complete diffusion of the palm stearin molecules out of the compact part best at 60^{0}C within 4 hours of extraction time.

Keywords: Metal injection moulding; magnesium alloy; palm stearin; debinding rate

Abstrak

Penyelidikan pengaruh suhu rendaman terhadap proses penyahikatan pelarut bagi komponen jasad anum magnesium aloi ZK60 telah dijalankan. Di dalam ujikaji ini, molekul-molekul bahan pengikat boleh-larut lemak sawit dikeluarkan daripada komponen jasad anum dengan kaedah merendam komponen jasad anum tersebut di dalam larutan heptena. Kadar penyahikatan pelarut kemudiannya diselidiki di bawah keadaan beberapa suhu rendaman yang berbeza-beza. Peratus berat pengurangan lemak sawit dikira dan peningkatan struktur rongga dianalisa menggunakan mikroskop elekron imbasan. Keputusan menunjukkan molekul-molekul lemak sawit meresap keluar sepenuhnya daripada komponen jasad anum adalah optimum pada suhu 60^{0}C selama 4 jam masa rendaman.

Kata kunci: Acuan suntikan logam; magnesium aloi; lemak sawit; kadar penyahikatan

1.0 INTRODUCTION

Metal injection moulding (MIM) is a process for the production of near-net shape processing of wide range of material in mass quantities [1,2]. The process involves the mixing of metal powder with binders to form feedstock, the injection moulding of the feedstock to produce MIM compact, the elimination of binders through debinding to produce brown part and finally sintering to final part density, all of which are critically important to the overall success of the MIM process. In MIM process, common thermal debinding process is considered time consuming, whereby the debinding rate is often very slow and the debinding durations commonly took several hours. This technique was then combined with the solvent debinding technique in order to fasten the speed of debinding rate [3,4]. During solvent debinding, one of the binder components is dissolved by immersing the compact parts into solution, e.g. water, hexane and heptanes, thereby producing open pores. This step is then followed by a binder burnout, which are evaporated out of the surface through the open-pore channels.

The effectiveness of solvent debinding process commonly based on the optimal debinding rate without compromising the compact parts quality. Hwang *et al*. [5] investigates the debinding rate of solvent debinding for compact parts prepared with different particle sizes. The investigation concluded that particle size does not affect the debinding rate but it is determined by the cross-section thickness of the compact parts. Furthermore, they also concluded that with a high powder loading, the debinding rate decreases because the total porosity and flux area for the soluble binder component to diffuse through decreases. Krauss *et al*. [6] investigates the relation between PEG loss and immersion time in water for alumina ceramic injection moulding (CIM) of three different specimen thicknesses. They concluded that the thicker the sample, the longer the debinding time due to a longer path for diffusion. Jamaludin *et al*. [7] studied fine (11.225µm), coarse (19.606 µm) gas- and water-atomized powders SS316L,

and their effect on debinding time. Their work showed that the coarse powder feedstock debound faster than fine powder.

As for magnesium, processing them under hot working condition will create major drawbacks such as sublimation and formation of oxidation layer onto test sample especially under high temperature [8]. The sample oxidation is critically high risk and it has been a major challenging for the success of magnesium MIM. Therefore, processing under cold or warm working condition is considerable way for these specific debinding technologies. The investigation of pure magnesium MIM was first reported by Norbert & Martin [9] and they reported that the debinding process for magnesium MIM parts still has a plenty of problems i.e. oxidation layer occurred onto the part surface due to thermal effect during debinding process. This oxide layer will subsequently hamper the sintering activities as these two processes are thermally activated. To date, there is no significant report in academic literature on the debinding process for magnesium MIM and this has open great opportunities for MIM researcher to explore this area. In this work, binder system consisting of palm stearin and low density polyethylene were mixed with ZK60 magnesium alloy powder. Solvent debinding technique was deployed to remove the palm stearin in the MIM compact under cold working temperature. The objective of this paper is to provide the development of debinding method for magnesium MIM in order to enhance the removal of the binder component in the compact parts. In this investigation, solvent extraction method using heptanes solution was used whereby the immersion temperature and extraction time were analyzed for the determination of debinding rate.

2.0 EXPERIMENTAL PROCEDURES

The MIM process is performed following the diagram of Figure 1. In this experiment, the MIM feedstock was 64 vol.% of 45 µm of gas atomized spherical ZK60 magnesium alloy powder mixed with binder system of palm stearin (PS) and low density polyethylene (LDPE). The binder system composition is 60/40 wt. % of PS/PE, respectively. This feedstock displays the best combination of rheological properties based on previous study [10]. The characteristics of the powder and binder system are tabulated in Table 1 and 2. The MIM feedstocks are mixed by Sigma blade mixer at 150^0C for 1 hour. In injection moulding, the feedstocks are moulded into tensile shape as shown in Figure 2 using Battenfeld 250CDC injection moulding machine. During moulding experiments, the temperature and injection pressure were adjusted and the optimum conditions for green compact was obtained. The injection moulding parameters is best set at temperature of 180^0C and pressure 900kN [11]. The green compacts were then subjected to a solvent extraction where the soluble binder of PS is leach out by immersing the green compact in heptanes at leaching temperatures of 50, 60 and 70^0C until PS completely removed. The debinding rates of PS were calculated based on weight percentage removed and the evolution of pore structure was analyzed using SEM. Subsequently, the debound specimens were dried at 50^0C for about 1 hour to evaporate the solvent from the pores.

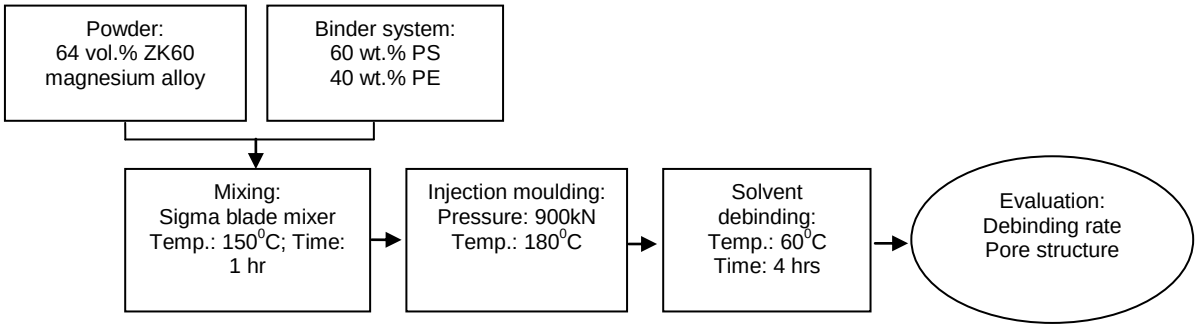


Figure 1 Diagram of the experimental procedures

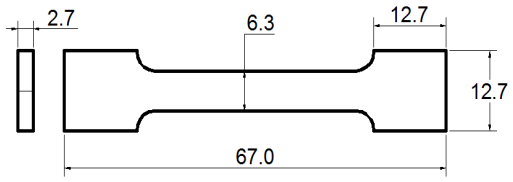


Figure 2 Injection mold part and geometry. All dimensions are in mm.

Table 1 Powder characteristics of ZK60

Particle size (μm)	Particle shape	Tap density (g/cm^3)	Pycnometer density (g/cm^3)	Width of particle size distribution, S_w*
D_{10}=24.63, D_{50}=43.03, D_{90}=67.75	Spherical	1.07	1.83	5.82

* $S_w = 2.56 / \log_{10} (D_{90} / D_{10})$

Table 2 Chemical composition of the binders

Component	T_m (^0C)	ρ (g/cm^3)	(TGA) Decompose temp.(^oC)
PS (palm stearin)	52	0.89	288-463
LDPE (low density polyethylene)	130	0.91	389-501

3.0 RESULTS AND DISCUSSION

3.1 Injection Moulding

During injection moulding process, the injection moulding parameters were adjusted until the optimal condition was obtained. It starts with low pressures, flow rates and temperatures until a complete tensile shape was accomplished. After several trials, the feedstock was considered successfully injection molded at a temperature of 180^0C with the injection pressure of 900kN. All the green compacts were quite good and free from normal defects such as short shot, flashes and binder separation. The fracture surface and outer surface of green parts were observed by SEM. Figure 3 (a-b) clearly shows that the binder fills practically all the interstitial spaces between the powder particles. It is also observed that binder content was pronounced at the outer surfaces than those in fracture regions. Many pores can be seen at outer surface due to the entrapped air or binder shrinkage during cooling process.

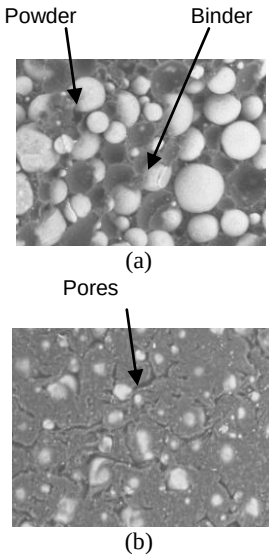


Figure 3 SEM observations on (a) fracture and (b) outer surface at 600X

3.2 Solvent Debinding

The PS removal was studied as a function of time and temperature in heptane solution. Experiments were performed at 50, 60 and 70^0C because the PS starts to soften at 50^0C [12]. The soften effect of PS is prerequisite for the heptanes solution to diffuse into the MIM compact. The binder removal was quantified by mass loss measurements. The total weight of the magnesium compact part was typically around 2.86 g, containing approximately 0.36 g of PS and 0.24 g of PE. Since the weight fraction of the binder is 60/40 of PS/PE, the mass loss percent of PS binder can be calculated with the following formula [13].

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

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

where φ is the powder loading, ρ_p and ρ_B are the powder and binder theoretical density, respectively. W_p is the powder weight and W_B is the binder system weight.

On immersion in heptane in the pyrex cylinder, the paraffin wax begins to dissolve in the heptane and fine pore channels begin to form. As the time increased, the weight loss of paraffin wax increased and the pore channels enlarged. Figure 4 shows the relation between PS removed and various immersion times for different leaching temperatures during solvent debinding. The finding was that all the given heptanes temperature influences the leaching rate of PS which is dramatically for the first 2 hours. This is because the diffusion distance for the heptanes and PS is short in the early stage. As the debinding process continues, the pore channels extend to the inner region of the compacts. Thus, the longer diffusion length slows down the debinding rate [6]. The results clearly demonstrate that the speed removal of the PS occurred at higher leaching temperature of 70^0C within 3 hours of leaching. However, the too fast removal of PS result the brown part to distort as shown in Figure 5. The distortion occurred due to the softening effect of PE binder. In fact, this much loss in binder over a short period of time can cause defects to form, such as blisters or fractures due to rapid release of the decomposition gas [13]. On the other hand, at leaching temperature of 50^0C, the PS was not completely removed even subjected to extraction time up to 7 hours. Considering that, at such temperature, the diffusion in the liquid phase does not reach the part surface. From these studies, it is considered that a complete diffusion of PS molecules out of the compact part best at 60^0C within 4 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. Thus, leaching temperature at 60^0C was suitable for solvent debinding of ZK60 magnesium alloy compact. The same result is also reported by Hafiez *et al.* [14] on solvent debinding of titanium alloy.

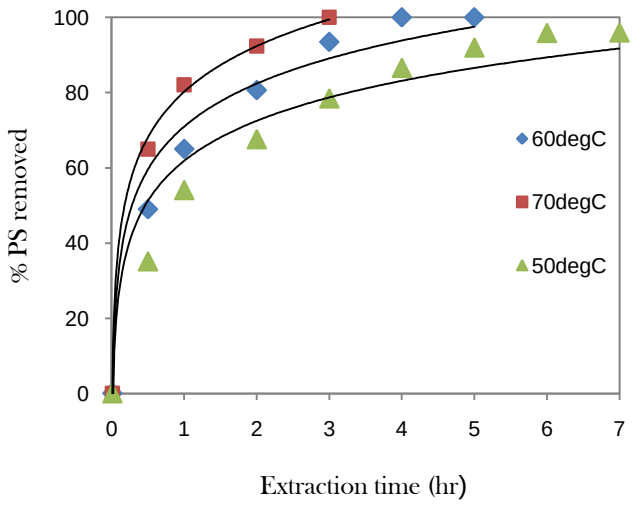


Figure 4 The % of PS removal as a function of temperature, for different extraction time

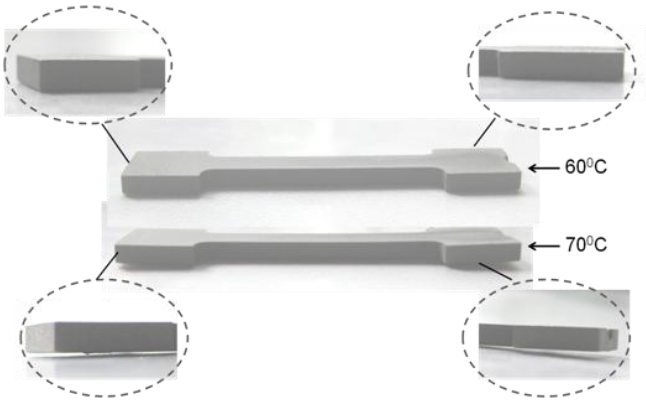


Figure 5 Brown part after solvent debinding at different leaching temperature

Figure 6 (a-b) presents the SEM of the brown part after leaching for 4 hours at 60^0C which shows open-pore channels evolution formed by the removal of PS. For fracture surface, after 30 minutes of leaching the pores with different sizes were formed, some were interparticle pores and some were within the binder. After 2 hours, large amount of PS leave interstices between the powder particles increases the pore size. The insoluble binder, i.e. PE remained as interconnected capillary porosity inside the brown part which provided sufficient strength to hold the powder particles together prior to sintering process. For the outer surface, irregular shaped holes appeared on the surface after leaching for 30 minutes. After 1 hour of solvent extraction, most of the binder was found at the outer edge of the part. Thus it shows that the binder dissolution is from the surface of the part. It is also notice that the depth of binder extraction and pore size increases with debinding time both for the fracture and outer surface. After 4 hours, ligaments of PE can be seen on the surface of the debound specimens.

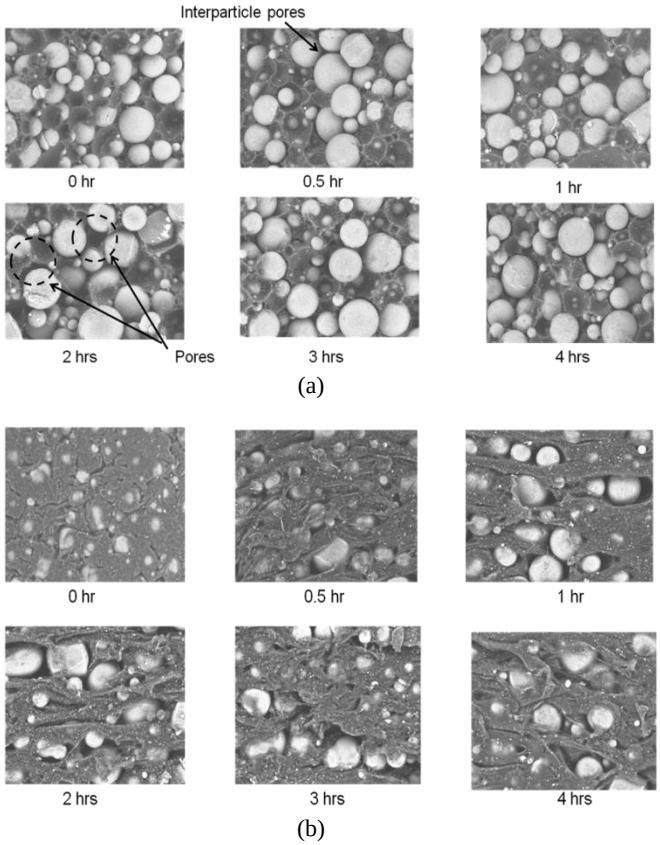


Figure 6 Open-pore evolution as a function of extraction time, leaching at 60^0C for (a) fracture surface and (b) outer surface

4.0 CONCLUSION

The effects of leaching temperatures on the solvent debinding of palm stearin in magnesium MIM compact parts were investigated. The study has shown clearly that leaching temperatures play an important role in debinding rate of PS. Even though increasing this parameter increases the debinding rate but it can also cause physical defects to the brown part. Since the PS is completely removed in solvent debinding, the PE will be removed by the subsequent thermal pyrolysis debinding through the formation of open-pore channels created by the removal of PS. Thus, it is clear that the solvent debinding of PS needs to be carefully optimised.

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