

Development of Waste Rubber as a Binder Component for Metal Injection Moulding Process

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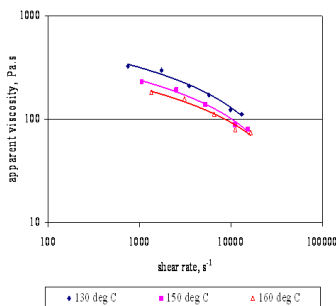
Article history

Received : 28 March 2012

Received in revised form : 26 June 2012

Accepted : 30 October 2012

Graphical abstract



Abstract

One of the keys to successful production of MIM is the selection of the binders. The binder-system provides the metal powder with the fluidity necessary for moulding. It also strongly influences the maximum solid fraction of the mixture that can be moulded, the green strength of the moulded part and, the properties of the final products after the debinding process. There are various binder-systems that have been developed for use in practice of the MIM. To overcome at least some of the problems associated with the other systems, a eco-friendly biopolymer composite binder based on natural sources and waste materials had been successfully developed at the Structural Materials Programme of SIRIM Berhad. This consists of natural sources based biopolymer constituents, particularly palm stearin and waste rubber as a back bone polymer. The new system has been successfully used for metal powders to produce precision metal component. The physical and mechanical properties of the sintered samples will be discussed. The highest sintered density achieved was 99.8% of theoretical density with the tensile strength of 517 MPa.

Keywords: Metal Injection Molding , debinding, sintering, palm stearin, waste rubber

Abstrak

Salah satu ciri kejayaan di dalam penghasilan produk MIM adalah pemilihan bahan pengikat. Bahan pengikat diperlukan bagi serbuk logam mengalir dalam proses acuan. Ianya memberi kesan terhadap pecahan pepejal maksima terhadap bahan suapan yang dapat di acukan, kekuatan bahan acuan serta pencirian kepada produk akhir selepas proses nyahikatan. Terdapat banyak bahan pengikat yang telah dimajukan untuk kegunaan MIM. Untuk mengatasi beberapa masalah yang terdapat pada bahan pengikat terdahulu, pihak SIRIM Berhad telah berjaya menghasilkan bahan pengikat yang terdiri daripada sumber semulajadi dan bahan buangan. Bahan pengikat ini terdiri daripada stearin sawit dan bahan buangan getah. Sistem bahan pengikat ini telah berjaya digunakan bagi penghasilan bahan suapan serbuk logam bagi pembuatan bahan logam. Densiti pangsinteran yang tertinggi dicapai adalah 99.8% daripada teori density dengan kekuatan regangan 517 MPa.

Kata kunci: acuan suntikan logam, penyahikatan, pangsinterang, stearin sawit, getah terbuang

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1.0 INTRODUCTION

Metal Injection Moulding (MIM) is increasingly being accepted as a suitable and cost effective method for the high volume production of small, complex-shaped and high performance parts. One of the keys to successful production of MIM is the selection of the binders. [1-4]. The binder-system provides the metal powder with the fluidity necessary for moulding. It also strongly influences the maximum solid fraction of the mixture that can be moulded, the green strength of the moulded part and, the properties of the final products after the debinding process. There are various binder-systems that have been developed for use in

practice of the MIM. However, there are problems associated in the use of said binder-systems. Among the problems include resulting of hazardous vapours, which can cause irritation to eyes and respiratory system and damage the environment, during mixing, moulding and/or debinding processes. Furthermore, some of the binder-systems produce low molded strength in the feedstock or causing long debinding process time and are also expensive to prepare.

To overcome at least some of the problems associated with the other systems, a eco-friendly biopolymer composite binder based on natural sources and waste materials had been successfully developed at the Structural Materials Programme of

SIRIM Berhad. This consists of natural sources based biopolymer constituents, particularly natural rubber, starch and palm stearin and waste rubber or waste plastic as a back bone polymer. The new system has been successfully used for metal powders to produce precision metal component. With properly controlled hydrolysis, these natural sources and recycle waste rubber (from rejected glove) also provide a spectrum of rheological processing conditions, suitable for the processing of metal powders. It is hope that this new binder system can be developed to replace the conventional binder system which mainly comprise of three to four components

2.0 EXPERIMENTAL METHOD

One formulations consists of 65 vol.% of 316L stainless steel powders were prepared. The metal powder was dry mixed with binder containing 35 vol.% which consists of 55% palm stearin,+ 35% (60% polyethylene+40% waste rubber)+10% stearic acid followed by the mixing in Z-blade mixer at temperature 170 °C for 90 min at speed 60 rpm. The metal mixtures were dried and granulated by using commercially available granulator. The feed stocks were characterized by using TGA and capillary rheometer CFT-500D. A vertical injection molder 100 KSA was used to produce tensile specimens according to MPIF-50 standard. The test samples were injected at 160°C and injection pressure 300 bar. Injection time was varied between 20-30 s. No cracks were observed on green parts.

The binder was removed from green parts in two steps (i) solvent extraction followed by (ii) thermal debinding. Solvent extraction process was carried out by immersing specimens in n-heptane at temperature 60°C for 5 hrs to ensure the complete removal of palm stearin followed by thermal debinding at 450°C at heating rate 3°C for 1 hr to remove the remaining binder.[5-6] The debound specimens were sintered in vacuum at 1300 to 1360°C for 2 hr. Two heating and cooling rates of 5°C/min and 10°C/min were used to study the effect of heating and cooling rate on the mechanical properties and the corrosion resistance of sintered test samples.

The sintered density of test samples was measured using water immersion technique and hardness was measured according to ASTM standard E140-02 at various locations on the surface of test samples by using BREVETTI ® hardness testing machine. The tensile testing was performed by using Amsler 100(Zwick/Roell) according to ASTM standard method E8M-00. The extensometer was used to measure the elongation. Test samples were observed under SEM to study the structure and porosity.

3.0 RESULTS & DISCUSSION

3.1 Rheological Behaviour

In the MIM process, the shear rate during moulding usually ranges between 100 and 10 000 s⁻¹. In this shear rate range, empirical studies have shown that the maximum viscosity for moulding is 1000 Pa.s at the moulding temperature [2]. The capillary rheometer has been utilized widely in order to characterize the rheological behavior of MIM feedstock [1,2,3]. The rheological results of the feedstock are shown in Fig. 1. The feedstock's viscosity decreases with increasing shear rate and this behavior is generally called pseudoplastic flow. Their viscosity is also observed to be temperature sensitive. As temperature increase, there is noticeable decrease in the feedstock's viscosity. This phenomena is mainly due to (a) a decrease in the powder

volume caused by the larger expansion of the binder when heat is introduced; and (b) disentanglement of the molecular chain when more heat is distributed to fluctuate the random molecular structure [2]. With a shear rate varying from 100 to 10 000s⁻¹, the viscosity of the feedstock falls below 1000 Pa s. Thus, the feedstock is suitable for injection moulding.

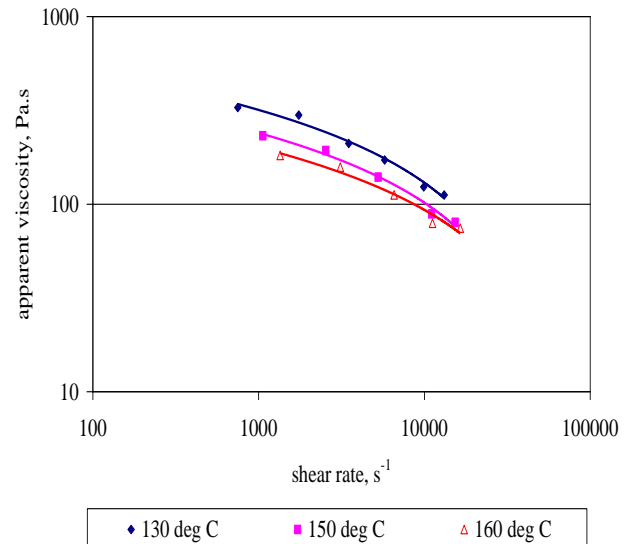


Figure 1 The rheological behavior of the feedstock

3.2 Moulding Behaviour

After several trials and error, the feedstock was successfully injection moulded at the nozzle temperature of 210°C and injection pressure of 30 MPa. Total cycle time for each injection was 6 seconds. Fig. 2 clearly shows the SEM of the green parts at two different regions; (a) fracture and (b) outer surface. It can be seen that, the binder fills practically all the interstitial spaces between the powder particles.

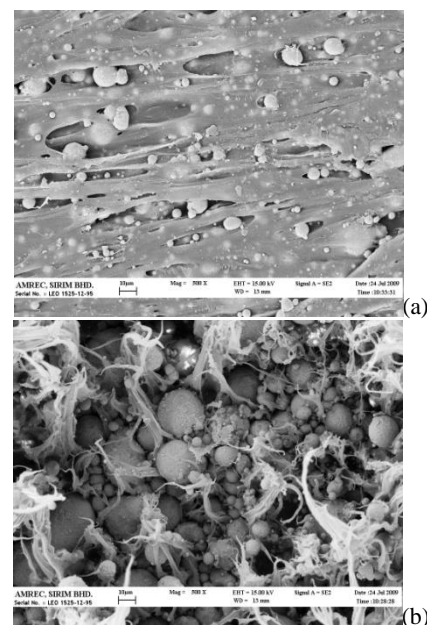
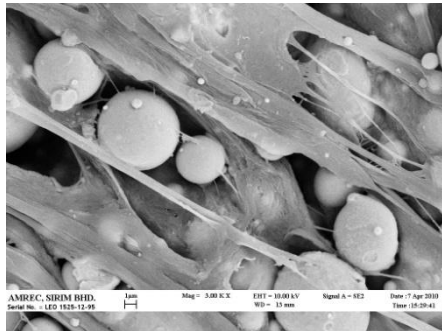


Figure 2 SEM of the as-moulded sample a) surface and b) fracture

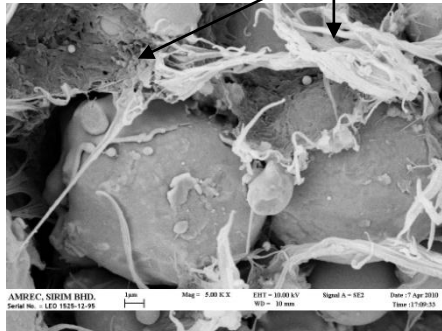
3.3 Debinding and Sintering Proses

For safe and rapid binder removal with minimum possibility of cracks and blister formation, solvent debinding followed by thermal debinding was used. The binder chosen includes the lower stability components of palm stearin which was removed in early stage of debinding. These generate pore channels inside of the part that allow gaseous product of degradation of remaining binder harmlessly diffuse out of the structure.



a)After 30 minutes

Mixture of poly ethylene and waste rubber ligaments



b)After 240 minutes

Figure 3 SEM of fracture surface after solvent extraction in heptane after 30 and 240 minutes

SEM of debound specimen with extracting time of 30 minutes (Fig. 3 (a)) showing that there are much amount of binder left interstices between the powder particles. Fig. 3(b) indicates all the palm stearin was removed after 240 minutes. It is clearly seen that a network of porous polyethylene ligaments and waste rubber remained, binding and holding the powder particles together to provide the moulding with sufficient brown strength to be handled. The remaining binder of waste rubber and poly ethylene has a function of holding particles together during and after extraction lowers stability components to maintain the part shape.

The following process of thermal debinding to remove the remaining binder; polyethylene has been done by placed the sample at furnace from room temperature to 450°C. The temperature increased with heating rate of 3°C/min and remained at 450°C for 30 min. Figure 4 shows scanning electron micrograph of a debinded specimen cross-section. As can be seen, nearly all the binder has been removed from the specimen.

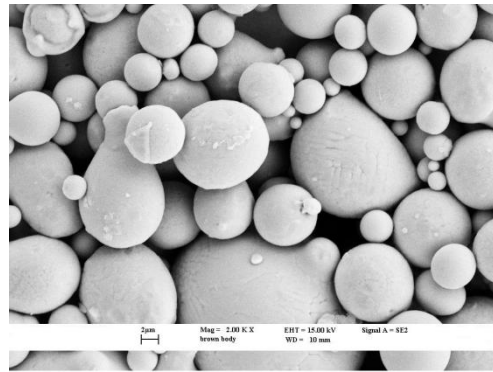
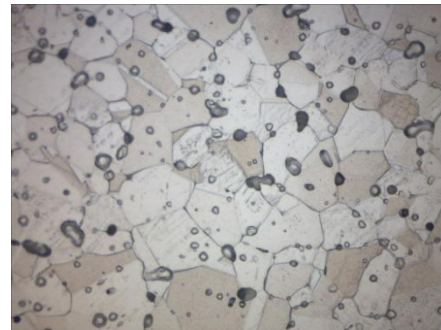
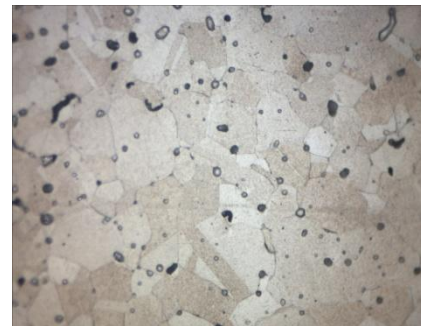


Figure 4 SEM observation of fracture surface after thermal pyrolysis process

No defects such as cracks, distortion which might affect the properties were observed in the sintered samples. Maximum density of 7.85 g/cm³ (99.4 %) was achieved from the specimen sintered at sintering temperature of 1360°C for 2 hours. Lower sintering temperatures result in microstructures with necking between the particles and open porous features. Fig. 5 a and b show the microstructures of etched samples sintered at 1300°C and 1360°C. These show how the substantial grain growth occurred at the highest sintering temperature.



(a)



(b)

Figure 5 show the microstructures of etched samples sintered at a)1300°C and b)1360°C.

Fig. 6 illustrates the effect of sintering temperature on the hardness and tensile strength. It is clearly shown that increasing the sintering temperature increased the hardness and tensile strength of the sintered specimen presumably due to better densification. The hardness increased from 148 Hv for the moulding sintered at 1300°C to 312 Hv for the moulding sintered at 1360°C.

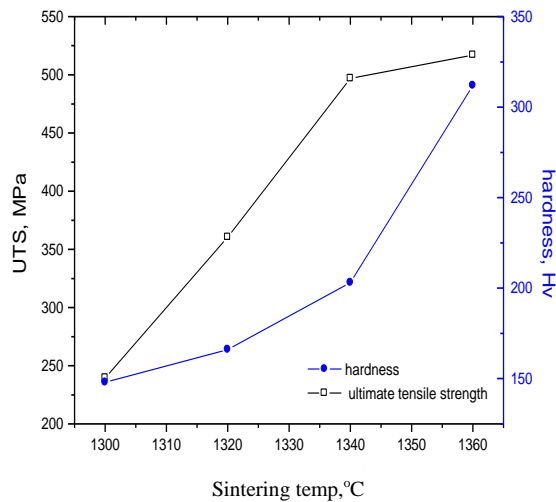


Figure 6 The effect of sintering temperature on hardness and tensile strength

As the sintering temperature increased, the tensile property improve, increasing at faster rate when the temperature changes from 1320 to 1360°C. For the ultimate tensile strength, the strength increased from 240 MPa to 517 MPa. The improvement in mechanical properties with increased sintering temperature is obviously attributed to increased density and pore roundness. The results achieved in this study are comparable to the wrought 316L SS (according to ASTM standard) [7] and a study using starch based binder system [8] and conventional binder [9].

4.0 CONCLUSION

From the study, it has been found possible to use the waste rubber as one of the component in binder system. The injection moulded of the test samples were successfully moulded using a temperature of 160°C with maximum injection pressure of 300 bar. The moulded samples were good and free from normal defects such as

short moulding, flashing and parting surfaces. The highest final sintered densities were about 99% of theoretical maximum value with good mechanical properties which comply to the standard. Sintering at 1360°C gave the highest mechanical and physical properties.

Acknowledgement

The authors wish to thank MOSTI for financial support under Techno Fund grant no. TF1208D168, Science Fund grant no. 03-03-02-SF0124 and SIRIM Bhd.

References

- [1] German, R. M & A. Bose, A. 1997. *Injection Molding of Metals and Ceramic*. Metal Powder Industries Federation, Princeton, New Jersey.
- [2] Huang, B., S. Liang and X. Qu. 2003. The Rheological of Metal Injection Moulding. *Journal of Material Processing Technology*. 137: 132–137.
- [3] Liu, Z. Y., N. H. Loh, S. B. Tor and K. A. Khor. 2003. Characterization of Powder Injection Moulding Feedstock. *Material Characterization*. 49: 313–320.
- [4] Omar, M. A., R. Ibrahim, M. I. Sidik, M. Mustaph and M. Mohamad. 2003. Rapid Debinding of 316L Stainless Steel Injection Moulding Component. *Journal of Materials Processing Technology*. 140: 397–400.
- [5] Subuki, I., M. A. Omar, M. H. Ismail and Z. Halim. 2007. Injection Moulding of 316L Stainless Steel Using Palm Stearin Based Binder. *Proc. Of AESEAP*. 27–30.
- [6] Omar, M. A. and I. Subuki. 2007. Rapid Debinding of 316L Stainless Steel Injection Moulded Component Using Palm Based Biopolymer Binder. *Proceedings of 3rd Colloquium on Materials, Minerals and Polymers*. 196–198.
- [7] Decker, B. S., J. D. Bolton, A. M. Eagles. 2000. Sintering of 316L Stainless Steel to High Density via the Addition of Chromium–Molybdenum Dibromide Powders, Part 1: Sintering Performance and Mechanical Properties. *Proc Instn Mech Engrs*. 214(1): 20.
- [8] Abolhasani, H. and N. Muhammad. 2010. A New Starch Based Binder for Metal Injection Moulding. *Journal of Materials Processing Technology*. 210(6–7): 961–968.
- [9] Razza, R. F., F. Ahmad, M. A. Omar and R. M. German. 2012. Effects of Colling Rate on mechanical and Corrosion Resistance of Vacuum sintered Powder Injection Moulding. *Journal of Materials Processing Technology*. 212(1): 164–170.