Journal of Applied Sciences

ISSN 1812-5654

science alert

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Journal of Applied Sciences 10 (24): 3295-3300, 2010
ISSN 1812-5654
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This article was presented in International Conference on Process Engineering and Advanced Materials 24th Symposium of Malaysian Chemical Engineers (15th-17th June, 2010)

Preparation and Characterization of Copper Feedstock for Metal Injection Molding

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Abstract: Powder loading is one of the most critical factors which have important influence on metal injection molding processes. In this study, four different loading feedstocks were prepared from gas atomized copper powder with wax-based binders. Mixes of four feedstocks with 2 Vol. % incremental powders loading from 55% to 61 Vol. % were carried out in a Z-blade mixer. The injection molding was carried out at low pressure. A combination of solvent and thermal debinding was used for binder removal from the samples and then the sintering process take place in argon gas at 900°C. It was observed that the feedstock containing 59 Vol. % of copper produce a free defect samples which was selected as the optimum feedstock.

Key words: MIM, compounding, feedstock, binder, copper powder, debinding, sintering, density, shrinkage

INTRODUCTION

Metal Injection Molding (MIM) is one of the most rapidly growing areas of powder metallurgy due to the fact that the parts produced by MIM can offer wrought properties, characteristics and microstructures. Due to the near fully dense fine-grained microstructures of MIM alloys, the resultant mechanical properties are typically equivalent to wrought properties and well in excess of those of castings and traditional PM (press and sinter) parts. In principle, injection molding is a relatively simple process. A feedstock is plasticized in a plasticizing unit, which is comprised of a heated until the injection temperature and then a controlled volume of the molten feedstock is injected from the plasticator under pressure into a closed mold, with solidification beginning on the mold's cavity wall (German and Bose, 1997; Matula *et al*., 2008; Rosato *et al*., 2000).

Although, MIM technique can be used for shaping various metal powders, its usage is usually limited to iron, steel and their alloy powders. This is due to the prevalent use in various applications such as producing automobile parts, office machinery and medical and dental instruments. Here, it is interesting to mention that there are no significant reports of using copper powder which is applicable in producing some special copper products such as heat sinks (Moballegh *et al*., 2005).

Generally, the powder injection molding is composed of four basic steps consisting of preparation of feedstock, injection molding, the debinding process and sintering process (Rosato *et al*., 2000; Ye *et al*., 2008). Figure 1 shows a schematic of the individual steps involved in MIM forming.

In general, the feedstock is a mixture of metal powder and binder system. However, the factors determining the attributes of feedstock are type of metal powder, particle

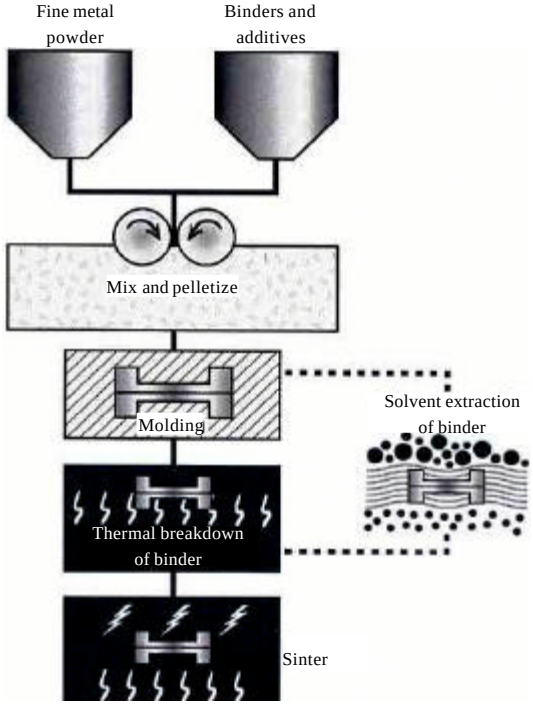

Fig. 1: Metal injection Process (http://www.remingtonpmpd.com/process/mim.asp)

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size and shape, binder composition, powder-binder ratio and mixing methods. The feedstock should be designed to be stable with time, easy to mold and have sufficient uniformity to provide dimensional control suitable for commercial application. A minimum of binder is needed to fill all voids between particles and to lubricate particles sliding during molding. The powder-binder ratio influences the viscosity of the feedstock (Loh *et al*., 2001; Zlatkov *et al*., 2008).

In molding step, conventional plastic injection molding equipment is used to mold the feedstock into desired shapes. The feedstock is injected into a mold under pressure, where it takes the shape of the mold and hardens through (German and Bose 1994, 1997; Ye *et al*., 2008).

In debinding, the binder must be removed from molded specimens. Different debinding methods have been developed for this purpose and can be classified into thermal and solvent debinding. Thermal debinding is the most commonly used method, which is often very slow and debinding durations of several days are not uncommon, while in solvent debinding, the binder is removed by dissolving one or more binder components with significantly faster speed (German and Bose, 1994, 1997; Xiang-quan *et al*., 2008).

Finally, sintering process provides strong bonds between powder particles and reduces the void spaces by means of densification, so high linear shrinkage of 10-20% takes place in the samples (German and Bose, 1997; Moballegh *et al*., 2005). The resulted density can reach over 97% of the theoretical value (Moballegh *et al*., 2005), depending on the powder properties like initial density and type of material and also sintering conditions such as atmosphere, temperature, heating rate and sintering time (German and Bose, 1994, 1997).

In this study, copper samples was produced by injection molding technique and investigated. A Paraffin wax based binder used due to the ease of processing and short debinding time. Different powder loading Feedstock were prepared and used for injection molding at low pressure. A combination of solvent and thermal debinding used for removing the binder from the samples and defect free samples produced after sintering process.

MATERIALS AND METHODS

Powder: A 99.95% pure spherical shape copper powder with particle size distribution of 4-22 μm, produced by gas atomization was supplied by Sandvik Malaysia SDN BHD Steel. A Scanning Electron Microscope (SEM) image of gas atomized Copper powder is shown in Fig. 2 reveals a spherical morphology and a broad particle size distribution which helps packing and densification in processing steps.

Table 1: Physical characterization of binder composition (Moballegh *et al*., 2005; Zlatkov *et al*., 2008)

Binder constituents	Density (g cm^{-3})	T_m (°C)
Paraffin wax	0.89-0.91	60-65
HDPE	0.954-0.957	130
Stearic acid	0.96	67-69

Table 2: Binder system formulation

	Weight %		
Binder component	Formula 1	Formula 2	Formula 3
Paraffin wax	55	65	70
HDPE	40	30	25
Stearic acid	5	5	5

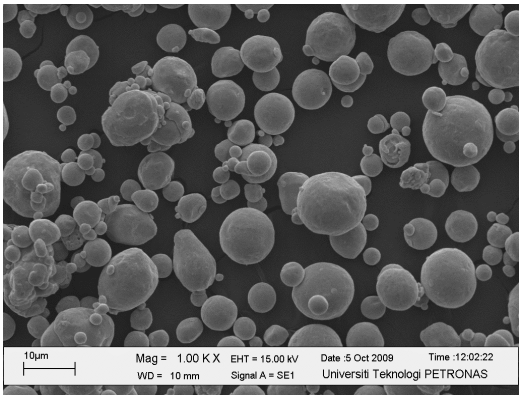

Fig. 2: SEM of copper powder

Binder system: The polymeric binder system selected for incorporation of the solids consisted of paraffin wax (112073, Merck) as a major component and high density polyethylene (Titan pet chem. (M) Sdn Bhd, Johor, Malaysia) as a minor component and stearic acid (Acros organics) as an aid flow; Table 1 shows the properties of these polymeric materials used as a binder in this research.

For the binder system used in this research, three formulations were prepared to study the rheological properties of this binder system as shown in Table 2.

Characterization of MIM feedstocks: Several equipments and processing methods have been used to characterize binders and feedstocks. The common equipment used in MIM characterization are capillary rheometer, torque rheometer, Differential Scanning Calorimetry (DSC), microscopy and Thermo Gravimetric Analysis (TGA). The processing techniques are usually only practical with the feedstock, e.g., injection molding and debinding.

Rheological characterization: A capillary rheometer (Shimodzu flow tester CFT-500D) with capillary diameter of 1 mm and L/D =10 used to measure the rheological properties, while the thermal degradation properties were determined by Perkin Elmer, thermal gravimetrical analyzer, under nitrogen atmosphere and heating rate of 10°C min^{-1}. However, a scanning electron microscope was used to observe the micrograph of samples and Archimedes method used to determine the density of sintered parts.

Experimental procedure: Four feedstocks were prepared using a Z-blade mixer. The mixing of the feedstock with 2 Vol.% incremental powder loading from 55% to 61 Vol. % done at mixing temperature of 140°C and speed of 50 rpm for 40 min. Vertical plunger injection machine (MCP-100 KSA) with dumbbell and strip shaped molds used for injection molding. The strip shaped mold dimensions were 60×15×3 mm. Debinding process was carried out in two stages. First, solvent debinding by means of Heptane as a solvent followed by thermal debinding in furnace under argon gas with flow rate of 0.9 L h^{-1} and then sintering was carried out in the same furnace under the same environment.

RESULTS AND DISCUSSION

Rheological properties: Minimizing sintering shrinkage and increasing dimensional accuracy requires maximum quantity of metal powder, while marinating sufficient binder to keep a good flow behavior. To maintain high viscosity and sensitivity to shear rate and temperature, which are preferable for injection molding, solid loading has to be increased. It is noted that the usual shear rate in mixing and injection molding should be in the range of 100-1000 sec^{-1}, while the viscosity of feedstock has to be lower than 1000 Pa.s, which in turns lead to lower temperature sensitivity (Moballegh *et al*., 2005).

The rheological properties of four feedstocks with powder loading of 55, 57, 59 and 61 Vol. % and the binder system formulations were studied at 160°C and die aspect ratio of L/D = 10. The comparison of viscosities of the feedstocks is shown in Fig. 3. This comparison shows that the viscosity increases as the powder loading increases at 100 (sec^{-1}) and 160°C. Here, the feedstock with 59 Vol. % powder loading, the viscosity approximately equals 500 Pa.s, which is suitable for injection molding. However, Fig. 4 shows that the binder system with 65 wt. % of PW, 30 wt. % HDPE and 5 wt. % SA is suitable to use in this research since its viscosity lies in the range of 10-20 Pa-s (German and Bose, 1997).

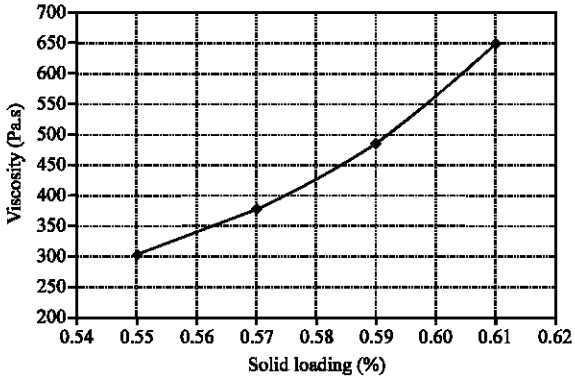

Fig. 3: Effect of powder loading on the viscosity

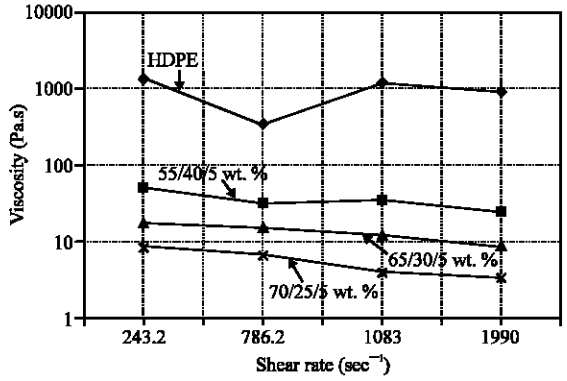

Fig. 4: Comparison between the different binder system formulations

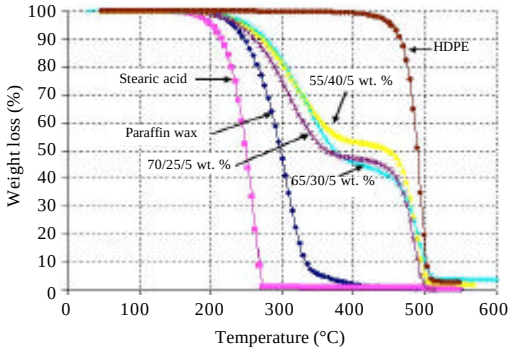

Fig. 5: TGA analysis for the binder system

Thermal degradation properties: The TGA curves can be used for establishing the upper limit of the melt temperature during injection molding and guidelines for a suitable debinding process.

Figure 5 shows the TGA curve of the binder system and components and indicates that the degradation starts at 170°C and ends at 500°C. The different binder degradation rates between the two temperatures are due

Table 3: Injection molding parameters

Feedstock (Vol. % Cu)	Injection temperature (°C)	Injection pressure (Bar)	Injection time (min)
55	160	4	0.15
57	160	4	0.15
59	160	4	0.15
61	160	4	0.30

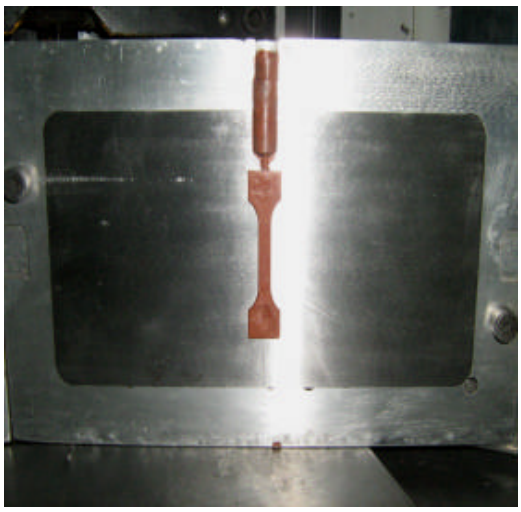

Fig. 6: The injection molding mold

to the use of a multi-component binder system. Thus, a multi-step debinding process where the binder components are removed progressively can be used, which helps to prevent defects and maintain the shape integrity of the microstructure.

The TGA results show that to avoid binder degradation, the processing temperatures such as mixing and injection molding should be lower than the start temperature of 170°C.

Injection molding: The four feedstocks formulations were injected at 160°C and 4 bars with the mold temperature 30-40°C and samples with dumbbell and strip were obtained as shown in Fig. 6. The injection molding was performed in a short time except the forth feedstock (61 Vol. % Cu) takes more time than the others as shown in Table 3. No sign of defects within the sample were observed after the injection molding process.

Debinding process: To minimize the possibility of defects with safe and fast binder removal, solvent debinding followed by thermal debinding was used. The multi-component binder chosen includes the lower stability components of paraffin wax and stearic acid, which are removed in early stage of debinding and generate pore channels. Inside of the part that allow gaseous products of degradation of remaining binder harmlessly diffuse out

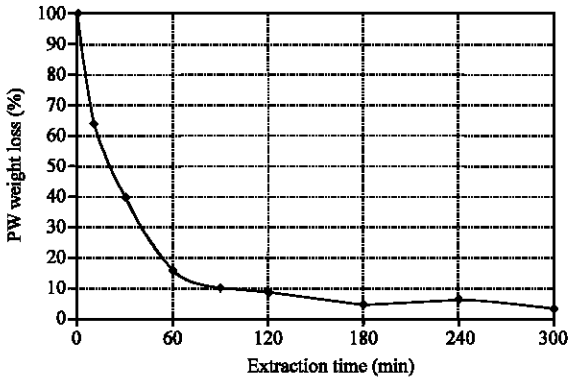

Fig. 7: Weight loss of PW and SA versus time

of the structure, while HDPE has a function of holding particles together during and after extracting lower stability components to maintain the part shape.

Solvent debinding was carried out by means of Heptane as a solvent at 60°C only for 45 min. Figure 7 shows the kinetics of the paraffin wax and Stearic acid weight loss versus time for the molded samples which indicates that after 45 min, 70% and after 5 h, 96% of paraffin wax and stearic acid can be removed.

It is considered that after removing 40% of binder, there exists some interconnected capillary porosity inside of samples which makes leaving of gaseous products in subsequent thermal debinding easy in short time (Moballegh *et al*., 2005; Li *et al*., 2003). Since nearly 70% of paraffin wax (Fig. 7) was removed in solvent debinding step, subsequent thermal debinding can be performed with higher speed in comparison with usual thermal debinding process.

Thermal debinding and sintering process: In this stage the thermal debinding and sintering process were carried out at the same time. To optimize the sintering heating rates and environments, different heating rates and environments were performed. Figure 8 shows the samples at different heating rates and sintering environments. As can be seen from Fig. 8, the feedstock of 59% Cu which is sintered at low heating rate (From room temperature to 450°C, the temperature was increased by 1°C min^{-1} and had been held at that temperature for 1 h, which removed the binder system used. From 450° to the sintering temperature (900°C) the temperature was increased by 3°C min^{-1} and had been held at this temperature for 1 h) in Argon gas environment produced a defect free sintered parts.

Density measurements: It was observed that with increasing the powder loading, density varies from 7.47 to

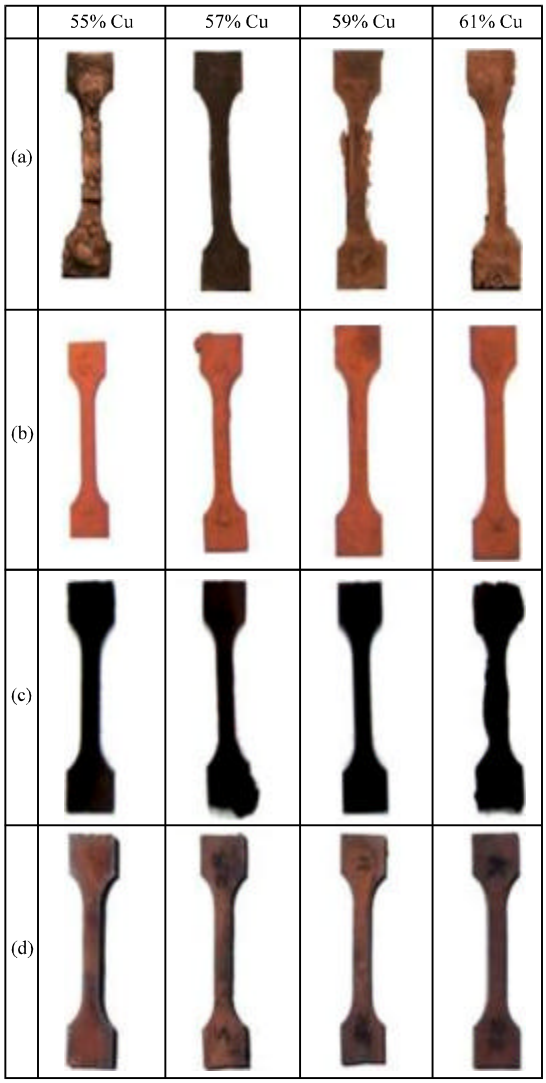

Fig. 8: Heating rates used during thermal debinding and sintering process, (a) 1°C min^{-1} until 450°C hold for 1 h at 450°C, 3°C min^{-1} from 450 to 900°C, hold for 1 h at 900°C, Cool down to room temperature in Vacuum, (b) 1°C min^{-1} until 450°C hold for 1 h at 450°C, 3°C min^{-1} from 450 to 900°C, hold for 1 h at 900°C, Cool down to room temperature in presence of Argon gas, (c) 3°C min^{-1} until 450°C hold for 1 h at 450°C, 5°C min^{-1} from 450 to 900°C, hold for 1 h at 900°C, Cool down to room temperature in presence of Argon gas and (d) 3°C min^{-1} until 100°C, 1°C min^{-1} from 100 to 250°C, Hold for 30 min at 250°C, 2°C min^{-1} from 250 to 500°C, Hold for 40 min at 500°C, 5°C min^{-1} from 500 to 900°C, Hold for 1 h at 900°C, Cool down to room temperature In the furnace in presence of Argon gas

Table 4: Dimensional shrinkage

Feedstock (Vol.% Cu)	Shrinkage (%)		
	Length	Width	Thickness
55	11.89	11.21	11.29
57	10.52	10.54	10.56
59	10.13	10.02	10.07
61	9.62	9.21	9.94

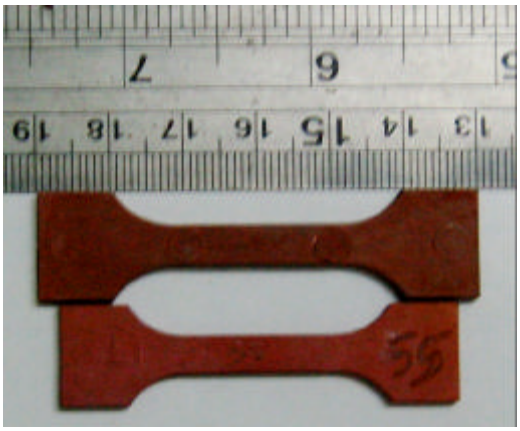

Fig. 9: Molded and sintered sample

7.87 g cm^{-3} which are nearly 84 to 88% of the theoretical density. However the technology has the ability to achieve over 97% of theoretical density, so some of the processing conditions should be optimized to achieve high density and good mechanical properties of sintered parts.

Shrinkage: The dimensions of the samples before and after sintering were measured and compared to study the shrinkage quality. Table 4 shows the percentage of shrinkage in three dimensions which indicates that the shrinkage is almost the same in three dimensions which means that the homogeneity of the feedstock is relatively good. Figure 9 shows the molded and sintered sample.

CONCLUSIONS

The preparation of different feedstocks with different solid loading was prepared and studied. The binder system with the formulation of 65% PW, 30% HDPE and 5% SA weight percentage was used based on the rheological properties. It found that the degradation temperature started at 170°C, which means that the processing temperature such as mixing and injection molding temperatures must be lower in order that binder degradation doesn't occur.

The injection molding done at low pressure and debinding and sintering of the molded sample by using solvent and thermal debinding, followed by sintering

under different heating rates and environment performed and the feedstock contains of 59 Vol.% Cu with the slower heating rate results a free defect sample.

The dimensional shrinkage of the sintered samples of different feedstocks prepared showed that the shrinkage is nearly the same in three dimensions.

The sintered samples had 84 to 88% of the theoretical density.

ACKNOWLEDGMENTS

The authors would like to thank the Universiti Teknologi PETRONAS for funding this research through Short Term Internal Research Fund (STIRF) scheme. The authors would also like to thank the Metal Injection Molding laboratory at the Advanced Materials Research Centre (AMREC) for their help and support.

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