Advanced Materials Research Vols. 314-316 (2011) pp 713-718
© (2011) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/AMR.314-316.713

Viscous Behaviours of Feedstocks for Micro MIM

Zhiqiang Cheng [1,a], Cedric Quinard [2,b], Xiangji Kong [2,c], Thierry Barriere [2,d], Baosheng Liu [1,e] and Jean Claude Gelin [2,f]

[1]School of Mechanics and Engineering, Southwest Jiaotong University, 610031 Chengdu, China.

[2] FEMTO-ST Institute / Applied Mechanics Department, 25000 Besançon, France.

[a] zqcheng@netease.com, [b] cquinard@gmail.com, [c] kongxiangji@live.fr, [d] tbarrier@univ-fcomte.fr, [e] bsliu@swjtu.edu.cn, [f] jean-claude.gelin@ens2m.fr

Keywords: Micro MIM, Binder composition, Viscous behaviour, Temperature, Powder loading in volume

Abstract. The viscous behavior of feedstocks plays the crucial role in Micro MIM. It affects directly injectability of the components and finally quality of the sintered component, because of the possible segregation induced by injection. The studies on viscosity of the feedstocks are realized by a series of the tests, in which a torque rheometer and a capillary rheometer are employed. The effects of binder composition, powder size, temperature and powder loading in volume on viscosity of the feedstocks are investigated. The mixtures of three kinds of binder composition, mixed with 5μm or 16μm 316L stainless steel powders, are evaluated. The best binder composition is determined by comparison of the viscous behaviors among the self-mixed feedstocks and the commercial one. It results in the suitable ranges of heating temperature and powder loading in volume for the feedstocks. The critical powder loading in volume is determined by a series of the capillary tests with the gradual increase of powder loading. These works provide the valuable reference for the research on binder composition and the process of micro metal injection molding.

Introduction

Manufacture of the micro metallic components is very important for MEMS industry [1]. The requirement for production by Micro MIM increases rapidly in the last few years, because of its high performance in manufacture of 3D intricate micro components [2]. Similar to the conventional MIM technologies, Micro MIM technique includes the same four principle stages: mixture of the metallic powder and thermoplastic binder to get the feedstocks [3], injection of the mixed feedstocks into the micro mold cavity [4], removal of the binder by thermal/catalytic or solvent debinding, and finally the solid state sintering to get the final products [5]. Preparation of feedstocks is the first stage of Micro MIM process. The feedstocks properties, such as fluidity and homogeneity, is the critical condition to ensure quality of the final products. It relates to possibility of the defects [6] such as segregation and jetting in injection stage, and distortion and warping in the later sintering stage. In the studies of viscous behavior of feedstocks for Micro MIM, Wang analyzed the rheological properties of the feedstocks (mixture of carbonyl iron powders and binder) based on a powder-binder two fluid model by numerical simulation [7]. Liu evaluated properties of the feedstocks for micro injection molding and its feasibility[8]. Cheng analyzed the viscous behavior of 316L stainless feedstocks by a series of the capillary dies [9]. In the present paper, the effects of binder composition, powder size, temperature and powder loading in volume on viscosity of the feedstocks are evaluated by a series of the tests. A torque rheometer and a capillary rheometer are employed. Three kinds of binder composition, for their mixture with 5μm and 16μm 316L stainless steel powder, are investigated.

The Characteristics of Powder and Binder

Properties of the powders

Table 1. Properties of 316L stainless steel powder

Average size	D_{10}, µm	D_{50}, µm	D_{90}, µm	Density, g/cm^3
16 µm	**4.1**	**10.5**	**21.9**	**7.9**
5 µm	**1.8**	**3.4**	**6.0**	

The 316L stainless steel powders in feedstock are provided by Osprey Company. In order to compare the effect of powder size on feedstock properties, two powders of average size 16 µm and 5 µm are used for feedstocks mixture. Distribution of the powder sizes is shown in Table 1. D_{10} is the hole size that 10% powder can pass through, the same are D_{50} and D_{90}. For reference, the feedstock produced by Advanced Metalworking Practises Company is mentioned. The average size of 316L powder in AMW feedstock is 16 µm. Density of the metallic powder material is the same, said 7.9 g.cm^{-3}.

Both 16µm and 5µm powders observed by SEM show that the particles are well in the spherical shapes. It is favorable to provide better fluidity of the feedstock.

Choice of the binders

The appropriate feedstocks in MIM and µMIM process should represent the fluidity for injection molding. The ability of shape keeping in debinding and sintering process is another necessary property. The homogeneity after injection is important to avoid the distortion and warping in sintering stage. Inhomogeneity in the injected components may caused by initial state of the feedstock, or by the effect of segregation in injection processes. Binder composition is the most important factor to qualify the feedstock. Binder is composed generally of the primary binder, the secondary binder and the surfactant. The primary binder, such as polypropylene or polyethylene, is used to keep the component in shape after injection molding and debinding. The secondary binder such as paraffin wax decreases viscosity of the feedstock and hence increases its injectability. The surfactants, such as stearic acid or oleic acid, play the role to make wetting the powder easily.

Three different binder compositions are shown in table 2, where PP represents polypropylene for short, PE is polyethylene, PW is paraffin wax, SA is stearic acid, and OA is oleic acid. The melting temperature and density of each composition in the binder are shown in table 3.

Table 2: Compositions of the binders

No.	*PP*, %	*PE*, %	*PW*, %	*SA*, %	*OA*, %
1	-	40	-	60	-
2	40	-	55	5	-
3	94	-	-	-	6

Table 3: Properties of binder compositions

Name	Type	Melting temperature ,°C	Density, g/cm^3
PE	Primary	130	0.91
PP	Primary	140	0.90
PW	Secondary	58-60	0.91
SA	Surfactant	70.1	0.94
OA	Surfactant	16.7	0.91

Tests of viscosity of feedstock

Effect of binder composition and powder size

Performance of the mixer is very important to prepare the homogenous mixture of powder and binder. In the present studies, the Plastograph Mixer EC W50EHT made by a Brabender company is employed for the mixing operations. This mixer represents the following characteristics: mixing temperature in the range from 20°C to 500°C, volume of the mixer bowl up to 55 cm^3, a pair of screws with spatial curved face, angular speed of the screws from 0 to 100 rpm, maximal torque 120 Nm. Moreover, the mixer can be applied as a torque rheometer for the tests of viscosity.

Three binders of different compositions shown in table 2 are mixed with the 16µm or 5 µm powders of 316L stainless steel respectively. The mixtures are made in a Brabender Plastograph Mixer with 30 rpm of the screw speed for more than 20 min. The mixed feedstock are numbered according to the type of binder composition. Each type of the binder is mixed with two 316L powders of size 16 µm or 5 µm respectively. So there are totally six kinds of the feedstock. The powder loading in volume of all the six feedstocks are 62 %. The temperature of mixing chamber is set initially at 160 °C. Torque of the screws should be the stable ones for good mixing quality. If the torque appears to be instable, the temperature should be increased to make the mixed materials more fluid. It happens a peak value for torque during starting of the mixer. Afterwards the mixing torque becomes smaller and stable. The peak values for starting are excluded from the records of mixing torques, as they represent only the transition to achieve the stable values. In the experiments, mixing torques of all the six feedstocks, as well as that of the 16 µm AMW commercial feedstock, are tested for 25 minutes. With the same angular speed, evolutions of the torques are shown in figure 1. It can be observed that the torque values depend mainly on composition of the binders, rather than particle size of the powders. Viscosity of the mixture measured by torque rheometer appears not sensitive to particle size of the powders. Among three binder compositions, the Feedstock No.2 represents the smallest mixing torque. Its torque values are even smaller than that of the 16µm commercial AMW feedstock. Then feedstock No.2 represents the lowest viscosity or the best fluidity. The feedstock No.3 should be heated to higher temperatures to get the relatively stable torques. The temperature values are respectively 193 °C or 183 °C for particle size 16µm and 5µm of the 316L powder, see table 4. The reason is that the binder of feedstock No.3 consists of 94% polypropylene in volume. The melting temperature of polypropylene is 140 °C, much higher than that of the other ingredients such as paraffin wax, stearic acid and oleic acid, see table 2 and table 3. So the feedstock No.3 needs higher temperature to melt quickly enough the 94% polypropylene in binder.

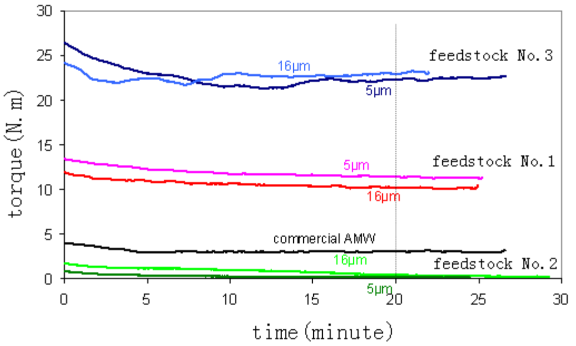

Fig. 1. Evolution of the Mixing torques for different feedstocks

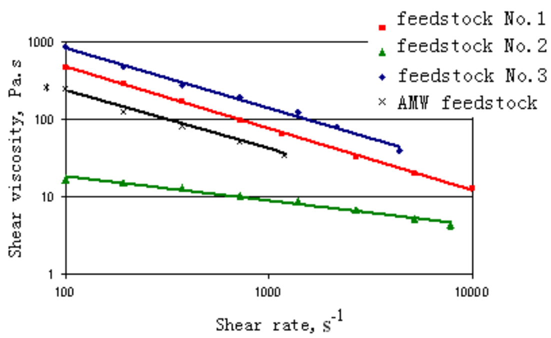

Fig. 2. Values of viscosity for 4 feedstocks of 16 µm powder at 160 °C

Table 4. Mixing torques of different feedstocks after 20 minute

Powder	binder	Temperature, °C	Torque, N.m
16μm AMW	Commercial	160	3.1
16μm 316L	No.1	165	10.2
	No.2	160	0.35
	No.3	193	22.9
5μm 316L	No.1	165	11.6
	No.2	160	0.15
	No.3	183	22.3

During injection moding, the molten feedstock subjects to the high shear rate. The capillary test is a suitable way to evaluate injectability of the feedstocks under high shear rate. A RH2000 capillary rheometer is employed for the tests of viscosity. A capillary die of 1 mm diameter and 16 mm length is used. The viscous behaviours of three feedstocks with different binder compositions, and that of the commercial AMW feedstock, are measured. These 4 feedstocks are selected to have the same 16μm powder size. Results of the measurement are shown in figure 2. In the same way, the viscous behaviours of three feedstocks with 5μm powder size are measure and compared, too. The results are shown in figure 3.

The measured values of viscosity are almost the same for the feedstocks of binder No.2, whatever the powder size is 16μm or 5μm. The measurement on feedstocks of binder No.2 represents the same phenomena. These results demonstrate that the values of viscosity for binder No.1 and No.2, measured by capillary rheometer, are not sensitive to the powder sizes. It should be mentioned that the same conclusion has been achieved when the tests are realized by a torque rheometer. However, such a conclusion is not appropriate for the feedstocks of binder No.3. For binder composition 3, the powder size represents the significant effect on fluidity of the feedstock. At shear rate $100s^{-1}$and temperature 160 °C, viscosity of the feedstock with binder No.3 is 800 Pa.s for 16μm powder, and 300 Pa.s for 5μm powder. The above mentioned results can be observed in figure 2 and figure 3.

It should be mentioned again that the tests by torque rheometer did not arrive to the same conclusion. In the tests by a torque rheometer, the values of viscosity appear to be not sensitive to powder size for the feedstocks of binder No.3. It indicates that the effect of powder size on fluidity of the feedstocks depends on the flow patterns. The results of capillary tests and mixing torque tests show the advantage of binder No.2. The feedstocks of binder No.2 represent better fluidity than the others. Among the feedstocks of same powder size, the feedstocks of binder No.2 represent even the fluidity better than that of the commercial AMW one. Viscous behaviour of the feedstocks of binder No.2 is quite stable with respect to the powder size and flowing pattern. The flow pattern depends on the mixing equipment and the details of injection molding. So the feedstocks of binder No.2 are proven to provide the proper and stable fluidity for injection.

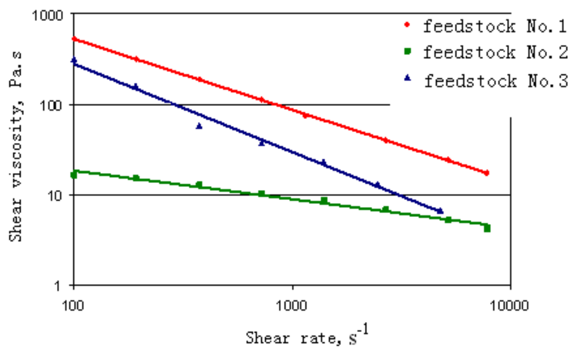

Fig. 3. Values of viscosity for 3 feedstocks of 5 μm powder at 160 °C

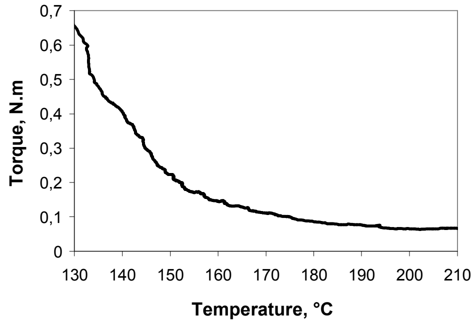

Fig. 4. Effect of temperature on mixing torque

Effect of temperature on viscosity

For the purpose of evaluating the thermal effect on viscosity of the feedstock, feedstock No.2 at 5 µm powder size is tested by Brabender Plastograph Mixer. The temperature varies from 130°C to 210°C at heating rate 1°C/min. For the feedstock to be kneaded in chamber of the mixer, the better fluidity and smaller mixing torque is desired. It is shown in Figure 4 that the mixing torque drops significantly when the temperature increases up to 160°C. When the temperature gets higher than 200°C, the mixing torque represents no more drops. So the proper temperature ranges from 160°C and 200°C, for lower viscosity of the feedstocks of binder No.2. As degradation of the binder ingredients must be considered, the temperature 160 °C is better for quality of the molded components.

Effect of powder loading in volume on viscosity

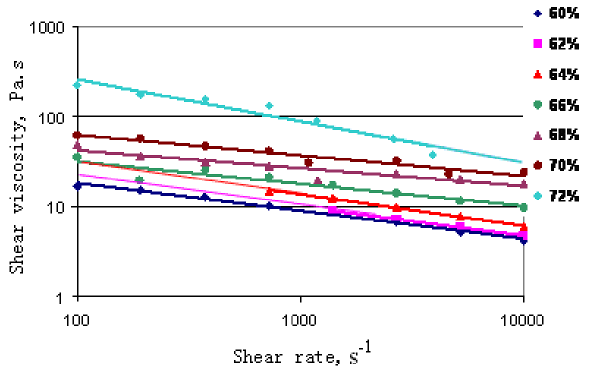

Fig. 5. Apparent viscosity under different powder volume loading at 160**°C**

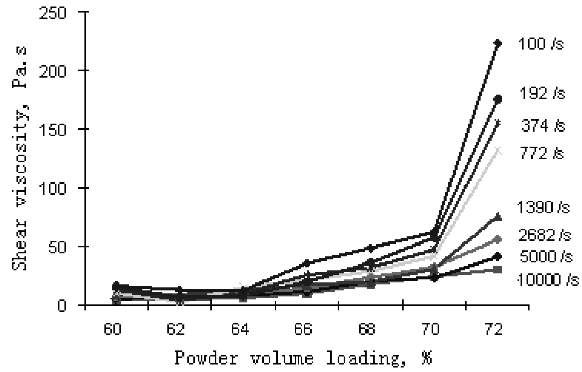

Fig.6. Effect of powder loading in volume on viscosity of the feedstock

Sintering of the components expects maximum quantity of the powder in the feedstock. But powder loading in the feedstocks is limited by the requirement of fluility for injection molding. So the effect of powder loading in volume on viscosity of the feedstocks should be investigated. The tests are realized by a RH2000 capillary rheometer. The feedstocks of binder No.2 and 5 µm powder are tested at temperature 160°C, for their powder loading increasing from 60% to 72% in volume. Based on the results in figure 5, the effect of powder loading on viscosity of the feedstocks at different shear rates can be shown in figure 6. Variation of the measured viscosity values with respect to powder loading can be divided into three obviously different stages: (1) when the powder loading increases from 60% to 64% in volume, the viscosity remain in the small values less than 15 Pa.s; (2) when it increases from 64% to 70%, the viscosity increases regularly in a small extent; (3) when it gets higher than 70% in volume, sharp increase in viscosity is observed. Then the critical powder loading is 70% in volume. The suitable powder loading ranges from 64% to 70% in volume. In injection process, the feedstock of binder No.2 behaves insensible to variation of the powder loading in volume. However, the critical powder loading is found 66% in volume in the previous studies by a torque rheometer [10]. It means that fluidity of the feedstocks is relevant to its flowing pattern, too. In fact, the injection molding represents the similar flowing pattern as that of the capillary tests. So the test by a capillary rheometer is well applicable for the injection process.

Conclusion

A series of the tests are realized by Brabender Plastograph Mixer and RH2000 capillary rheometer. The feedstocks are mixed by three kinds of the binders and two powders of 5µm and 16µm in particle sizes. The powders are both the ones of 316L stainless steel. To make a reference, the commercial AMW feedstock of powder size 16µm is tested, too. For the feedstocks viscosity, the effects of binder composition, powder size, temperature and powder loading in volume are investigated. Among all the samples, the feedstocks of binder No.2 represent the best performance in fluidity. The composition of binder No.2 is the best choice to produce the feedstocks for Micro MIM.

Based on the choice of binder composition, the appropriate ranges of temperature and powder loading in volume are determined for the feedstock No.2 of 5μm powder. The critical powder loading in volume is determined by measurements of the viscosity with successive increase of the powder loading in mixture. The present work provides a valuable reference for the researches in micro metal injection molding and its feedstock.

Acknowledgements

The work is supported by the Fundamental Research Funds for the Central Universities (SWJTU09CX67).

References

[1] R. Zauner, Micro powder injection moulding, *Microelectron. Eng.*, Vol. 83(2006), p. 1442-1444.
[2] R.M. German, Divergences in global powder injection moulding, *Powder Injection Moulding*, Vol. 2, No. 1, 2008.
[3] C. Quinard, T. Barriere, J.C. Gelin, , *Powder Technology*, Vol. 190(2009), p.123-128.
[4] Cheng zhiqiang, Barriere T, Liu Baosheng, Gelin J C. *Journal of Southwest Jiaotong University*, Vol.44(2010),p.635-638. (In Chinese)
[5] SONG J， BARRIERE T， LIU B，GELIN J C. *Chinese Journal of Mechanical Engineering*, Vol.44(2008),p.157-163. (In Chinese)
[6] LU Z，ZHANG K F. *Chinese Journal of Mechanical Engineering*, Vol.45(2009),p.295-299.
[7] Q. Wang, H. Yin, X. Qu and J. L. Johnson, *Powder Technology* ,Vol.193(2009), p.15-19.
[8] L. Liu, N.H. Loh, B.Y. Tay, S.B. Tor, Y. Murakoshi and R. Maeda, *Materials Characterization*, Vol. 54(2005), p. 230-238.
[9] CHENG Zhiqiang, QUINARD C, BARRIERE T, LIU Baosheng, GELIN J C. *Journal of Mechanical Engineering*, Vol. 46(2010), p.43-47. (In Chinese)
[10] C. Quinard, J. Song, T. Barriere, J.C. Gelin. , *Powder Technology*, Vol.208(2011), p.383-389.