DOI: https://doi.org/10.24425/amm.2022.141084

JIN MAN JANG[1,2*]

EVALUATION OF REPETITIVELY USED FEEDSTOCK FOR POWDER INJECTION MOLDING

This study investigates the effects of repetitive injection molding on the properties of feedstock using the AISI 4140 feedstock. The properties of feedstock are evaluated from the mixing homogeneity of powder and binder, rheological properties, and dimensional accuracy of parts sintered. The feedstock after the 1st injection molding shows a better homogeneity than as-received feedstock due to re-mixing effects between the screw and barrel during the injection molding process. As the number of recycling numbers increases, the homogeneity, viscosities ad shrinkage ratio of recycled feedstocks show slight differences with those of the as-received feedstock until the 6th molding injection. However, some rheological parameters like the moldability index sharply increased up to the 4th injection but shows a tendency to decrease thereafter.

Keywords: powder injection molding; recycling; rheology; homogeneity; feedstock

1. Introduction

Powder injection molding (PIM) process combines the traditional shape-making capability of plastic injection molding with the material flexibility of powder metallurgy. The process, in general, follows four steps: (1) feedstock preparation by mixing metal powders with organic binders, (2) injection molding to shape components, (3) debinding process to remove the organic binders, and (4) sintering process to consolidate the debounded components in the desired dimensional properties and density [1-6].

Nevertheless, the PIM process has a problem originating from several parts such as runners, sprues, and gates, which are essential parts of PIM. These parts are only required in the injection molding process so that they are treated as waste materials, resulting in the increase of manufacturing costs. Thus, the unused parts are often recycled so as to lower the manufacturing costs. However, the effect of recycling is still unclear while several researchers have reported the effect of recycled feedstock on molding and dimensional accuracy [7,8]. Jang. et al recently suggested the methods to evaluate feedstocks used in a PIM process without carrying out the entire process, from injection to sintering [9]. From this work, the evaluation parameters are the function of homogeneity and rheological property, which are respectively measured by pycnometer and TGA and capillary rheometer.

In this study, we characterize the properties of recycled feedstocks as the number of recycling increases. In addition the effects of recycled numbers on the injection molding and the sintering behavior are discussed in terms of the feedstock's rheological properties and dimensional accuracy.

2. Experimental

The feedstock used in this study was prepared by mixing a polymer binder, wax, and an AISI4140 powder. For the PIM process, the injection molding temperature was determined to be 180°C based on the thermal analysis results and each feedstock was injected into a rectangular mold cavity ($30 \times 10 \times 7$ mm^3).

We measured the variations of weight during heating and the changes in density to evaluate the homogeneity of as-received and recycled feedstocks. The indices for the degree of mixing homogeneity are based on the standard deviation of the densities and contents of the binder of samples taken from the mixture. A Hg-displacement method using a pycnometer was carried out for density measurement. For the weight change as a function of temperature, the TGA experiments were performed to evaluate the feedstock's macroscopic homogeneity. We also estimated the degree of homogeneity by observing the distribution of powder and binder in the feedstock from the recorded microstructures using SEM.

1 KOREA INSTITUTE OF INDUSTRIAL TECHNOLOGY, INCHEON, 21999, REPUBLIC OF KOREA
2 INHA UNIVERSITY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING, INCHEON, 22212, REPUBLIC OF KOREA

* Corresponding author: man4502@kitech.re.kr

The rheological behavior of PIM feedstocks was investigated using a capillary rheometer. The test temperatures are chosen 170°C, 180°C, and 190°C by thermal analysis results, and shear rate are chosen in the 100 to the 3,000s^{-1} range.

After injection, green parts were debound to remove the used polymer binders. The debound brown parts were sintered at 1360°C for 3h. The changes in shape and dimensions were investigated with a precision measuring microscope. The degree of dimensional precision was evaluated from at least five samples based on the standard deviations.

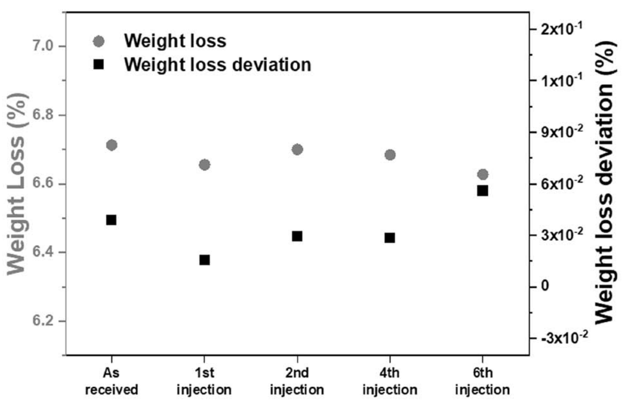
3. Results

The homogeneities of the feedstocks were examined by the binder burnout test, density measurements, and observations of the feedstock's microstructure. Fig. 1(a) shows the thermogravimetric results of as-received and recycled feedstocks. The weight loss (%) and standard deviation of the recycled feedstock have little differences with those of as-received feedstock until 4th injection. Then, the weight loss decreases and the standard deviation increases until 6th injection. This indicates that the recycled feedstocks have similar homogeneity until the 4th injection, while the homogeneity of feedstock decreases for every subsequent injection due to binder degradation.

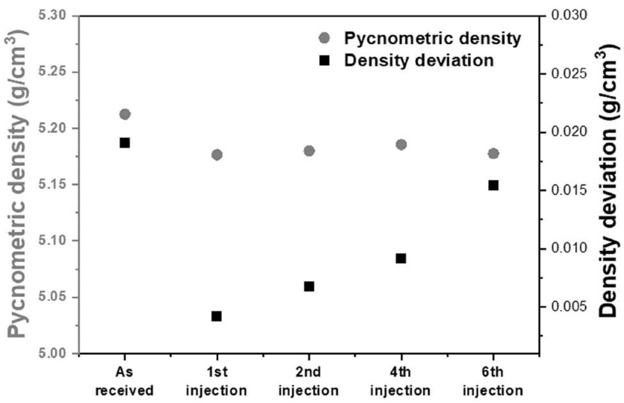
Fig. 1(b) shows the densities and the standard deviations measured from each feedstock. Unlike the TGA results, the densities and standard deviations of the recycled feedstock after the 1st injection are smaller than those of the as-received feedstock. This means that the metal powders and polymer binders of as-received feedstock are not fully mixed to achieve enough homogeneity due to the short kneading period. And it is considered that the homogeneity after 1st injection is secured through the remixing effect by the shear force between the screw and the barrel. After the 1st injection, the density of the feedstock does not show a significant change, while the standard deviation shows a tendency to decrease until the 4th injection and then to increase again. This shows that the homogeneity of feedstock is similar from the 1st to 4th injection, but after that, the homogeneity decreases until the 6th injection. The homogeneity was also measured by observing the powder and binder distribution in the feedstock. Fig. 1(c) shows the representative SEM fractographs of the feedstocks used in this study. The microstructure shows that the particles of powder are homogeneously dispersed are surrounded by the polymer binder in all feedstocks.

The rheological behavior of feedstocks is critical for the injection-molding process, as it influences the steady flow into, and the uniform filling of, the mold. The rheological properties are evaluated based on the viscosity, shear sensitivity, and temperature sensitivity [10]. Fig. 2(a) shows the viscosities of as-received and recycled feedstocks as a function of the shear rate.

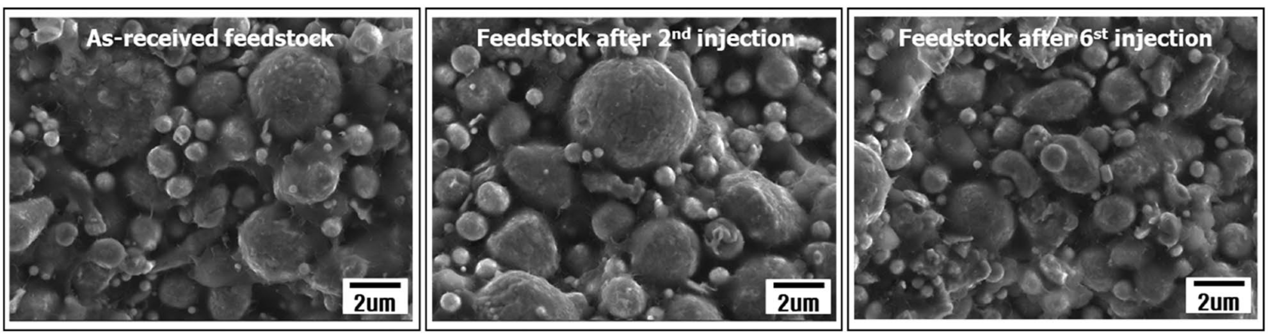
All tested feedstocks show similar viscosity and typical rheological behavior of PIM feedstocks regardless of recycling



(a) Weight loss and its standard deviation

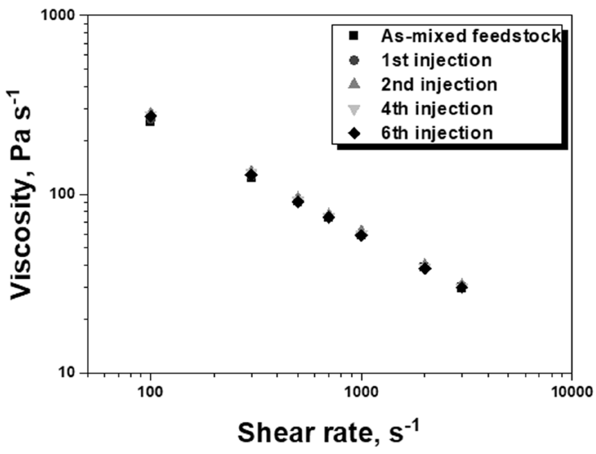


(b) Pycnometric density and its standard deviation

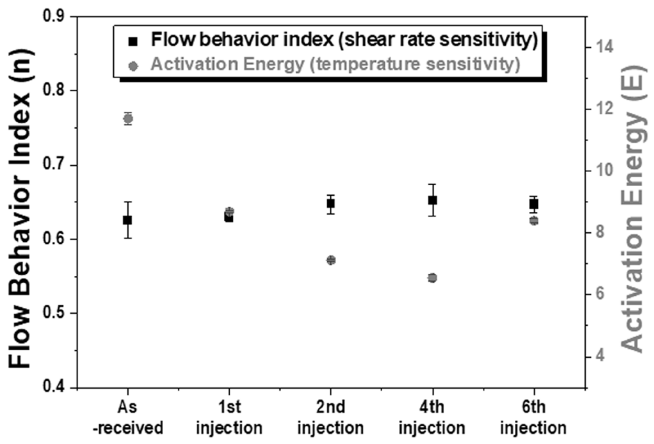


(c) Representative fractured surfaces of each feedstock

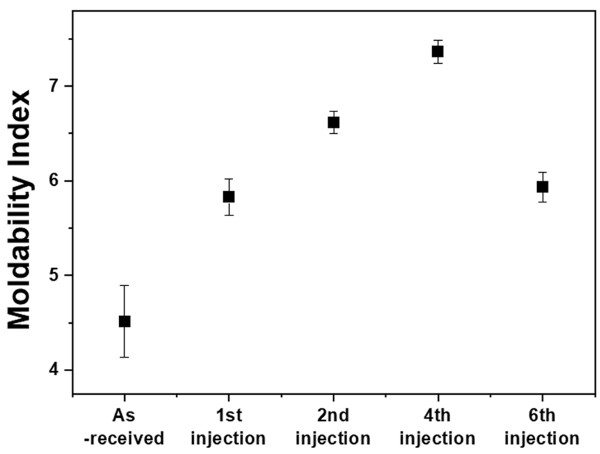
Fig. 1. Evaluation of homogeneities for as-received and recycled feedstocks



(a) Viscosity curves with shear rate



(b) Flow behavior index and activation energy



(c) Moldability index with numbers of recycling

Fig. 2. Rheological analysis results of as-received and recycled feedstocks

number. This indicates a pseudo-plastic flow behavior. A pseudo-plastic fluid can be expressed by

$$\eta = K\gamma^{n-1}$$

where η is the viscosity, γ is the shear rate, K is a constant, and n is a flow behavior exponent [11]. The value of n indicates the degree of shear sensitivity; the lower the n value, the more quickly the viscosity changes with increasing shear rate. The values of n were determined from the slope of logarithmic plots of the feedstock's viscosity curves. From the result, the n values of most are less than 1 so that the tested feedstocks exhibit pseudo-plastic behavior and show a high flowability.

The viscosity of feedstock exponentially depends on the absolute temperature, T,

$$\eta = \eta_0 \exp(E/RT)$$

where η_0 is the reference viscosity, E is the flow activation energy, R is the gas constant, and T is the absolute temperature [12]. The value of E indicates the degree of temperature sensitivity. If the value of E is low, the viscosity is not sensitive to temperature variations. That is, small fluctuations in temperature have not resulted in an abrupt viscosity change. The values of E were calculated from the slope of the logarithmic plots of the feedstock's viscosity curves against the temperature reciprocal. The flow activation energy decreases as the number of injections increases from the as-received feedstock up to the 4th injected feedstock. The recycled feedstock has the lowest E value of 6.55 kJ/mol for the 4th injection-molded feedstock. Then, E increases as the number of injection increases from four times to six times. Therefore, when the number of injections increases up to a 4th injection the repetitive injection is beneficial for the feedstock's rheological properties in terms of temperature sensitivity, differently to the effect of recycling on the flow exponent. This means that after a 4th injection the feedstock could be injection molded using a relatively wide temperature range.

The moldability index (α), which takes into account the effects of viscosity, shear rate, and temperature [13], is typically used to express the general rheological properties of polymers or PIM feedstocks, The moldability is calculated using the equation,

$$\alpha = |n - 1|/\eta_0 \cdot (R/E)$$

where n is the flow behavior index, η_0 is the viscosity at a reference temperature, R is the gas constant, and E is the flow activation energy. Using the above equation, the α of as-received and recycled-feedstocks is calculated for the reference temperature of 180°C and the reference shear rate of $1000s^{-1}$ (Fig. 2(c)). The α increases as the number of injections increased from the as-received feedstock to the 4^{th} injected feedstock. Then, the value decreases as the number of injections increases from four times to six times. These results indicate that the repetitive injection molding enhances the rheological properties of feedstock until the 4^{th} injection in this study.

The dimensional properties of PIM parts are a critical issue in PIM technology. Moreover, the dimensional accuracy mainly depends on the sintering process because of the large shrinkage experienced by the molded part during sintering [14,15]. Fig. 3 shows the average linear shrinkage and dimensional accuracy of feedstocks calculated from a different number of recycling. The results show that the average shrinkage ratio and the dimensional accuracy are almost constant. The densities of sintered samples were measured to be 7.39 ± 0.015 g/cm^3. It is of interest that the sintered densities of all samples are almost constant, although the green density is noticeably different as shown in Fig. 1(b). As the number of injections increases, the powder packing ratio changes resulting in a green part density difference, but the similar density is considered to be due to the same binder volume ratio.

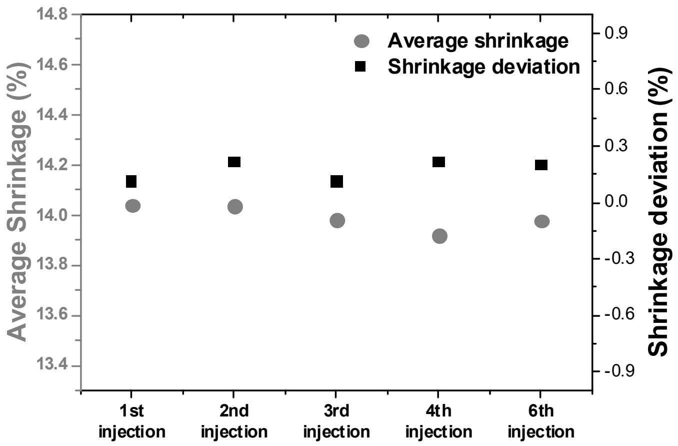


Fig. 3. Shrinkage ratio and dimensional accuracy of samples sintered at 1360°C under a 760 Torr Ar atmosphere

4. Discussion

Fig. 4 shows the degree of mixing between metal powders and polymer binders for three different conditions [16]. First, critical solid loading indicates the dense packing of powder particles (Fig. 4(b)). The feedstock, the solid loading is higher than the critical solid loading, has a high viscosity, and is difficult to mold (Fig. 4(c)). On the other hand, the polymer binder is too enough if the solid loading is lower than the critical loading volume (Fig. 4(a)). Generally, because feedstock with critical solid loading is hard to use, the binder volume in the optimal powder volume is 2-5% higher than that of the critical volume. Therefore, powder clusters of relatively low packing ratio remain in the PIM feedstock, but the packing ratio of those clusters improves by the shear, compressive stress, heat, etc. during the repetitive injection molding process. This has the same effect as increasing the solid loading. In other words, the higher packing of the mixture allows more binder to act as a lubricant when the packing ratio of the powder clusters is improved at a constant solid loading.

It is well known that the moldability of recycled feedstock decreases because of the deterioration of the binder component [7]. The above results (Fig. 2(c)), however, show that moldability is improved until the 4^{th} injection. From these results, it could be carefully suggested that the cause for the alteration of the rheological properties of the feedstock that occurs due to recycling of the feedstock is explained by the improvement in the packing ratio of the powder particles resulting from shear and compressive stresses generated during the injection process step. However, it should be noted that these recycling numbers could be different by the size and shape of parts, which determines the mold design and thus the molding parameters, such as pressure and temperature.

5. Conclusions

In this study, the effects of recycling numbers on the properties of feedstock were investigated by measuring the mixing

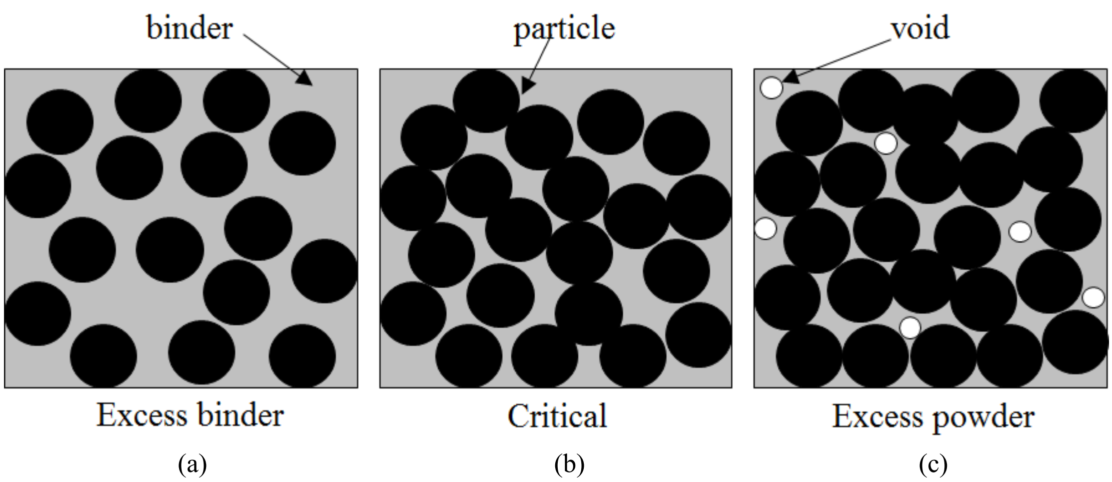


Fig. 4. Mixing degree of the powder-binder mixture when (a) there is an excess of binder, (b) the solid loading is at its critical value, and (c) there in an excess of powder

homogeneity of metal powder and polymer binders, rheological properties, and shrinkage ratio after sintering. From the results, the feedstock after the 1^{st} injection shows a better homogeneity than the as-received feedstock because the distribution of powder-polymer binders becomes more homogeneous due to re-mixing effects between the screw and barrel during the injection molding process. As the number of recycling numbers increases up to six-times, the feedstock shows little differences in terms of the homogeneity and the shrinkage ratio after sintering. However, the rheological properties affecting the injection process are enhanced up to the 4^{th} injection, but shows a tendency to decrease thereafter.

Therefore, recycling up to four-times is advantageous for the injection process, but recycling more than that has an adverse effect on the flowability of the injection process, in the case of feedstock used in this study. It is considered to be the cause of the change in the packing ratio of the powder particles in the feedstock due to the shear and compressive stress generated during the repeated injection process. However, it should be noted that these numbers of recycling could be different by the size and shape of parts.

Acknowledgments

This study has been conducted with the support of the Korea Institute of Industrial Technology as "Development of root technology for multi-product flexible production (kitech EO-21-0008)".

REFERENCES

[1] R.M. German, A. Bose, Injection Molding of Metals and Ceramics, Princeton 1997.
[2] S.T. Paul Lin, R.M. German, J. Mater. Sci. **29**, 5367-5373 (1994).
[3] J.M. Jang, W. Lee, S.-H. Ko, C. Han, H. Choi, Archives of Metall. and Mater. **60** (2), 1281-1285 (2015).
[4] A. Dehghan-Manshadi, P. Yu, M. Dargusch, D. Stjohn, M. Qian, Powder Tech. **364** (15), 189-204 (2020).
[5] K.M. Kulkarni, Future looking bright for PIM, Met. Powder Rep. **55**, 40-42 (2000).
[6] Y.S. Zu, S.T. Lin, J. Mater. Peocess. Technol. **71**, 337-342 (1997).
[7] L.H. Cheng, K.S. Hwang, Y.L. Fan, Metall. Mater. Trans. **40A**, 3210 (2009).
[8] A. Manonukul, W. Likityingwara, P. Rungkiatnawin, N. Muenya, S. Amoranan, W. Kittinatapol, S. Surapunt, J. Solid Mechanics and Mater. Eng. **1** (4), 411 (2007).
[9] J.M. Jang, H. Lee, W. Lee, Y.-I. Kim, S.-H. Ko, J.H. Kim, J.-S. Lee, J.-P. Choi, JPN. J. Appl. Phys. **53** (5S3), 05HA03 (2014).
[10] Y. Li, B. Huang, X. Qu, Powder Metall. **41** (1), 86 (1999).
[11] C.M. James, L. Daeyoung, Int. J. Powder Metall. **30**, 103 (1994).
[12] O.A. Novac, D. Anton, T. Novac, PM'94 Paris **2**, 1113 (1994).
[13] C. Karatas, A. Kocer, H.I. Unal, S. Saritas, J. Mat. Proc. Tech. **152**, 77 (2004).
[14] T.J. Garino, A.M. Morales, B.L. Boyce, Microsyst. Tech. **10** (6-7), 506 (2004).
[15] B.-H. Cha, J.-S. Lee, J. Kor. Powd. Met. Inst. **16** (5), 342 (2009).
[16] Y.M. Li, S.J. Liu, X.H. Qu, B. Huang, J. Mater. Proc. Tech. **137**, 65 (2003).