DOI: 10.24425/amm.2021.136360

MIN CHUL OH[1,2], BYUNGMIN AHN[1*]

DEVELOPMENT OF ADMIXED LUBRICANT FOR WARM DIE AND WARM COMPACTION OF HIGH-DENSITY PM IRON

The objective of the present research is to develop new admixed lubricants which can be used for high-density sintered iron when processed using warm die and warm compaction. Depending on various lubricants, the effect of compaction temperature on the ejection behavior and sintered properties was studied. Lubricants were prepared by mixing of Zn-stearate and ethylene bis stearamide (EBS) in various compositions. The iron powders blended with lubricants were compacted under the pressure of 700 MPa at various temperatures. The green compacts were sintered at 1120°C for 30 min. Microstructure, density, hardness, and transverse rupture strength of sintered materials with different lubricants were investigated in detail.

Keywords: powder metallurgy, lubricants, ejection pressure, warm die, warm compaction

1. Introduction

When manufacturing products by powder metallurgy processes, it is possible to manufacture near-net shaped products without special post processing, such as machining [1-4]. One of major issues in conventional powder compaction and sintering processes is to improve the relative density. To resolve this issue for Fe-based PM alloys, great efforts have been made to reduce internal pores via various methods, such as copper infiltration, double pressing and sintering, and hot forging have been tried [5-7]. However, these approaches reduce productivity and increase production costs.

In the mass production of sintered Fe alloys, the warm die process or warm compaction can be applied instead for higher density. The warm die process is one of powder compaction methods in which the die is heated in advance of powder filling, then non-heated powder is filled. In contrast, the warm compaction is one that after powder is filled then both the die and powder are heated simultaneously. Although the control of temperature and load is important during the warm die process and warm compaction, the role of lubricants is also very critical to achieve high-density for sintered alloys. Lubricants can increase the flowability between die and powders and reduce the friction created against the die wall [8-9]. However, when a large amount of lubricant is used, excessive lubricant may remain even after sintering and/or may form coarse pores which deteriorate sintered properties, such as density and mechanical properties. The metallic residues in lubricants may also cause unnecessary reactions with base alloy powders which cause surface soot and dimensional inaccuracy.

Therefore, in the present study, a lubricant suitable for warm die process and warm compaction of high-density iron PM alloy was developed. The density variation, ejection behavior, and mechanical properties were characterized depending on the processes and various lubricants. These characteristics were discussed in detail for both warm die and warm compaction processes and compared with those of room temperature compaction.

2. Experimental

To develop lubricants for warm die process and warm compaction of iron-based PM alloys, two commercial lubricants, ethylene bis stearamide (EBS) and Zn-stearate, were mixed in five different compositions as shown in Table 1. In this study, the admixed lubrication process was used in which lubricant is pre-mixed with metal powders prior to the compaction process. The lubricant of 0.5 wt% was added to pure iron powders (AHC 100.29 manufactured by Höganäs), and the mixture was then blended in a tubular mixer for 30 min.

[1] AJOU UNIVERSITY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING AND DEPARTMENT OF ENERGY SYSTEMS RESEARCH, 206 WORLDCUP-RO, SUWON, GYEONGGI, 16499, KOREA
[2] AI & MECHANICAL SYSTEM CENTER, INSTITUTE FOR ADVANCED ENGINEERING, 175-28 GOAN-RO 51 BEON-GIL, YONGIN, GYEONGGI, 17180, KOREA

* Corresponding author:byungmin@ajou.ac.kr

TABLE 1

Composition of newly developed lubricants

Lubricant notation	EBS (wt%)	Zn-stearate (wt%)
EZ 19	10	90
EZ 37	30	70
EZ 55	50	50
EZ 73	70	30
EZ 91	90	10

For the warm die process, the non-heated mixture of lubricant and iron powder was filled into the pre-heated die. For the warm compaction process, both the powder-lubricant mixture and die were simultaneously heated. As initial tests, for both warm die and warm compaction processes, the powders with five lubricants were compacted at 70°C with a fixed pressure of 700 MPa using a uniaxial hydraulic press. The green compacts were sintered in an atmosphere of N_2 and H_2 at 1120°C for 30 min with the heating rate of 10°C/min. While heating to the sintering temperature, the temperature was held at 760°C for 30 min for de-lubrication. Both the green and sintered densities were measured for specimens with five different lubricants.

After the initial tests, two lubricants (EZ55 and EZ73) out of five were selected. Then using these two lubricants, the warm die and warm compaction processes were repeated at different temperatures of 70, 80 and 90°C, respectively. For each condition and specimen, the ejection behavior (peak and sliding loads), hardness, transverse rupture strength (TRS), and microstructure were characterized.

3. Results and discussion

The densification behavior of iron powder mixed with different lubricants and compacted using different processing methods is shown in Fig. 1. When the powder-lubricant mixture is compacted using conventional room temperature process (Fig. 1a), as the amount of EBS increases from EZ 19 to EZ 91, the green density slightly decreases. This stemmed from both that the density of EBS (0.98 g/cm^3) is lower than that of Zn-stearate (1.10 g/cm^3) and that the Zn-stearate is generally more efficient in lubrication than EBS at room temperature. On the contrary, when warm temperature is applied (warm die in Fig. 1b and warm compaction in Fig. 1c), the green densities first increase with EBS contents then decrease after the peak values at EZ 55. The increase of density from EZ 19 to EZ 37 stemmed from the partial softening of EBS which occurred during the warm die and warm compaction processes. The softening temperature of EBS is lower than that of Zn-stearate, and thereby the densification is more efficient with the presence of EBS. However, when EBS contents exceed the optimum amount (EZ 55), the green densities decrease as EBS contents increase. This decrease is attributed to the increase of non-softened EBS which has lower the lubrication efficiency. It can be improved by increase of either processing temperature or heating time.

Both the warm die (Fig. 1b) and warm compacted (Fig. 1c) specimens exhibit greater green and sintered densities than those compacted using conventional room temperature process (Fig. 1a). Both the green and sintered densities obtained using warm compaction process, where the powder and die are pre-heated at the same time, are greater than those from the warm die process. During the warm compaction process, due to the pre-heating of powder-lubricant mixture, softening of lubricant occurs uniformly so that it flows well between the iron powders during the compaction because of its low viscosity [10]. In this consequence, the warm compaction process is more efficient for high-density compaction than the warm die process.

In all three processing methods (Fig. 1a, 1b, and 1c), for specimens with higher Zn-stearate contents (EZ 19 and EZ 37), the density decreases even after sintering, while the sintering improves the density for specimens with lower Zn-stearate contents (EZ 55, EZ 73, and EZ 91). The metallic soap lubricants, such as Zn-stearate or Al-stearate, have excellent lubrication efficiency, however metallic residues from these metallic soap lubricants which remain after de-lubrication decreases the sintered density [11,12]. On the other hand, the amide-based lubricants, such as EBS, have relatively lower lubrication performance compared to the metallic soap lubricants but have excellent de-lubrication behavior. Therefore, when the EBS is added more than a certain amount, the sintered densities are greater than the green densities.

After the tests on densification behavior shown in Fig. 1, two lubricants (EZ 55 and EZ 73) out of five were selected. Figure 2 shows the variation of sintered density and ejection behavior (peak load and sliding load) for iron powders mixed with these two selected lubricants, depending on warm process methods (warm die and warm compaction) and processing temperatures. As shown in Fig. 2a, in all processing temperatures

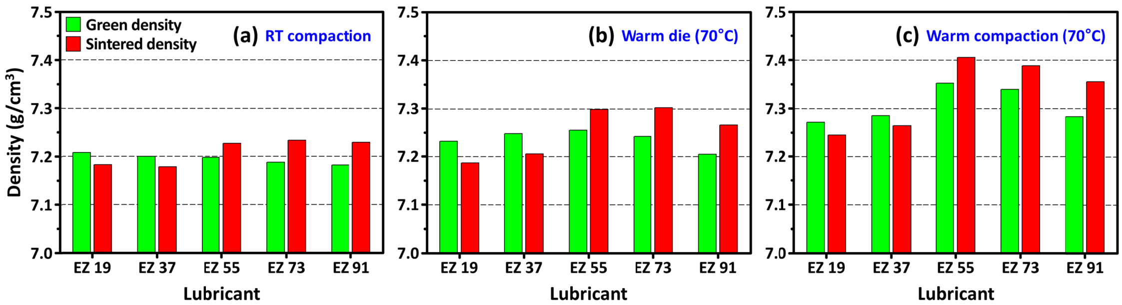

Fig. 1. Densification behavior of iron powder according to lubricant compositions and compaction methods: (a) room temperature compaction, (b) warm die process at 70°C, and c) warm compaction process at 70°C

(70, 80, and 90°C), warm compaction provides greater sintered density than warm die process. For both warm die and warm compaction processes and for both EZ 55 and EZ 73 lubricants, the processing temperature of 80°C provides highest sintered density than other two temperatures. In general, at higher temperatures, lubrication becomes more efficient because of its softening. However, if the temperature is too high, the green density exceeds a certain level during compaction so that relatively large number of closed pores can be generated. These closed pores impede delubrication by clogging the passage which evaporated lubricant is released during sintering. Therefore, the decrease of sintered density from 80°C to 90°C is attributed to both the presence of closed pores and residual lubricant.

Regarding the friction reduction by lubricants, Fig. 2b and Fig. 2c show the ejection behavior of sintered iron depending on the lubricants and processing temperatures. Once the pressurization is completed in the compaction, the ejection stroke involves a peak pressure (peak load) to start sliding the compact out of the tool, followed by a lower pressure (sliding load) to sustain the ejection motion. As expected, both peak and sliding loads in warm compaction is much lower than those in warm die process due to the softening and melting of lubricants. The peak load is more sensitive to the process temperature so that it decreases as the temperature increases. On the contrary, the sliding load is virtually independent on the process temperature.

Figure 3 shows TRS and micro-Vickers hardness of sintered iron depending on the lubricants and processing temperatures. These results of mechanical properties are consistent with the densification behavior mentioned earlier in Fig. 1 and 2, because elimination of pores is a crucial factor for controlling mechanical properties in powder metallurgy. Similar to the sintered density, both TRS and hardness are greater when processed by the warm compaction compared to the warm die process. Also, both TRS and hardness increase from 70°C to 80°C but decrease from 80°C to 90°C. As discussed above, the closed pores formed at 90°C processing temperature cannot be completely eliminated by sintering because evaporated lubricant is trapped inside the closed pores.

Figure 4 shows SEM microstructure of sintered iron with EZ 73 lubricant processed by warm die and warm compaction at 80°C and 90°C, respectively. For both warm die and warm compaction processes, more pores were observed in specimens processed at 90°C (Fig. 4c and 4d) than in those processed at 80°C (Fig. 4a and 4b). This result is consistent with density

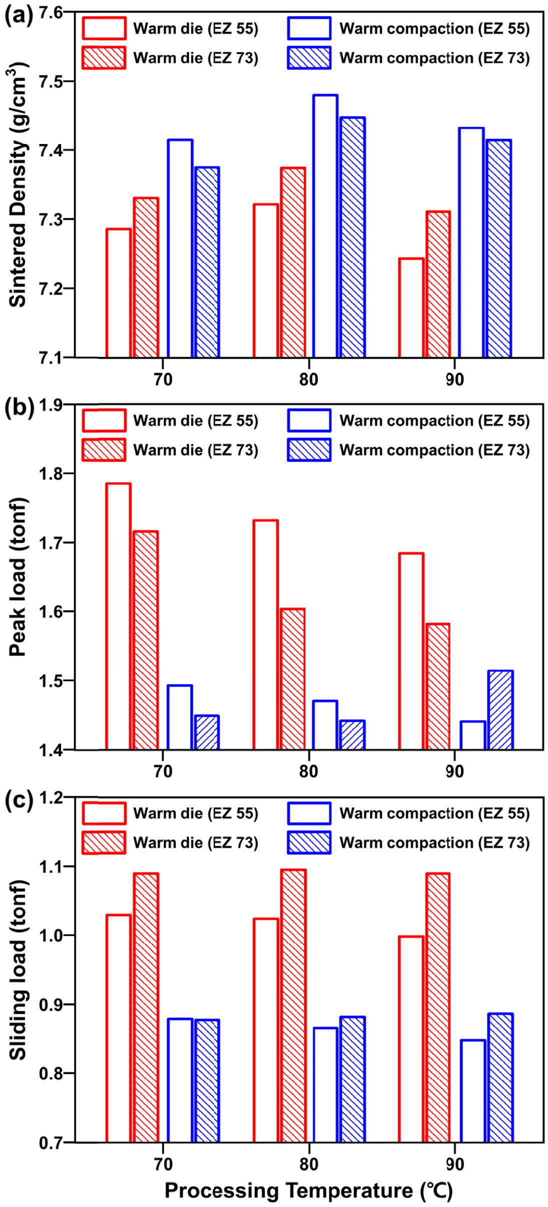

Fig. 2. (a) Sintered density, (b) peak load of ejection, and (c) sliding load of ejection according to warm die and warm compaction temperature of iron powder and to lubricant compositions, EZ 55 and EZ 73

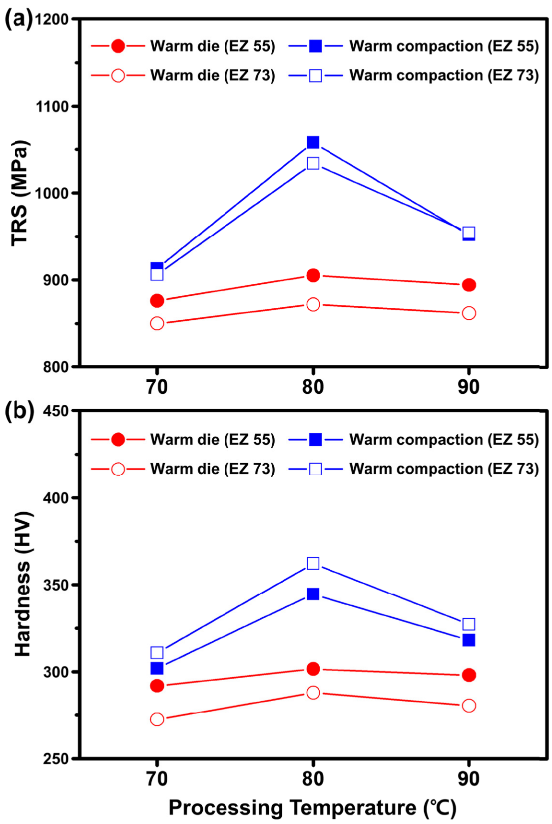

Fig. 3. (a) Transverse rupture strength (TRS) and (b) micro-Vickers hardness of sintered iron according to warm die and warm compaction temperature of iron powder and to lubricant compositions, EZ 55 and EZ 73

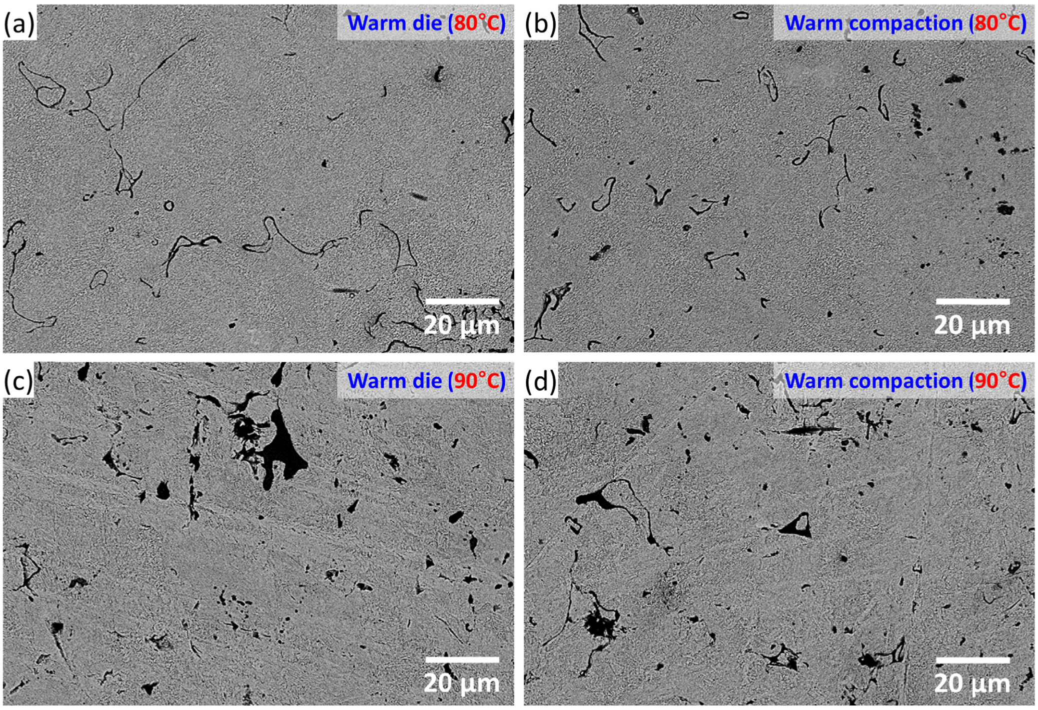

Fig. 4. SEM microstructure of sintered iron mixed with EZ 73 lubricant according to compaction methods and temperatures: (a) warm die at 80°C, (b) warm compaction at 80°C, (c) warm die at 90°C, and (d) warm compacting at 90°C

measurements and mechanical properties and is attributed to trapped gas evaporated from lubricant. When the process temperature was 80°C, a few thin pores in the size range of 2~3 µm were observed both in sintered specimens processed by warm die (Fig. 4a) and warm compaction (Fig. 4b), and there is no significant difference in microstructure between warm die and warm compaction processes. However, when the process temperature was 90°C, relatively larger closed pores up to about 15 µm were observed in warm die processed specimen (Fig. 4c), while 5~10 µm pores were observed in warm compaction processed specimen (Fig. 4d). This result stemmed from the fact that densification was more efficient in warm compaction because both iron powder and lubricant were pre-heated simultaneously.

4. Conclusions

In this study, two major commercial lubricants (EBS and Zn-stearate) were combined in various ratios to develop new lubricant for high density iron-based PM alloys. Zn-stearate has superior lubrication properties than EBS, but the melting point of EBS is lower so it softens rapidly at a relatively low temperature. Therefore, during warm die and warm compaction processes, EZ 55 (EBS 50% and Zn-stearate 50%) and EZ 73 (EBS 70% and Zn-stearate 30%) show excellent lubrication behavior. However, when a large amount of EBS is added, un-softened EBS lowers lubrication properties. For high sintered density and improved mechanical properties of iron-based PM alloys, it is recommended to use a lubricant with an appropriate amount of EBS and to perform warm compaction process at temperatures between 75 and 85°C.

Acknowledgments

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (No. 2021R1A2C1005478).

REFERENCES

[1] Y.Y. Li, T.L. Nagi, D.T. Zhang, Y. Long, W. Xia, J. Mater. Process. Technol. **129**, 354 (2002).
[2] N.G. Tupper, J.K. Elbaum, H.M. Burtle, JOM **30**, 7 (1978).
[3] W. Kehl, M. Bugajska, H.F. Fischmeister, Powder Metall. **26**, 221 (1983)
[4] G. Welsch, Y.-T. Lee, P.C. Eloff, D. Eylon, F.H. Froes, Metall. Trans. A **14**, 761 (1983).
[5] G. Hammes, R. Schroeder, C. Binder, A.N. Klein, J.D.B. Mello, Tribol. Trans. **70**, 119 (2014).
[6] S. Unami, Y. Ozaki, S. Uenosono, JFE Technical Report **4**, 81 (2004).
[7] M.C. Oh, M. Kim, J. Lee, B. Ahn, Arch. Metall. Mater. **64**, 539 (2019).
[8] Y. Huang, J. Mater. Sci. **48**, 4484 (2013).
[9] G. Jiang, G.S. Daehn, J.J. Lannutti, Y. Fu, R.H. Wagoner, Acta Mater. **49**, 1471 (2001).
[10] M.M. Rahman, S.S.M. Nor, A.K. Ariffin, Procedia Eng. **68**, 425 (2013).
[11] M.C. Oh, H. Seok, H.-J. Kim, B. Ahn, Arch. Metall. Mater. **60**, 1427 (2015)