Abstract:

Dewaxing behavior of iron powder compacts containing zinc stearate (ZnSt) or ethylene-bisstearamide (EBS) was directly observed during sintering. First, small droplets of lubricant melt appeared scattered on the surface. Then, they gradually formed large pools, and finally vaporized. In addition, melt of ZnSt more remarkably bubbled than that of EBS during vaporization. After sintering the compact containing ZnSt in nitrogen, spot stains of rough surface and concentrated zinc with the diameter of 1 mm were found on the top of their surface. It is believed that the lubricant melt inhomogeneously few out through the easier paths in the compacts to the surface accompanied by the exhausting gas of the decomposed lubricants.

## 1. Introduction

In the iron powder compacting process, it is general practice to add an organic lubricant powder such as zinc stearate (ZnSt) or ethylene-bisstearamide (EBS) in order to improve the flowability of the powder, increase green density, and reduce ejection force. Although the larger part of such organic lubricants is removed in the sintering process, in some cases, the lubricant remains on the surface of the sintered compact in the form of soot or stain, which reduces the quality and yield of sintered products.

The decomposition behaviors of simple ZnSt and EBS have been clarified previously in a number of studies[1–3]. The decomposition behavior of organic materials contained in metal powders had also been studied by German, particularly as it pertains to injection molded metal parts[4]. Nayar et al. reported on the causes of typical defects in the sintering of metal powder compacts and the related countermeasures[5], and Kameoka et al. analyzed the mechanism of rough surfaces of sintered

## 2. Experimental Method

### 2.1 Raw Materials and Blending Method

The raw materials used were water-atomized iron powder (JIP®300A), electrolytic copper powder, and graphite powder. These materials were weighed to obtain a mass ratio of 97.2 : 2 : 0.8. As lubricants, 1 mass% of either zinc stearate (hereinafter, ZnSt) powder or ethylene-bisstearamide (EBS) powder was added to the raw material powder. These powders were then mixed using a V blender in order to prepare mixed powders. A segregation-free treated mixed powder (ZnSt-CMX) was also prepared by adding 1 mass% of ZnSt. The lubricant compositions and mixing methods used in preparing the experimental materials are shown in Table 1.

### 2.2 Experimental Conditions

The mixed powders described above were compacted in cylindrical shapes with a diameter of 25 mm and thickness of 5 mm or 25 mm so as to obtain a green density of 7.0 or 7.2 Mg/m$^3$. As shown in Fig. 1(a), these green compacts were dewaxed for 20 min at 700°C, followed by sintering with a holding time of 20 min at 1 130°C. As the atmospheric gas, endothermic gas produced from propane gas (RX gas), a mixed gas of 80%$N_2$-20%$H_2$ ($N_2$-$H_2$) gas, or $N_2$ gas was used.

(1) When green compacts containing ZnSt were sintered in $N_2$ gas, staining of the sintered compacts was remarkable; however, stains were not observed with EBS added green compacts.
(2) The temperature at which leaching of the lubricant melt began was 50°C higher with the ZnSt-added compacts than with EBS addition.
(3) With ZnSt-added segregation-free treated iron powder, the starting temperature of leaching of the lubricant and the starting temperature of bubbling were approximately 20°C lower than with the ZnSt mixed powder.
(4) In tests of specimens with green densities of 7.0 and 7.2 $Mg/m^3$, no differences in dewaxing behavior related to the green density were observed.
(5) The leaching temperature of the lubricant and thickness of the green compact were unrelated. However, the size of the lubricant melt pools became larger as the thickness of the green compacts increased.
(6) In the stains on sintered compacts of the ZnSt-added material, the surface morphology and degree of oxidation changed in a concentric manner, in which Zn had segregated to the central part of the stain. This was considered to show that the ZnSt melt leached out from the central part of the stain.
(7) In order to avoid stains on sintered compacts, it is important to control the properties of the raw materials, including the lubricant and iron powder, the process of migration of the lubricant melt, and the 3-way reaction of the lubricant, green compact, and atmospheric gas.

## References

1) Legzdins, C. F.; Samarasekera, I. V.; Troczinski, T. Proc. 1998 PM World Congress. 1998, p. 266.
2) Dwyer, J.; Nayar, H.; Gerristead, W.; Wasicziko, B. Adv. Powder Metall. & Particul. Mater. 1992, p. 3–183.
3) White, G.; Nayar, H. Adv. Powder Metall. & Particul. Mater. 1996, vol. 3, p. 10–27.
4) German, R. M. Inter. J. Powder Met. 1987, vol. 23, no. 4, p. 237.
5) Nayar, H. S. Metals Handbook 9th Ed. vol. 7, compiled by Klar, E. American Society for Metals, Ohio, 1984, p. 339.
6) Kameoka, T.; Ishihara, K.; Hamamoto, H.; Obayashi, M. Abstracts of Jpn. Soc. Powder and Powder Metall. 1983, p. 78.
7) Kondoh, M.; Takemoto, S.; Urata, I. J. Jpn. Soc. Powder and Powder Metall. 1998, vol. 45, no. 5, p. 412.
8) Kawano, T.; Ono, T.; Ozaki, Y. Euro PM2009 Congress Proceedings (Sintering). 2009.
9) Kawano, T.; Ono, T.; Ozaki, Y. Abstracts of Spring Meeting of J. Jpn. Soc. Powder and Powder Metall. 2009, p. 132.
10) Kawano, T.; Ono, T.; Ozaki, Y. Curr. Adv. Mater. Process. 2009, vol. 22, no. 2, p. 1094.