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"Cleanmix" HDX Providing High Green Densities and "Cleanmix" LEX Eliminating Trouble in Compaction Process†

ONO Tomoshige*1 OZAKI Yukiko*2

Abstract:

"Cleanmix" HDX providing high green densities, and "Cleanmix" LEX eliminating troubles at compaction process were developed for the purpose of corresponding to the high-strength and complicated form of sintered parts. "Cleanmix" HDX realizes high green density, 7.3 Mg/m^3, at 686 MPa, which is equal density obtained by warm compaction processes. For HDX, a compression process was analysed and the factor controlling green density was considered with the filling die density. And it was confirmed that the green density of HDX is not affected by die temperature. It was certified that the lubricant for LEX has high lubricity at low lubricant concentration.

1. Introduction

In sintered parts, centering on automobile parts, the trends toward downsizing of parts for weight reduction and integration of multiple parts for cost reduction are accelerating. Downsizing of parts requires higher strength by high density compaction, or densification of the compact, while integration of multiple parts requires easy compaction because the shapes of integrated parts tend to be more complex.

A variety of high density compaction methods for sintered parts have been adopted, such as the 2 press, 2 sinter (2P2S) method, warm compaction, die wall lubrication compaction, and high pressure compaction at 700 MPa and higher. All of these methods have the drawback of low productivity, which is a factor in increased part manufacturing costs. On the other hand, with more complex part shapes, compaction is generally more difficult. Because metal powders are compacted by compression in a die, "springback" is a problem in powder metallurgy. Springback is a phenomenon in which the volume of the compact expands when the elastic deformation of the compact is relaxed during the product is ejected from the die by pressing the die down after compaction. This is accompanied by frictional force between the green compact and the die wall. Moreover, because the area of contact between the compact and the die increases with more complex part shapes, this frictional force also increases, causing surface scratches on green compacts. This invites damage of the green compacts, resulting in reduced product quality and yield.

Against this background, JFE Steel developed a segregation-free-treatment powder[1)], "Cleanmix" HDX (hereinafter, HDX) which can realize high strength in general compaction at pressures under 700 MPa without using a special high density compaction process, and "Cleanmix" LEX (hereinafter, LEX) which reduces frictional force during the ejection of green compacts after compaction, with the aim of reducing part manufacturing costs, improving quality, and increasing yield.

This paper introduces the features of these products, and describes the results of studies on the mechanism of densification and mechanism of low ejection force.

2. Experimental Method

2.1 Sample Materials

In order to study the properties of HDX, 2.0 mass% of electrolytic copper powder (CE-25; manufactured by Fukuda Metal Foil & Powder Co., Ltd.), 0.8 mass% of natural graphite powder (KGr; manufactured by Asbury

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*1 Senior Researcher Manager,
Iron Powder & Magnetic Materials Res. Dept.,
Steel Res. Lab.,
JFE Steel

*2 Dr. Sci.,
General Manager, Iron Powder & Magnetic Materials Res. Dept.,
Steel Res. Lab.,
JFE Steel

Graphite Mills, Inc.), and 0.5 mass% of the HDX lubricant were added to high compressibility atomized iron power “JIP®” 304AS, and segregation-free treatment was performed. For comparison purposes, a simply mixed powder was also prepared by adding 0.8 mass% of zinc stearate powder (hereinafter, ZnSt), which is a widely used general-purpose lubricant. For comparison with warm compaction, which is one densification process, a segregation-free powder for warm compaction use (hereinafter, WC)[2)] was also prepared by adding 0.6 mass% of a warm compaction lubricant.

The LEX samples were prepared by adding 2.0 mass% of electrolytic copper powder (CE-25; manufactured by Fukuda Metal Foil & Powder Co., Ltd.), 0.8 mass% of natural graphite powder (KGr; manufactured by Asbury Graphite Mills, Inc.) and 0.8 mass% of the LEX lubricant to the general-purpose atomized iron power “JIP®” 301A as the base iron powder and performing segregation-free treatment. As with HDX, a simply mixed powder containing 0.8 mass% of added ZnSt was also prepared as a comparison material.

In all cases, the simply mixed powders were blended for 15 min using a V blender, and segregation-free treatment was performed using a dedicated blender.

2.2 Method of Evaluating Basic Properties

The apparent density and flow rate of the sample powders were measured in accordance with JIS Z 2504 and JIS Z 2502 (JIS: Japanese Industrial Standards), respectively. The sample powders were filled in a superhard die with an inner diameter of 11.3 mm and compacted at 490, 588, or 686 MPa. The ejection force when the die was pressed down during ejection of the green compact was measured. Green density was calculated from the dimensions and weight of the obtained green compacts.

Compaction of WC was performed at a temperature of 130°C for both the powder and the die.

2.3 Measurement of Density-Pressure during Compaction

The features of the HDX and LEX described in this paper are demonstrated during compaction. In particular, the green density of HDX increases in compaction at the same compaction pressure. In compaction, densification progresses due to the rearrangement and deformation of the particles. In order to study which compression processes have distinctive features, the sample powders were filled in a tablet-shaped die with an inner diameter of 25 mm and compressed by raising the lower punch while keeping the upper punch in a fixed condition, and the compacting force was recorded with a data logger. The rising speed of the lower punch and the maximum compaction force were set at 10 mm/min and 686 MPa, respectively.

During compaction under mass production conditions, it is said that the die temperature rises as the number of compacting operations increases due to the frictional heat generated between the die and the green compacts, finally reaching a temperature on the order of 60–100°C. Therefore, in this research, the effect of the die temperature on green density and ejection force was studied by making the measurements described above with the die temperature increased to 60–100°C with a heater and the sample powders at room temperature.

A comparative study was also made using an Fe-4mass%Ni-1.5mass%Cu-0.5mass%Mo partially alloyed steel powder (SIGMALOY® 415S), which is an alloy steel powder used in parts requiring high strength, and 0.5 mass% natural graphite powder (CE-25; manufactured by Fukuda Foil & Powder Co., Ltd). In this case, a mixed powder was prepared by adding 0.5 mass% of the HDX lubricant to this material and performing segregation-free treatment, and a mixed powder was prepared by simple mixing of 0.5 mass% ZnSt.

2.4 Study of Die Concentration of LEX Lubricant

In LEX, a special lubricant which was developed in order to reduce ejection force is used. To clarify the lubricity of this lubricant on the die surface, specified amounts of the lubricant were coated on the die in advance, and compaction was performed. The relationship between the coating weight and ejection force was investigated. At the same time, simply mixed powders were prepared by adding 0.4 mass% or 0.8 mass% of the LEX lubricant to “JIP®” 301A and compacting the powders. The degree of concentration of the lubricant on the die wall was then estimated by comparing the ejection force with these specimens and the ejection force in compaction when the die wall was coated in advance, as described above.

3. Results and Discussion

3.1 Basic Properties of Powders

Tables 1 and **2** summarize the properties of the respective mixed powders.

As powder properties, HDX has a high apparent density and low flow rate (high flowability) in comparison with the simply mixed ZnSt-added material used as a comparison material. LEX shows a low apparent density and low flow rate. This difference is considered to be attributed to the change in the shape of the iron powder particles and change in the surface properties of the iron powder particles due to mixing treatment in segregation-free treatment.

Figure 1 shows the relationship between green den-

Table 1 Powder and compaction properties of "Cleanmix" HDX

	Apparent Density (Mg/m^3)	Flowability (s/50 g)	Green density* (Mg/m^3)	Ejection force* (MPa)
HDX	3.26	21.9	7.30	15
ZnSt	3.26	25.8	7.23	16
WC	3.30	21.5	7.30	26

*Compacted at 686 MPa

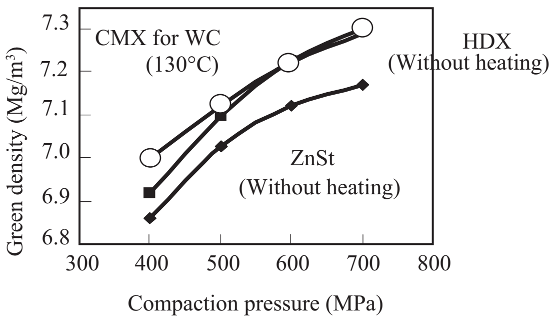
Mix composition: Fe-2.0mass%Cu-0.8mass%Graphite-Lubricant**
**ZnSt : 0.8 mass% Zinc stearate
WC: 0.6 mass% Heat resistant lubricant
HDX: 0.5 mass% Newly developed lubricant

Table 2 Powder and compaction properties of "Cleanmix" LEX

	Apparent Density (Mg/m^3)	Flowability (s/50 g)	Green density* (Mg/m^3)	Ejection force* (MPa)
LEX	3.21	24.9	7.15	10
ZnSt	3.28	31.0	7.15	13

*Compacted at 686 MPa

Mix composition: Fe-2.0mass%Cu-0.8mass%Graphite-Lubricant**
**ZnSt : 0.8 mass% Zinc stearate
LEX: 0.8 mass% Newly developed lubricant

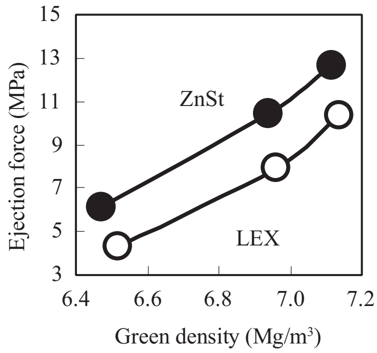


Mix composition: Fe-2.0mass%Cu-0.8mass%Graphite-Lubricant**
**ZnSt : 0.8 mass% Zinc stearate
WC: 0.6 mass% Heat resistant lubricant
HDX: 0.5 mass% Newly developed lubricant

Fig. 1 Relationship between green density and compaction pressure

sity and compaction pressure for HDX, the ZnSt-added simply mixed powder, and the WC powder. In all cases, the green density increased by 10% accompanying increased compaction pressure. However, when compared at the same compaction pressure, the green density of HDX is higher than that of the ZnSt-added mixed powder in compaction at 686 MPa at room temperature, and is comparable to that of warm-compacted WC, at least in compaction at pressures higher than 500 MPa.

Figure 2 shows the relationship between ejection force and green density for LEX and the ZnSt simply mixed powder. With both powders, the ejection force



Mix composition: Fe-2.0mass%Cu-0.8mass%Graphite-Lubricant**
**ZnSt : 0.8 mass% Zinc stearate
LEX : 0.8 mass% Newly developed lubricant

Fig. 2 Relationship between ejection force and green density

increases as the green density becomes larger. However, when compared at the same green density, it can be understood that the ejection force of LEX is 20% lower.

3.2 Study of Mechanism of Densification with HDX

The compressive stress-stroke curve obtained when HDX was filled in a tablet-shaped die with an inner diameter of 25 mm and compacted was analyzed using a Cooper-Eaton type empirical equation[3)], as shown in Eq. (1), and the contribution of particle rearrangement and contribution of plastic deformation to the final green density at the specified compaction pressure were calculated.

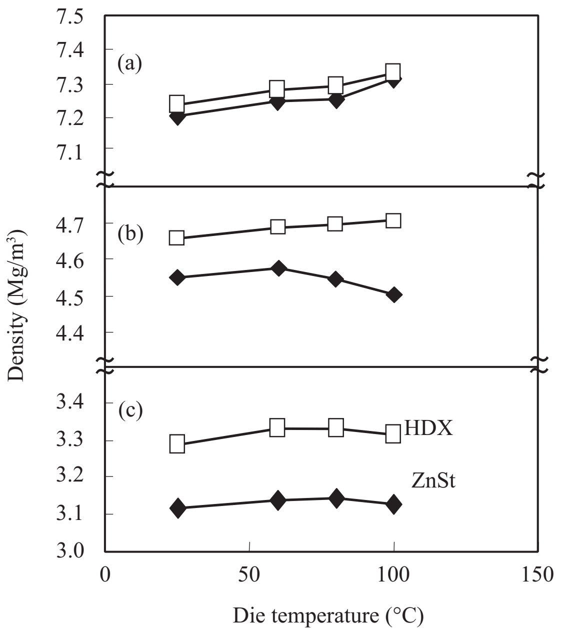
$$\frac{\varepsilon(0)-\varepsilon(P)}{\varepsilon(0)} = a_1 \exp\left(-\frac{b_1}{P}\right) + a_2 \exp\left(-\frac{b_2}{P}\right) \quad \cdots\cdots (1)$$

P: Pressure
$\varepsilon(0)$: Initial void ratio
$\varepsilon(P)$: Void ratio at pressure P
a_1, a_2, b_1, b_2: Constants

The left side of Eq. (1) shows the void reduction ratio at compaction pressure P. On the right side, the first and second terms show the contribution of particle rearrangement and the contribution of particle deformation to the void reduction ratio, respectively. Here, the void ratio was obtained from the ratio between the density of the mixed materials assuming that the true densities of the alloyed steel powder (SIGMALOY® 415S), graphite, and lubricant are 7.93, 2.20, and 1.00 Mg/m^3, respectively, and the density of the mixed materials being 7.57 Mg/m^3 assuming a void ratio of 0. The void reduction ratio on the left side corresponds to the increase in the green density from a compaction pressure of 0 to P. The right side of the equation separates the increase in green density into the increment attributable to particle rearrangement and the increment attributable to particle deformation. In general, it is thought that densification due to particle rearrangement (first term on the right side) converges at around 300 MPa, and in high pressure compaction above this pressure, densification occurs mainly by particle deformation[3)].

Figure 3 shows (a) the final compaction density at a maximum compaction pressure of 686 MPa, (b) the green density at the time of convergence of particle rearrangement, and (c) the die filling density for sample powders with die temperatures from 25°C to 100°C. The density at the start of compaction corresponds to the filling density, i.e., the density when the powder filled in the die. The filling density of HDX is higher than that of ZnSt and displays a roughly constant value in all cases, independent of the die temperature. Although Table 1 shows that the apparent densities of HDX and ZnSt are the same, the filling density of HDX is higher. This is considered to be because HDX has superior flowability, resulting in an improved filling property.

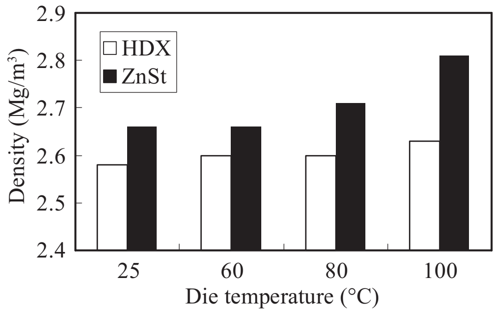
The difference in green density becomes smaller at convergence of particle rearrangement, but as above, the density of HDX is higher. The density of HDX tends to increase with temperature, which can be interpreted as showing that particle rearrangement accelerates with increasing temperature. It can be conjectured that this occurs because the lubricant is softened by heating, and



Mix composition: JIP® SIGMALOY® 415S-0.5mass%Graphite-Lubricant**
**ZnSt : 0.5 mass% Zinc stearate
HDX: 0.5 mass% Newly developed lubricant

(a) Final compaction density at 686 MPa
(b) Compaction density caused by particle re-arrangement
(c) Filling density

Fig. 3 Effect of die temperature on each compaction process



Mix composition: JIP® SIGMALOY® 415S-0.5mass%Graphite-Lubricant**
**ZnSt : 0.5 mass% Zinc stearate
HDX: 0.5 mass% Newly developed lubricant

Fig. 4 Increment of density caused by particle deformation

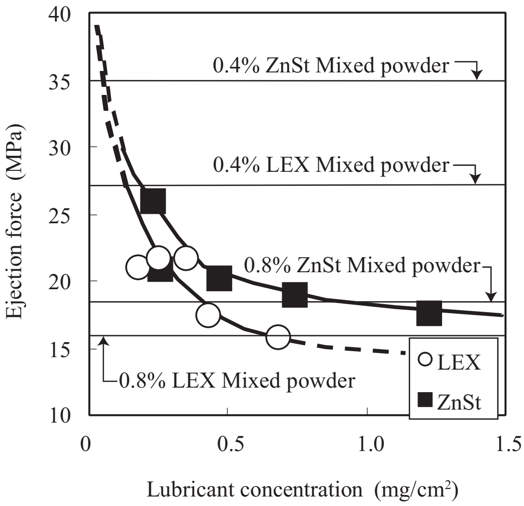
this accelerates particle movement. On the other hand, with ZnSt, the increase in density in the high temperature region is small in comparison with that at the start of compaction. It appears that ZnSt impedes the movement of particles at higher temperatures.

The final compaction density, as mentioned above, increases accompanying increasing die temperatures with both lubricants. **Figure 4** shows the temperature dependence of densification from the convergence of particle rearrangement to the final compaction temperature. The increment of density attributable to particle deformation is substantially constant with HDX, but in contrast to this, with ZnSt, the increment of density increases with temperature. From this, it is considered that the contribution of particle deformation to densification is larger with ZnSt than that with HDX. This suggests that increased green density is difficult to achieve unless the material possesses good compressibility.

Based on the above analysis, the factors which make it possible to obtain high density green compacts with HDX are considered to be high initial filling density and promotion of particle rearrangement at higher die temperatures.

3.3 Study of Mechanism of Low Ejection Force with LEX

Figure 5 shows the relationship between ejection force and the die wall lubricant concentration (amount of lubricant adhering to the die per unit of area) in die wall lubrication compaction. With both the ZnSt lubricant and the LEX lubricant, the ejection force decreases as the die wall lubricant concentration increases. The ratio of reduction in ejection force also decreases as the die wall lubricant concentration increases. Although an experiment with 0 wall lubricant was not possible as this would damage the die, the curves shown in this figure would cross at some point on the vertical axis. In other words, at low die wall lubricant concentrations, even a



ZnSt : Zinc stearate
LEX : Newly developed lubricant

Fig. 5 Relations between ejection force and wall lubricant concentration

small increase in the lubricant concentration results in a large decrease in ejection force, whereas at high die wall lubricant concentrations, there is virtually no change in the ejection force regardless of changes in the lubricant concentration. Furthermore, when LEX and ZnSt were compared, there were no large differences in ejection force in the low die wall lubricant concentration region, but a remarkable difference could be seen in the high concentration region. This shows that, in the high wall lubrication concentration region, differences in ejection force occur due to differences in the type of lubricant, in other words, differences in lubricity. However, in the low die wall lubricant concentration region, the ejection force is determined by the amount of lubricant and does not depend on the type of lubricant.

The four solid horizontal lines in Fig. 5 show the ejection force for LEX and ZnSt premixed powders. With both lubricants, the point of intersection between these solid lines and the curve showing lubricity in die wall lubrication compaction are considered to indicate the lubricant concentration on the surface of the premixed powder compacts.

With the 0.8 mass% ZnSt mixed powder, this ejection force is 18 MPa, and this value shows that the wall lubricant concentration on the die surface in die wall lubrication compaction is 0.8–0.9 mg/cm^2. The 16 MPa ejection force with the 0.8 mass% LEX mixed powder is equivalent to 0.6 mg/cm^2. In these lubricant regions, there is virtually no change in the ejection force with either ZnSt or LEX, even when the amount of lubricant is changed, and the die wall surface is considered to be adequately coated when performing die wall lubrication. Accordingly, with premixed products containing 0.8 mass% of lubricant, the amount of lubricant in the compact is considered adequate to demonstrate lubricity, and the ejection force of LEX is lower than that of ZnSt because the friction coefficient between the die and compact is reduced by that lubricity.

4. Conclusion

The mechanism of densification in “Cleanmix” HDX, which is a product for high density compaction, and the mechanism of low ejection force in “Cleanmix” LEX, for low ejection force applications, were investigated. The following conclusions were obtained.

(1) HDX realizes a high green density, comparable to that in warm compaction, at room temperature with compaction pressures of at least 500 MPa or higher. In the mechanism of densification, the most important controlling factor is considered to be the high filling density of this material. Accordingly, HDX is effective in high density compaction of iron powders composed of hard particles with little deformation capacity, such as prealloyed alloy steel powders.

(2) The lubricant used in LEX provides high lubrication performance, making it possible to reduce frictional resistance with the die surface even with low lubricant concentrations. At the same amount of lubricant addition, the ejection force with LEX is approximately 20% lower than that with ZnSt. As this suggests high lubrication performance, application of the lubricant used in LEX to compaction of parts with complex shapes is expected.

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