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| Author(s)    | Kondoh, Katsuyoshi; Watanabe, Ryuzo   |
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# Analysis of Warm Compaction Behavior of Iron Powder Particles via Cooper-Eaton Equation†

KONDOH Katsuyoshi\* and WATANABE Ryuzo\*\*

## Abstract

The effect of lubricant particles premixed with iron alloy powder on warm compaction behavior was investigated by using the Cooper-Eaton equation based on a probabilistic approach. In particular, the melting point of the lubricant was examined. Particle rearrangement significantly progresses during warm compaction when the powder temperature is lower than the lubricant melting point. On the other hand, it is hindered when the lubricant is in a liquid state at the elevated temperature. This is due to the strong dependence of the particle rearrangement on the lubrication effect between iron particles, and the formability of the solid lubricant film is poor when the temperature is beyond its melting point. The plastic deformation of iron alloy powder shows little effectiveness on the warm compaction behavior because of the small change in its compaction proof-strength at applied temperatures below 473K. The calculated total fractional volume compaction corresponds to the measured green density. This analytical method is available for quantitatively evaluating the warm compaction behavior in the powder metallurgy (PM) process.

**KEY WORDS:** (Warm compaction behavior), (Cooper-Eaton equation), (Lubricant), (Melting point), (Friction force), (Particle rearrangement), (Plastic deformation)

## 1. Introduction

From the viewpoint of applying sintered materials to automotive components, new consolidating processes, such as double press and double sintering, sintering and powder forging<sup>1,2)</sup>, marquench-repressing<sup>3)</sup>, and warm compaction processes, have been developed to increase the strength of PM iron alloys. In particular, these processes useful to increase the density cause the significant improvement of the dynamic mechanical properties such as fatigue strength and impact energy of sintered materials. The warm compaction method is also advantageous in terms of dimensional tolerance of the sintered component because of the small dimensional change of its green compact and a higher relative density after sintering. It is important to clarify the warm compaction behavior in producing near-net shape components by the warm compaction process. The effect of the lubricant premixed in the raw powder, however, has not been examined quantitatively. The study focuses on the analysis of the compaction behavior of iron alloy powder particles during the warm compaction by using the Cooper-Eaton equation<sup>4)</sup>. In particular, the effect of the lubricant on the compaction behavior is evaluated. The adaptability of this method is also examined by comparing calculated and measured results.

† Received on November 10, 2006

\* Professor

\*\* Professor Emeritus of Tohoku University

## 2. Experimental

### 2.1 Characteristic of raw iron powder and lubricants

Iron alloy powder with a mean particle size of 85 $\mu$ m was used as raw material, where the chemical composition was Fe-0.5Ni-1.0Mo/mass% (Kobe steel, 46F4H). **Table 1** shows the characteristics of three kinds of stearic acid lubricant particles.

**Table 1 Characteristics of lubricant powders used in warm powder compaction test.**

|                | Melting point (K) | Aqua (%) | Acid (mgKOH/g) | Metallic (%) |
|----------------|-------------------|----------|----------------|--------------|
| <b>Lub I</b>   | 352               | 0.1      | 2.3            | 0.8          |
| <b>Lub II</b>  | 393               | 0.1      | 2.8            | 0.9          |
| <b>Lub III</b> | 431               | 0.1      | 2.9            | 1.2          |

The lubricant of 0.8mass% was added and mixed with the raw material. The melting point of the lubricant ranged from 352 to 431K by controlling its metallic content. Carbon particles were not included in the raw powder because the segregation or cohesion of carbon particles easily occurs. They also effect on the compaction behavior of the iron powder particles due to their lubrication effect. In using the Cooper-Eaton equation to

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estimate the effect of the plastic deformation of the powder particle, the compaction-proof strength of one iron particle is necessary. Exact measurement, however, is difficult. In the previous study<sup>5)</sup>, it has been shown that the compaction-proof strength of PM aluminum alloy consolidated by hot forging with relative density over 99% is available to estimate the contribution of the plastic deformation in the compaction behavior by using Cooper-Eaton equation. Therefore, the compaction-proof strength of the sintered and hot-forged iron material is used as the plastic deformability of the raw powder in this study.

### 2.2 Warm compaction test

Figure 1 shows a schematic illustration of the warm compaction test. The raw iron powder particles were filled into the die installed in the 250kN hydraulic press, and a uni-axial load was applied to them at the elevated temperature. The relationship between the applied pressure and the traveling displacement in the uni-axial compacting test was established as data for the compaction behavior analysis. Each raw particle in 7g amount including the lubricant was filled into a WC-Co alloy die of  $\phi 11.3\text{mm}$  diameter. The die was heated in the furnace attached to the hydraulic press, where the test temperature of the powder and the die before compaction was controlled at 296, 323, 373 and 423K. The loading speed was 0.5mm/s in warm compacting. Minimum and maximum applied loads in compacting are 98N and 78.4kN, respectively. The friction load between the green compact and the die wall was estimated by measuring the applied load in removing the green compact from the die immediately after compaction.

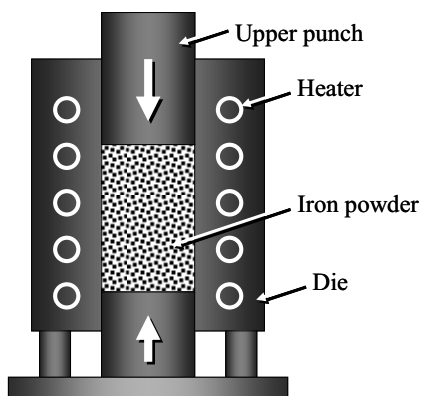


Figure 1 Schematic illustration of die set of warm compaction in using iron powders.

### 2.3 Analysis of compaction behavior of particle by Cooper-Eaton equation

The assumption included in this analysis is the same as that used in the previous study<sup>5)</sup> as follows: The compaction behavior of powder particles depends on the decrease of its volume by two independent probabilistic processes, the particle rearrangement and the plastic deformation of powder particle. The former means a

filling of large holes, which are formed in filling the powder particles into the die, by the movement of particles. The latter contributes to a filling of small pores by the deformation of each particle. The effect of the friction force at the particle surface on the particle compaction behavior is also considered in the calculation of the particle rearrangement in this study. The total fractional volume compaction  $V$  consists of the volume compactions by both the particle rearrangement and plastic deformation of the particle. Concretely speaking, it is calculated by the Copper-Eaton equation, which uses a probabilistic approach and consists of two terms of the exponential functions in terms of the applied pressure  $P$ , is shown in below.

$$V = \frac{V(0)-V(p)}{V(0)-V(\infty)} = a_1 \exp(-k_1/P) + a_2 \exp(-k_2/P) \quad (1)$$

$a_1, k_1$ ; Coefficient of particle rearrangement

$a_2, k_2$ ; Coefficient of plastic deformation of powder particle

( $a_1 + a_2 = 1$ )

$V(0)$  means the initial total volume when no hole is filled, i.e. at zero compaction pressure, and can be calculated by using the apparent density of each particle.  $V(\infty)$  means the volume of the green compact when all large holes, but no other holes, are filled. The first term of the right side of equation (1) corresponds to the compaction behavior due to the filling of holes by the movement and rearrangement of powder particles in the early stage. The second term indicates the compaction behavior by the plastic deformation of the particles. The dimensional coefficient "a" represents the fraction of theoretical compaction. Each one is achieved at the infinite pressure by the particle rearrangement or the particle plastic deformation process. The total,  $a_1 + a_2$ , is assumed to equal unity in this study when the compaction can be completely described in terms of the above three separate processes. In the case of  $a_1 > a_2$ , for example, the particle rearrangement is more dominant on the total fractional compaction volume than the plastic deformation of powder particle. The coefficient with units of pressure "k" corresponds to the magnitude of the pressure at the start of each compaction behavior. For example, it becomes difficult to consolidate the powder by the particle rearrangement when the coefficient  $k_1$  increases. All coefficients can be calculated by the non-linear optimizing method with the compacting test data of the relationship between the applied pressure and the compaction volume. This analysis enables the evaluation of not only the dependence of the particle compaction behavior on the applied pressure, but also the effects of the above two compaction mechanisms on the fractional compaction volume quantitatively.

### 3. Results

Figure 2 shows the dependence of green compact densities on the temperature ratio when applying the compaction pressure of 735MPa, where  $T/T_m$  ( $T_m$ ; melting point of each lubricant) is used as the normalized

parameter of the powder temperature. With an increase in the temperature ratio, the green density increases, and it is shown that the compaction behavior of iron alloy powder can be improved by warm compaction. When  $T/T_m$  approaches 1 and the lubricant particles premixed in the iron powder are in liquid state, the difference of the green density among the lubricants gradually decreases. When  $T/T_m$  is beyond 1, however, the density suddenly decreases, that is, the warm compaction behavior of powder is prevented. In removing the green compact from the die, irrespective of the kind of lubricant particles, the friction load gradually decreases at the temperature ratio of less than 1 as shown in Fig. 3. When the  $T/T_m$  exceeds 1, the friction load is about 9kN, which is twice of that in  $T/T_m$  below 1, and the green compact surface has a seizure or damaged area in the compacting direction.

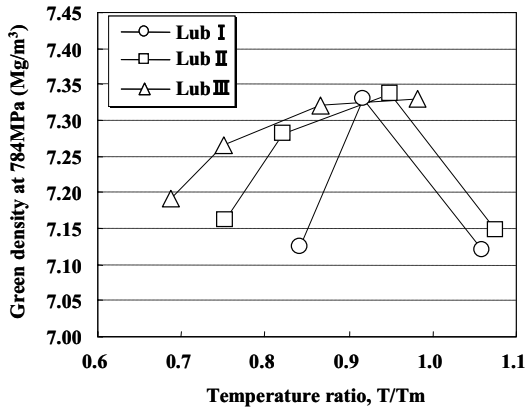


Figure 2 Dependence of measured green density of iron powder compact on temperature ratio in warm compaction.

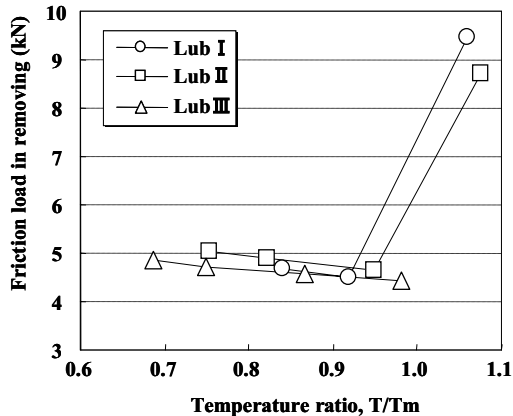


Figure 3 Changes in friction load during knocking out compacts from die under warm compaction.

Using the compacting test data, a dependence of the particle rearrangement coefficient  $a_1$  on the temperature ratio is shown in Fig. 4. Every premixed powder reveals a gradual increase of  $a_1$  with the increase in  $T/T_m$ , which means that the particle rearrangement occurs smoothly

during warm compaction. However,  $a_1$  suddenly decreases in the case of lubricant particle I or II when  $T/T_m$  exceeds 1, and the particle rearrangement behavior is obstructed. Figure 5 shows changes in the fractional volume compaction by particle rearrangement  $V_1$  with the compaction pressure.

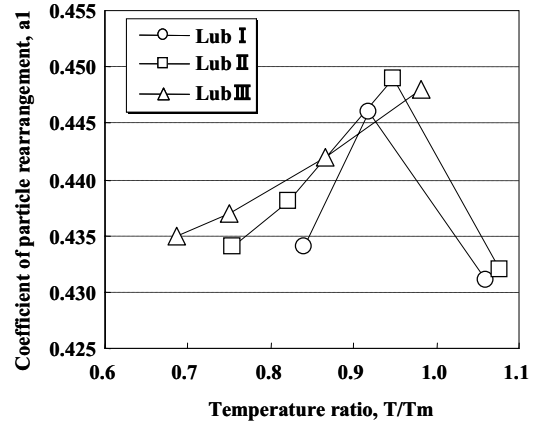


Figure 4 Calculation results on coefficient of particle rearrangement dependence on temperature.

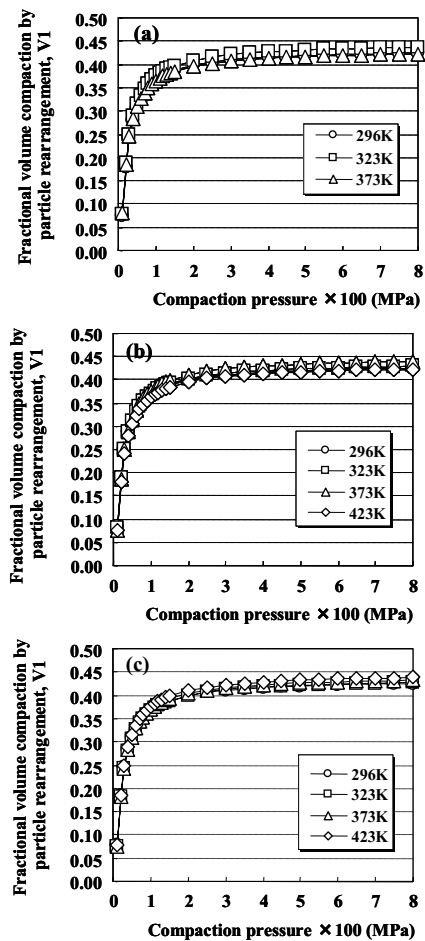
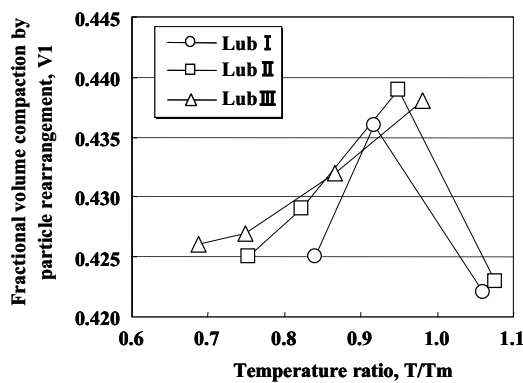


Figure 5 Calculated fractional volume compaction by particle rearrangement with compaction pressure, (a) lubricant I, (b) lubricant II and (c) lubricant III.

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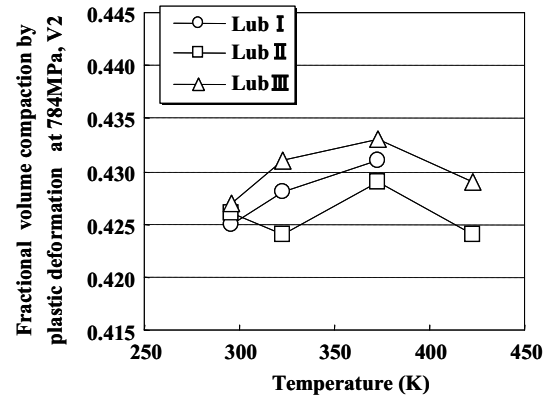
V1 of every premixed powder shows a rapid increase at the low compaction pressures of less than 300MPa, however, a remarkable increase of V1 is not obtained over 300 MPa. This means that the compaction behavior by the particle rearrangement almost stops at 300 MPa irrespective of the kind of the lubricant particles. On the other hand, the difference in the powder temperature changes the fractional volume compaction by a particle rearrangement. As shown in Fig. 6, V1 at the compaction pressure of 784 MPa gradually increases with T/Tm, and suddenly decreases when T/Tm is higher than 1. The dependence of V1 on T/Tm shows the same tendency as that of a<sub>1</sub> shown in Figure 4. This is because a<sub>1</sub> dominates V1, which is expressed as  $a_1 \cdot \exp(-k_1/P)$ , and the effect of k<sub>1</sub> on V1 is small due to slight differences between each coefficient k<sub>1</sub> such as 0.165~0.178. When considering the characteristic of the lubricants shown in Table 1, in particular, the difference of their melting points, the compaction behavior in a warm press strongly depends on the lubrication effect between the iron powder-powder or the die wall. As shown in Fig. 3, the friction load during the removal of the green compact from the die remarkably increased when the temperature ratio T/Tm exceeded 1. At the same time, the seizure phenomenon also occurred at the green compact surface. This means that when the lubricant particles contained in the iron alloy powder melt and become a liquid during warm compaction, it is then difficult to form a solid lubricant film between iron alloy particles, that is, the lubricant particles are not effective in the particle rearrangement. Therefore, irrespective of the kind of the lubricant particles, a<sub>1</sub> and V1 decrease remarkably when T/Tm is beyond 1.



**Figure 6 Dependence of fractional volume compaction by particle rearrangement on temperature ratio.**

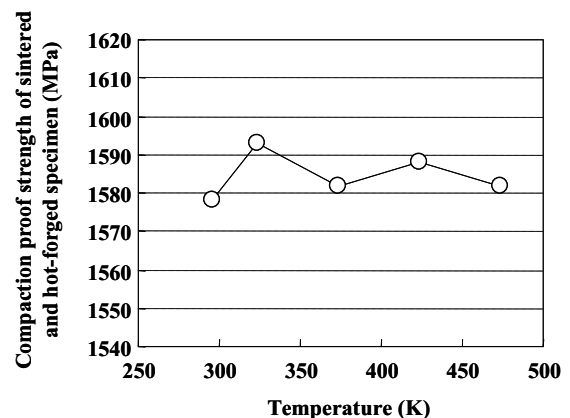
On the other hand, the frictional volume compaction by the plastic deformation V2 shows a slight change with the powder temperature in applying the compaction pressure of 784MPa as shown in Fig. 7. The difference of V1 between each lubricant is also negligible. This is because, showing in Fig.8, the compaction-proof strength of the hot-forged iron alloy specimen does not reveal a strong dependence on temperatures less than 473K, that is, the plastic deformability of the iron alloy powder does

not change significantly in warm compaction under the applied conditions.



**Figure 7 Calculated fractional volume compaction by particle rearrangement at 784 MPa with warm compaction temperature.**

Based on the calculated results on V1 and V2, the total fractional volume compaction in warm compaction depends on the temperature ratio as shown in Fig. 9. It increases proportionally with T/Tm, but, suddenly decreases when the powder temperature exceeds the melting point of each lubricant in the case of Lubricant I and II. As mentioned above, this is because V is V1+V2 and the dependence of V2 on the temperature ratio is small. Therefore, it is considered that the total fractional volume compaction depends strongly on the particle rearrangement during the warm compaction. Figure 10 shows the relationship between the total fractional volume compaction and the density after warm compaction with a pressure of 784MPa. Except for 2 data points with lower green density, which represent the results when T/Tm exceeds 1, the total fractional volume compaction corresponds well with the measured density. Accordingly, it is possible to analyze the warm compaction behavior quantitatively by using the suggested method based on the Cooper-Eaton equation.



**Figure 8 Compactability of sintered and hot forged PM iron alloy specimen at elevated temperature.**

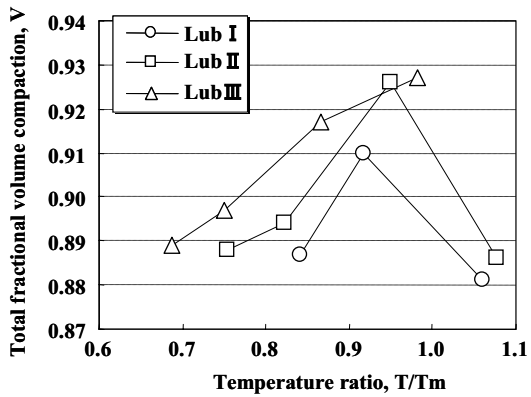


Figure 9 Dependence of calculated total fractional volume compaction on temperature ratio.

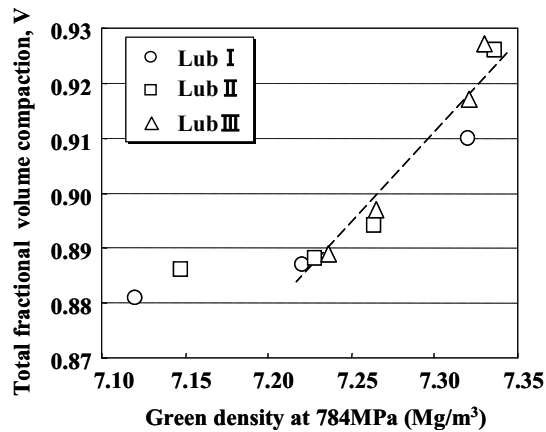


Figure 10 Relationship between calculated total fractional volume compaction and density of green compact.

#### 4. Conclusions

- (1) The particle rearrangement in warm compaction depends on the lubrication effect of the lubricant powder premixed in raw iron alloy powder, and is hindered when the compacting temperature exceeds the melting point of the lubricant because of the unstable solid lubricant film between the particle.
- (2) The particle compaction behavior depends strongly on the particle rearrangement at temperatures less than 423K.
- (3) The calculated total fractional volume compaction by the Cooper-Eaton equation corresponds well with the measured density of the green compact, and is available for the quantitative analysis of the warm compaction behavior of powder.

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