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Development of the Segregation-Free Iron Powder JIP™ Cleanmix™ ZERO Reducing Variation in Weight of Powder Metallurgy Green Parts in Mass Productive Pressing Line†

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Abstract:

The segregation-free iron powder JIP™ Cleanmix™ ZERO reducing variation in weight of green parts in powder metallurgy mass productive pressing line has been developed. It can improve the yield in the manufacturing process of the sintering part. When compaction pressing of continuous operation is restarted after the stop due to the unexpected reasons or break, Cleanmix ZERO can lower the difference between weight of parts before and after the stop. In addition, it contributes to superior filling ability to a die cavity and uniform filling in a cylinder cavity in simulated die for bush shape products. It results in the reduction of failures due to deviation from specification for part weight and dimensions at the time of the compacting. These characteristics could be realized by reducing the adhesion between the particle. It was confirmed that cohesion of Cleanmix ZERO was less than half of usual Cleanmix powder in the powders layer shear examination.

1. Introduction

Fe-based sintered machine parts are manufactured by filling an iron powder in a die and compacting the powder by pressing, followed by sintering at high temperature. Supply (filling) of the raw material to the die is generally performed by using a supplier called a filler shoe or feeder. A fixed amount of powder is supplied by reciprocal movement of the shoe above the die cavity, and excess powder is removed by leveling off the powder surface. Since the powder is not weighed, the weight of the green parts obtained in the compaction process may vary due to changes in powder density. This, in other words, results in variations in the weight of parts, which are treated as defective if they deviate from the standard specifications of the part. Much research has been done on filling behavior in the compaction process[1–9].

Figure 1 shows an example of the variation of weight in continuous compaction. Variation of weight can be broadly classified as three types. In this figure, (1) is variation of weight from part to part; small surface

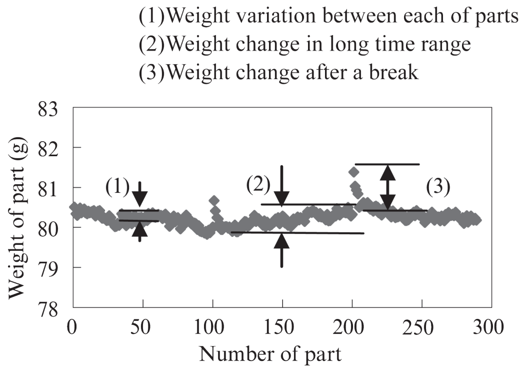

Fig. 1 Weight variation of parts during pressing

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irregularities correspond to this type. Type (2) is an undulation type, in which the weight varies with a long period. The above-mentioned individual variations take a form which is overlaid on these undulations. Type (3) is sudden, rapid variation of weight that occurs when pressing, which had been performed by continuous compaction, is stopped from some reason and then restarted. Normally, the weight of the first part after pressing is resumed is large in comparison with that before the stop, and then gradually converges on the average weight as continuous compaction proceeds[10–12].

Regarding the undulating long-period variation of weight shown above as (2), with progress in press technology, these variations have been reduced by measuring the weights of each part and feeding back this information to the compaction conditions.

However, in the case of large, discontinuous variations of weight like those which occur before or after a press stop, there is no choice but to depend on a response based on the experience of the operator, or to reject the defective parts from this portion of operation. However, this reduces product yield. In the present research, a new segregation-free iron powder JIP™ Cleanmix™ ZERO was developed to reduce defects of this type. This paper describes the advantages of this product and, in particular, reports the results of various experiments and discussion in connection with the filling property and discharging property of the new powder.

2. Experiments

2.1 Test Materials

The test materials used in this study were Cleanmix™ ZERO, in which 2% copper powder and 0.9% graphite powder as alloying powders were mixed with the atomized iron powder JIP™ 301A, and as a comparison material, conventional Cleanmix (hereinafter, "current product"), which was mixed with the same amounts of copper and graphite.

In addition, as general comparison materials, conventional mixes of zinc stearate or ethylene bisstearamide (EBS) were prepared by mixing for 15 min with a V type mixer.

2.2 Evaluation of Mixed Powders

2.2.1 Powder properties

Apparent density and flowability were measured with a 2.5 mmφ funnel and 25 cm^3 cup in accordance with JIS Z 2504 and JIS Z 2502, respectively (JIS: Japanese Industrial Standards). Each measurement was performed 3 times, and the average value was obtained.

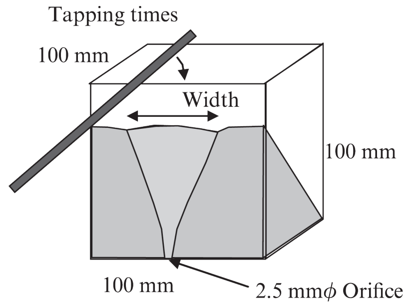

Fig. 2 Discharge test from hopper

2.2.2 Index of filling characteristic

The following two types of evaluations were performed for simple measurement of the flow and filling behavior from a filler shoe and feeder into a die cavity.

(1) Hopper Test[2]

As shown in **Fig. 2**, a specified amount of iron powder was introduced in a specified shape into an acryl container, after which the 2.5 mmφ discharge hole (orifice) was opened and the powder was discharged. A powder with excellent flowability and filling property begins to discharge simply upon opening of the orifice, the cave in the powder layer spreads as discharge proceeds, and finally, discharge stops when the cave reaches the top of the powder layer. On the other hand, a powder with inferior flowability and filling property does not discharge when the orifice is opened. At this time, discharge is encouraged by tapping the top of the container with a certain force. After the first tap, the cave in the powder layer does not spread or spreads only slightly, and discharge stops. In this case, tapping is performed again and is repeated until the cave penetrates through the top of the powder layer. The flowability and filling property of a powder can be judged to be inferior as the number of taps increases, and the flowability and filling property can be judged to be superior as the width of the aperture at the top of the powder layer becomes wider.

(2) Adhesion of Particles

The flowability and filling property of a powder is thought to be influenced by the interfacial interaction between the particles. Here, the cohesion and angle of internal friction between particles were measured by performing a shear failure test of the powder layer using a powder bed tester (PTHN-13BA; Manufactured by Sankyo Pio-Tech Co., Ltd.).

Figure 3 shows a schematic diagram of the shear test. The powder which is to be measured is formed into a powder bed with the thickness of 3 mm on the fixed plate, and preliminary pressing at 6.9 kPa is performed. Next, a weight of the specified weight is

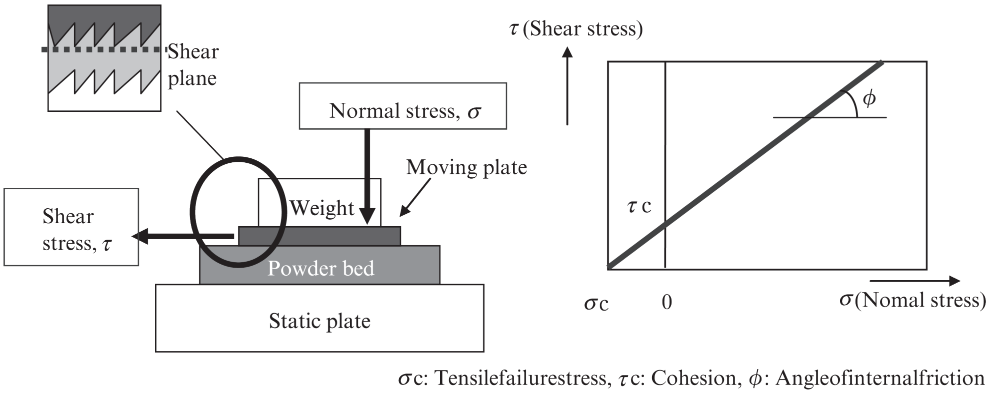

Fig. 3 Shear test of powder bed

placed on the powder bed, and this moving plate is moved horizontally at the speed of 20 mm/min by pulling. The horizontal pulling force at this time is measured, and the maximum load τ at the start of moving is measured. If the total weight of the weight and the moving plate is σ, the relationship between τ and σ can be obtained by measuring τ under different σ. Here, using the two points $\sigma = 0.21$ and 0.59 kPa, the respective values of τ are measured. Cohesion was obtained from the ordinate intercepts of the straight line passing through these 2 points, and the angle of internal friction was obtained from its slope.

2.3 Variation of Weight during Continuous Compaction

Bush-shaped green parts with the outer diameter of 30 mm, inner diameter of 20 mm and height of 30 mm were formed by continuous compaction, and the variation of weight of the current product and Cleanmix™ ZERO was compared by measuring the weights of each green part. The target density of the green parts was 6.9 Mg/m^3. Approximately 10 pieces were pressed at the start of pressing, and the filling depth was adjusted while measuring their dimensions and weight. The press used in this experiment was a 1 000 kN mechanical press, as shown in **Fig. 4**. A strain gauge was attached to the side of the punch of the die, and the load applied to the punch during pressing and ejection was measured.

The compaction rate was 400 pieces per hour, and the total number of pieces pressed was 300. During this process, compaction was stopped for 5 min after pressing 100 pieces, and pressing was then stopped again for 20 min after pressing 200 pieces. The weights of the full number of obtained green parts were measured.

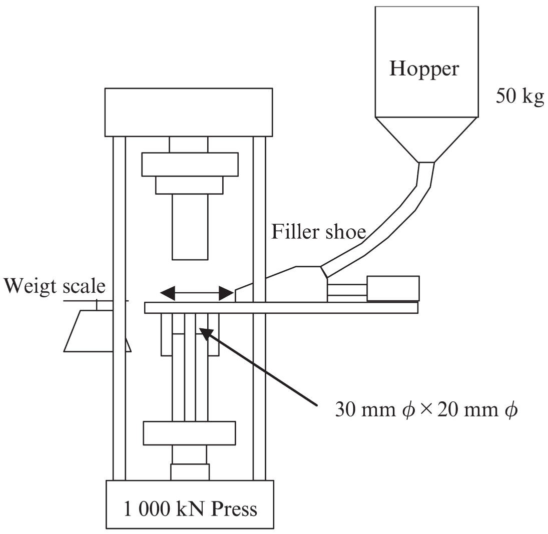

Fig. 4 Mass productive press

2.4 Filling Test

Using a cylindrical acryl cavity simulating a die for use with cylindrical parts, the filling behaviors of the respective powders were observed during reciprocal operation of a similar acryl filler shoe above the cavity. A schematic diagram of the powder filling test is shown in **Fig. 5**.

The cylindrical cavity was 50 mm in outer diameter, 40 mm in inner diameter and 60 mm in height. Eight partition plates were inserted in this cavity at equidistant intervals. At the bottom of the cavity, it was possible to open the bottom plate of each of the partitioned spaces (hereinafter, these spaces are referred to as “cells”), making it possible to measure the weight of the powder filled in each of the 8 cells.

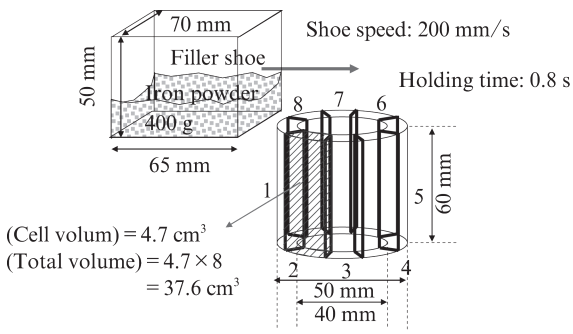

Fig. 5 Powder filling test to a bush cavity

The filler shoe was a rectangular parallelepiped 65 mm in length, 70 mm in width and 50 mm in height, and it was possible to open both the top and the bottom. The cavity was filled with the powder by reciprocal movement on an acryl plate and cavity simulating the die by charging 400 g of powder from the top using a 5 mmφ funnel.

In this test, the shoe speed was 200 mm/s, and the holding time directly above the cavity was 0.5 s. After 1 reciprocal movement, the weights of the powder filled in each of the cells in the cavity were measured. After the measurements were completed, new powder corresponding to the weight fed into the cavity was supplied from the funnel to the filler shoe so that the weight of the powder in the shoe was again 400 g, and the next measurement was performed. These measurements were repeated a total of 10 times.

In this experiment, the cell volume was 4.7 cm^3. The ratio of the weight of powder actually filled in the cells, and the theoretical full filling weight obtained by multiplying the volume of the cell by the apparent density of the powder was calculated, and this was defined as the filling rate, as shown in Eq. (1).

$$(\text{Filling rate}) = \frac{(\text{Filling weight})}{(\text{Apparent density}) \times (\text{Cell volume})} \quad \text{.... (1)}$$

3. Results and Discussion

3.1 Powder Properties of Mixed Powders

Table 1 shows the apparent density and flowabilty of the powders used in this study. It can be understood that the conventional mixes with EBS and zinc stearate have low apparent densities, and their flowability is also poor. In particular, the conventional mix with EBS did not flow in the 2.5 mm funnel. In contrast, with the Cleanmix™ powders, both the current product and Cleanmix ZERO have high apparent densities and small flowability. In particular, the flowability of Cleanmix ZERO was extremely small, at 21.3 s/50 g.

3.2 Index of Filling Characteristic of Mixed Powders

3.2.1 Hopper test[13–14)]

Table 1 also showed the results of hopper discharge, which is one index of the filling property. Here, the discharging property of the conventional mix with EBS was extremely poor, with 24 taps and a width of 8 mm. The flowability of the conventional mix with zinc stearate was also poor, but this mix showed a discharging property equal or superior to that of the current product, with 2 taps and a width of 29 mm. Cleanmix™ ZERO showed a satisfactory discharging property which could not be obtained with the other powders, requiring 0 taps and achieving a discharge width of 72 mm.

3.2.2 Adhesion of particles[15)]

Figure 6 shows the cohesion and angle of internal friction of Cleanmix™ ZERO. In comparison with the current product, the cohesion of Cleanmix ZERO is 1/2 or less. The angle of internal friction of Cleanmix ZERO is also somewhat smaller, but a particularly large difference was not observed. As the largest merit of Cleanmix ZERO is its small adhesion, material design was carried out to reduce adhesion.

The individual particles in a powder layer achieve stasis (i. e., reach the equilibrium state) as a result of the balancing of the interaction acting between particles, that is, balancing of adhesion, frictional force and gravity (buoyancy). In case large adhesion acts on the particles in comparison with gravity, flowability deteriorates due to agglomeration of the particles. However, because this agglomeration of the particles is unstable, agglomeration is avoided and flowability is recovered by relaxing the adhesion acting between the particles. Agglomeration is thought to occur during movement of a powder. Accordingly, in a continuous compaction operation, agglomeration of the powder occurs during transfer of the powder from the hopper to the filler shoe and during transfer accompanying the movement of the filler shoe, and the powder reaches a condition in which flow is difficult. Furthermore, when the compaction operation

Table 1 Powder properties

	Apparent density (Mg/m^3)	Flowability (s/50 g)	Discharge test from hopper	
			Tapping times	Width (mm)
Zinc stearate	3.23	33.9	2	29
Ethylene-bisstearamide	2.90	No flow	24	8
Current product	3.46	23.6	2	22
JIP™ Cleanmix™ ZERO	3.55	21.3	0	72

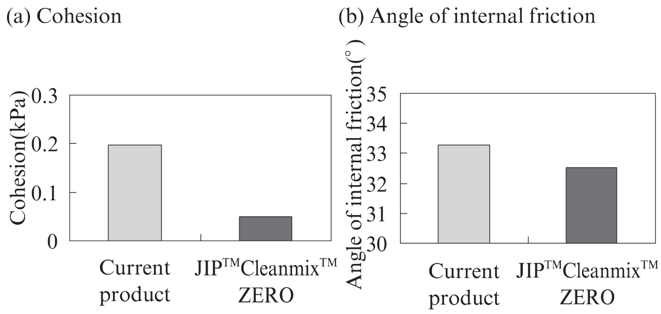

Fig. 6 Shear test of powder bed

stops, the powder is not transferred, agglomeration is broken up, and the flowability of the powder improves. For this reason, during the first compaction immediately after a stop, the flowability of the powder is good, the powder fills the cavity, and the weight becomes large. Movement of the powder becomes large with repeated compaction, and its flowability gradually deteriorates. Accompanying this, the weight of the green parts decreases, and at a certain point, the equilibrium state is reached.

Because the agglomeration force is large in powders with large adhesion, deterioration of flowability during continuous compaction is large. If compaction stops, flowability largely recovers, and the weight is large immediately after the operation is restarted. On the other hand, with low adhesion powders, it is considered that deterioration of flowability during continuous compaction is small, and accordingly, the increase in weight when compaction is resumed is also small.

3.3 Variation of Weight during Continuous Compaction

Figure 7 shows the variation of weight in continuous compaction of 300 bush-shaped parts with the outer diameter of 30 mmφ and height of 30 mm using the current product and Cleanmix™ ZERO. In both cases, large weight increases occur at 2 points. These show the weights of the 101st and 201st pieces, when the press was stopped for 5 min and for 20 min after compaction of 100 parts and 200 parts, respectively, after which press compaction was restarted. Although the weight increase after restarting is large, the weight gradually decreases and returns to approximately the average value of the weight of all pieces at the 3rd to 5th piece after operation resumes. In comparison with the current product, it can be understood that the weight increase rate is smaller with Cleanmix ZERO.

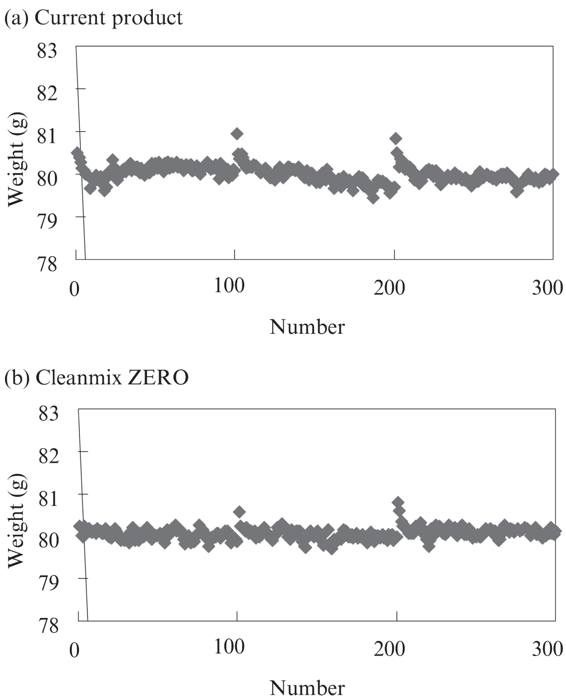

Fig. 7 Weight of green parts

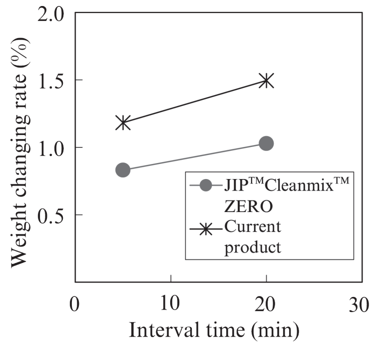

Fig. 8 Relation of weight change rate with interval time

Figure 8 shows the relationship of the weight change rate before and after press stops with the press stop interval time. With both the current product and Cleanmix ZERO, the weight change rate increases accompanying longer interval times. Moreover, the weight change rate of Cleanmix ZERO is approximately 60% that of the current product.

The fact that the weight change rate is smaller is the most important feature of Cleanmix ZERO. As the reason for this, as described previously, it is thought that changes in flowability over time are small because the adhesion between the particles is small.

3.4 Filling Test

In the continuous compaction in Section 3.3, no large difference was observed in the variation of weight of the individual green parts of the current product and Cleanmix™ ZERO. Here, a test simulating filling of powder from a filler box into a cavity during compaction was performed, and the difference in the filling behaviors of the current product and Cleanmix ZERO was investigated.

Figure 9 shows the filling rates of Cleanmix ZERO,

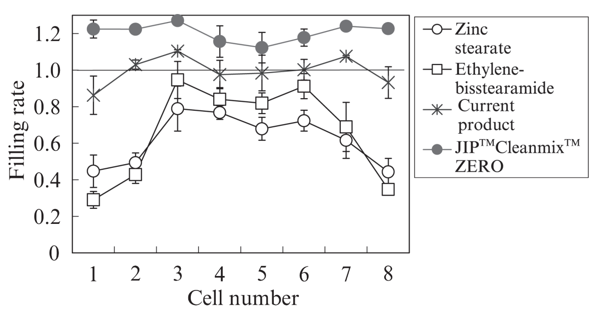

Fig. 9 Filling rate of mixed powders to bush cavity

the current product, a conventional mix of zinc stearate (hereinafter, ZnSt) and a conventional mix of EBS in the cells of a cylindrically-shaped cavity which was divided into 8 cells. In comparison with the other mixed powders, the filling rates of the two conventional powders (ZnSt, EBS) were poor. Furthermore, there were also large differences in the filling rate depending on the cell position. In all cases, the filling rates were low at positions 1, 2, and 8 and high at positions 3–6.

The filler shoe moves from the direction of 1 to the direction of 5, stops for 0.5 s above the cavity, and then moves in the reverse direction, returning to 1. For this reason, the time when the powder passes above position 1 is inherently long. Conversely, the time when the powder passes position 5 is the shortest. Accordingly, assuming the powder is dropped into the cavity from the shoe at a constant rate, the filling rate is highest at position 1 and lowest at position 5. However, the filling rates of the conventional powders showed the opposite tendency. This is thought to be because the flowability and filling property of these powders are poor, and the powder drops into the cavity due to the impact when the filler shoe stops.

In contrast to this, the filling rate of the current product is high overall, and differences in the filling rate depending on position are small. This is considered to be the result of improvement of the apparent density and flowability of the powder by the Cleanmix technology.

Cleanmix ZERO displays an even higher filling rate than the current product, and differences depending on position are also smaller. Further, its filling rate achieves a value exceeding 1. This is thought to be because the filling density when the powder is filled from the filler shoe into the cavity at one time is large in comparison with the apparent density, in which filling is performed while flowing the powder using the funnel described in JIS Z 2504.

The error bars in Fig. 9 shows the standard deviation of the filling rate in each cell when filling was repeated 10 times. The average value and the standard deviation of the filling rate of the entire cylindrical cavity (total of all cells) were 1.0 and 0.03 for the current product and 1.21 and 0.01 for Cleanmix ZERO, respectively. The standard deviation of the filling rate of Cleanmix ZERO was small in comparison with that of the current product. This fact shows that the variations in density within a part are small, and at the same time, the variation of weight in filling (compaction) of the individual parts is also small. Thus, this experiment clarified the difference between the filling properties of the current product and Cleanmix ZERO.

In the continuous compaction experiment in this study, the difference in the variation of weight of the current product and Cleanmix ZERO could not be clarified. This may be due to the fact that the volume ratio of the filler shoe and cavity ((Volume of filler shoe) / (Volume of cavity)) was approximately 70 in continuous compaction, but was small in the filling experiment, being approximately 6; as a result, the effect of replacement (transfer) of powder described previously was small in continuous compaction, and the difference in variation of weight was also less remarkable by a corresponding amount. It is thought that the variation of weight of individual parts during continuous compaction can also be reduced in comparison with the current product.

4. Conclusion

In order to investigate the powder properties and variation of weight during compaction of Cleanmix™ ZERO for high density compaction, its behavior was observed from various angles, and the following new knowledge was obtained.

(1) Regarding powder properties, Cleanmix ZERO has large apparent density and excellent flowability.
(2) In the hopper test, which is an index of the filling characteristic, Cleanmix ZERO displayed an extremely high discharging property and the width of the aperture after discharging was also large, suggesting that this product has an excellent filling property.
(3) In the powder layer shear examination, Cleanmix ZERO displayed small adhesion. Based on this, Cleanmix ZERO is considered to show a high discharging property and filling property.
(4) The weight change rate of Cleanmix ZERO after compaction stop and restart in continuous compaction was also found to be small in comparison with the conventional Cleanmix product.
(5) The high filling property of Cleanmix ZERO could also be confirmed in a filling test, and it was suggested that the internal density distribution within one part could also be reduced.

By using Cleanmix ZERO, it considered possible not only to reduce variations of weight between green parts during compaction, but also to reduce the internal density distribution in individual parts. Taking advantage of this feature, application of Cleanmix ZERO to mass production of hard-to-form parts, which could not be mass-produced in the past because the powder did not fill the die uniformly, is expected.

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