KAWASAKI STEEL TECHNICAL REPORT
No.42 (May 2000)

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Satoshi Uenosono, Hiroshi Sugihara, Kuniaki Ogura

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1 Introduction

In the field of iron powder metallurgy, high dimensional accuracy in sintered parts is an essential requirement.[1] On the other hand, high quality is also required in the raw material powder, because powder metallurgy technology competes in quality and economy with other machining methods.

Alloy powders such as copper powder, nickel powder, graphite powder, and others, and lubricants are generally mixed with iron powder, as the raw materials for iron powder sintered materials, and are sintered in iron powder metallurgy. Because the specific gravities of the various types of powder which comprise this mixed powder differ greatly, the alloying elements are easy to segregate between mixing and the compacting process, resulting in problems such as deviations in quality and dusting, in the manufacturing process for sintered parts.

Kawasaki Steel developed the premixed powder which prevents segregation of graphite by fixing the graphite or other alloying powder to the surface of the iron powder with an organic binder, in order to solve such problems.[2]

Zinc stearate is normally used as lubricant, in conventional segregation-free premixed powder. However, zinc stearate is decomposed to zinc oxide during sintering process, which adheres to the sintering furnace and also causes sooting on the surface of the sintered parts. For this reason, segregation-free premixed powders in which part of the zinc stearate is replaced with a wax, that does not contain zinc stearate, are also used. However, this type premix powder has bad flowability and, when a tube conveyor is used in transportation, the lubricant tends to stick to the transport coil in the conveyor. Sticking of the lubricant cause, the load on the conveyor motor to become excessive, resulting in a conveyor stop in worst case. Moreover, large pieces of lubricant which may separate from the coil, and be mixed into the sintered parts cause defects in sintered parts. Therefore, the development of a segregation-free premixed iron powder with a wax lubricant, which would offer improve flowability and eliminate the problem of adhesion of the lubricant to the inner parts of the tube conveyor during transportation, had been desired.

Ishikawa et al. conducted shearing tests of segregation-free premixed iron powders with wax lubricants,

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which revealed that flowability does not depend on the internal friction force, but on the adhesive force.[3] It is known that the adhesive force is made up of the liquid bridge force, electrostatic force, and van der Waals force.[4]

In this report, the liquid bridge force, electrostatic force, and van der Waals force of commercially available segregation-free premixed iron powder with wax lubricants were calculated. The factors which determine flowability were estimated. Further, the adhesive force was actually measured and compared with the calculated results.

Based on this knowledge, a new product (trade name KIP CLEANMIX KWAX-B) was developed. The new product offers powder characteristics and mechanical properties on the same level as the conventional segregation-free premixed iron powder with wax lubricant, together with significantly improved flowability. Additionally KWAX-B can eliminate lubricant sticking during transportation by the tube conveyor, since the start of production and sales in 1997, KWAX-B has earned an excellent evaluation from customers. This report describes the results of an analysis of the factors which dominate the flowability of premixed powder using wax type lubricants and the features of KIP CLEANMIX (KWAX-B).

2 Experimental Procedure

2.1 Analysis of Factors Determinating Flowability of Premixed Iron Powder with Wax Lubricant Type

A commercially available segregation-free premixed iron powder with wax lubricant (wax type KIP CLEANMIX manufactured by Kawasaki Steel; KWAX-A, composition: Fe-0.8% graphite powder-0.75% lubricant) was used in these experiments. The degree of adhesion of the graphite was measured as the ratio of the analysis value of C with a size of 75–150 μm in the segregation-free premixed powder to that of the total C content in the powder.[1] In order to observe the shape of particles, the segregation-free premixed powder, the iron powder, graphite powder, and lubricant used were observed with SEM.

The mean particle size of the iron powder was measured using a sieve; that of the other powders was measured by the micro track method. In order to calculate the electrostatic force, the charges of a mixed powder of iron powder and 1% lubricant and a mixed powder of iron powder and 0.8% graphite powder were measured by the blow off method.[5] Because the measured charge was expressed in units of C/g, the specific surface area (m^2/g) of each powder was measured by the BET method, and the results were used to convert the charge to a unit of C/m^2. To evaluate the humidity range in which the liquid force must be considered, the adsorption isotherm of water vapor of the segregation-free premixed iron powder with wax lubricant was measured at 25°C.

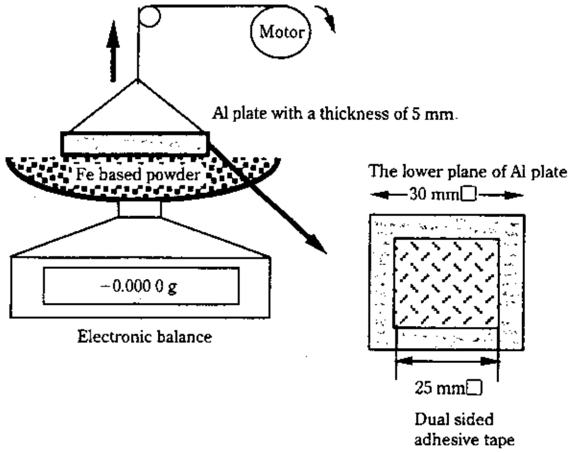


Fig. 1 Schematic description of the system to measure the van der Walls force of iron based powders

The adhesive force of the powder was obtained, referring to the methods proposed by Fukuzawa and Kimura.[6] Powder which had been filled was set on an electronic balance, and an aluminum plate, to which dual sided adhesive tape had been fixed, was placed on the surface of the powder bed from above (**Fig. 1**). Then the maximum weight change shown by the electronic balance when the plate was gradually raised above was measured. The number of particles which adhered to the dual sided tape was obtained by optical micrographs. Adhesive force was calculated as follows; {(maxmum weight change) − (the weight of powder adhered to the dual side tape)}/(the number of particle size).

2.2 Evaluation of Characteristics of KIP CLEANWAX (KWAX-B)

Segregation-free premixed iron powders with a composition of Fe-2%Cu-0.8%C were prepared. Water atomized iron powder (KIP301A), electrolytic copper powder (mean particle size; 32 μm) and natural graphite powder (mean particle size; 24 μm) were used as raw materials. Three types of lubricant were used in an amount of 0.8%, respectively; the zinc stearate type, the conventional wax type lubricant (KWAX-A) and the newly developed wax type lubricant (KWAX-B).

Apparent density, flowability, index of flow blocking (flowability from the hopper), green density at compacting pressures of 392, 490, and 588 MPa, Rattler value, and ejection force were also measured. The index of flow blocking was measured as follows; a simple hopper with a size of 100 mm × 100 mm × 100 mm and a cut hole in diameter of 2.5 mm in the bottom was filled with 1 kg of segregation-free premixed iron powder. Gentle vibration was then applied to the top surface of the sim-

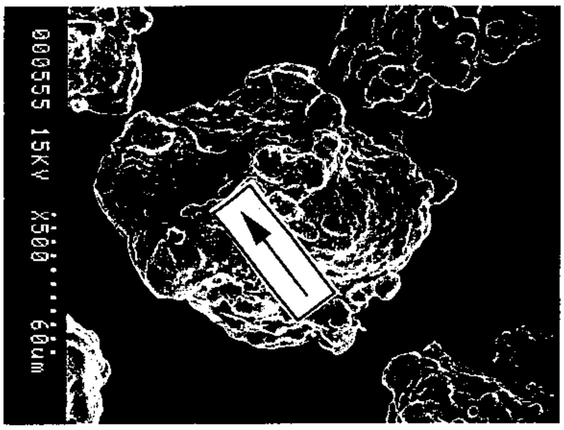


Photo 1 SEM image of the segregation-free iron based powder used

ple hopper, and the number of vibration cycles until the mixed powder was discharged was used as the index of flow blocking.

To observe the condition of lubricant adhesion during transportation, 1 t of segregation-free premixed iron powder was transported at a rate of 20 kg/min using a tube conveyor (manufactured by Nihon Kosan Corp.; TS-05-7AB). After repeating this procedure 10 times, the conveyor was dismantled and the condition of lubricant adhesion to the conveyor coil was observed.

The respective segregation-free premixed iron powders were compacted to a green density of 6.85 Mg/m^3, and were then sintered at 1 403 K for 20 min in an RX gas atmosphere. After sintering, the tensile strength, Charpy impact values, and dimensional change during sintering (change relative to the mold as standard) were measured.

3 Results and Discussion

3.1 Factors Determining Flowability of Premixed Iron Powder with Wax Type Lubricant

3.1.1 SEM observation of segregation-free premixed iron powder with wax lubricant (KWAX-A)

A SEM image of the segregation-free premixed iron powder is shown in **Photo 1**. The surface of the iron powder has a roughness on the order of 10 μm, and a binder component with a size of approximately 20 μm has adhered to a concave area, as shown by the arrow in the center of the photograph. The graphite adhesion ratio is 80%, and it is considered that virtually all the graphite particles have adhered on the surface of the iron powder by being covered with this binder. The lubricant was added as a free powder. These results reveales that the segregation-free premixed iron powder is considered to have a structure shown in **Fig. 2**. **Table 1** shows the mean particle size, the results of measurements of the specific area, and the Hamaker coefficients used in calculations of the powders which comprise the segregation-free premixed iron powder.7) Here, the Hamaker coefficients of the lubricant and binder were not actually measured. The organic substances measured were only ethyleneglycol and polystyrene, and the value of the latter was adopted in this report.

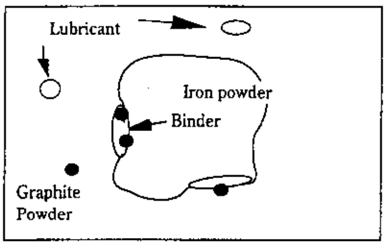


Fig. 2 Schematic description of the segregation-free iron based powder used

Table 1 Hamaker coefficients, mean particle sizes and specific surface areas of iron powder, graphite powder and lubricant powder

Material	Hamaker coefficient (10^{-19} J)	Mean particle size (μm)	Specific surface area (m^2/g)
Iron powder	2.12	80	0.04
Graphite podwer	4.70	22	9.18
Lubricant powder	0.70	20	7.50

3.1.2 Calculated results

(1) Liquid Bridge Force

The liquid bridge force is the force caused by the surface tension due to the adsorbed water film between particles. Assuming the contact angle between the liquid and the particle is 0, the size of the liquid bridge is sufficiently small in comparison with the particle diameter, and the liquid bridge force can be expressed by the following equation.8)

$$F_c = -\pi \cdot \gamma \cdot D \cdots\cdots\cdots\cdots (1)$$

Here, γ: surface tension of water (N/m), D: converted particle diameter of 2 particles (m). The converted particle diameter is given by the following equation.

$$D = (D_1 \cdot D_2)/(D_1 + D_2) \cdots\cdots\cdots\cdots (2)$$

Here, D_1, D_2 are the diameters (m) of the respective particles. From Eq. (1), the liquid bridge force is proportional to the particle diameter, and was therefore calculated between the iron powder particles, which are the largest of the particles comprising the segregation-free premixed iron powder. As a result, the liquid

Table 2 Electrostatic quantity measured and electrostatic force calculated

Combination of powder		Charge (μC/g)	Electrostatic force (10^{-12} N/particle)
Iron powder	Graphite powder	0.024	0.42
Iron powder	Lubricant powder	−0.084	2.20

bridge force was calculated at 1.8×10^{-5} N/particle as a largest number.

(2) Electrostatic Force

Electrostatic forces are generated by contact among different kinds of particles; the iron powder, alloy powders, lubricant, and other heterogeneous materials which comprise the segregation-free premixed iron powder. The electrostatic force is expressed by the following equation:[8]

$$F_c = \pi \cdot \sigma_1 \cdot \sigma_2 \cdot D_2/\varepsilon \cdots\cdots\cdots\cdots (3)$$

where, σ; charge (C/m^2), D; converted particle size (m), ε; dielectric coefficient of a vacuum (F·m^{-1}). Here, σ_1, σ_2 are the charges of respective particles. For example, **Table 2** shows that, in a combination of iron powder and graphite powder, the iron powder has a charge of $-0.024\,\mu$C/g, and the graphite, $+0.024\,\mu$C/g. However, because it was not possible to extract the binder components alone form from premixed powder used, it was not possible to measure the charge between the binder and the lubricant. Thus, Table 2 summarizes the measured values of the electrostatic charge and those of the electrostatic force. The electrostatic force is in the range of 10^{-13}–10^{-12} N per a particle.

(3) van der Waals Force

The van der Waals force is due to the orientation effect caused by intra-molecules having a permanent dipole moment, and the force generated when molecules with a permanent dipolar moment polarize molecules without such a moment. The van der Waals force is given by the following equation:[8]

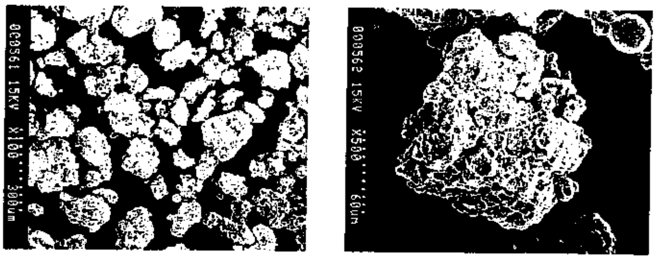
$$F_{vb} = -A \cdot D/\{24(Z+b)^2\} \cdots\cdots\cdots\cdots (4)$$

Here, A: Hamaker coefficient (J), D: converted particle size (m), Z: distance between particles (40 nm), b: surface roughness of particles (m). The Hamaker coefficient and surface roughness of two kinds of particles are expressed by the following equations:

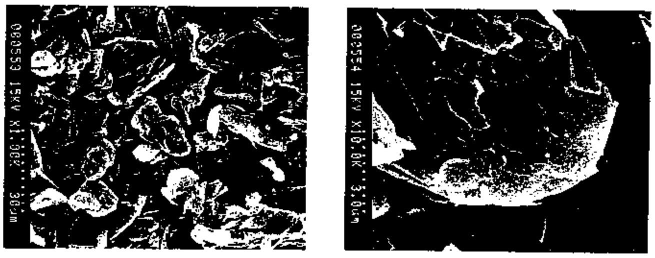
$$A = (A_1 \cdot A_2)^{0.5} \cdots\cdots\cdots\cdots (5)$$

$$b = (b_1 + b_2)/2 \cdots\cdots\cdots\cdots (6)$$

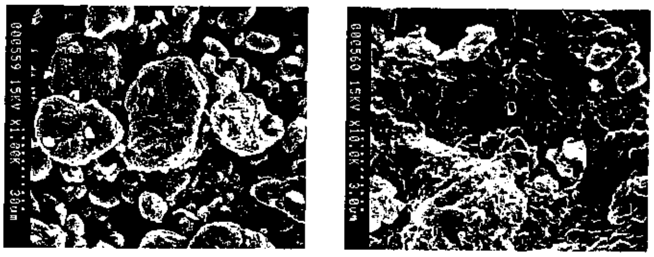
Here, A_1, A_2 are the Hamaker coefficients of the respective particles, and b_1, b_2 are the surface roughnesses of the particles. In this work, the van der Waals force was calculated between two kinds of particles in various combinations of the metal powders and lubricants that comprise the segregation-free premixed iron powder.



Iron powder



Graphite powder



Lubricant powder

Photo 2 SEM images of iron powder, graphite powder and lubricant powder

Photo 2 shows SEM images of the powders used in this experiment. The iron powder is composed of primary particles with a particle size of 10–20 μm, and the magnitude of the surface roughness is approximately 10 μm. The graphite powder is a flat shaped powder with a diameter of 20 μm. Because its surface was comprised of cleavage planes which seemed to have been formed during milling, the surface roughness was assumed to be 0 in these calculations. The outer surfaces of the lubricant and binder were smooth, as shown in Photos 1 and 2, and the van der Waals force was calculated in the range of 1.0-0.001% by the ratio of the surface roughness to the diameter (b/D).

Table 3 shows a summary of the van der Waals forces of the iron powder and respective particles. The van der Waals force between the iron powder and respective particles was on the order of 10^{-15} N/particle. **Figure 3** shows the effect of the ratio of the surface roughness and diameter of the particles on van der Waals force between the binder and lubricant and

Table 3 van der Waals force calculated

Combination of powder		van der Waals force (10^{-15} N/particle)
Iron powder	Graphite powder	9.07
Iron powder	Lubricant powder	3.25

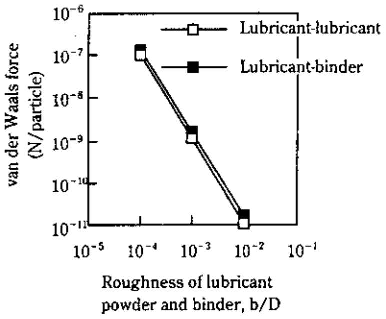


Fig. 3 Effect of roughness of lubricant powder and binder on van der Waals force between lubricants and that between lubricant powder and binder on iron powder

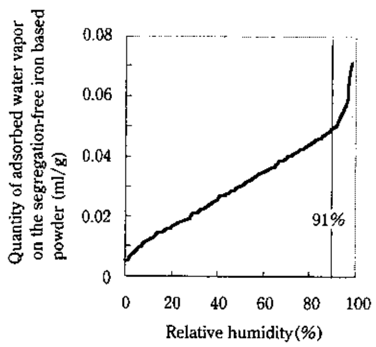


Fig. 4 Adsorption isotherm of water vapor on segregation-free iron based powder used

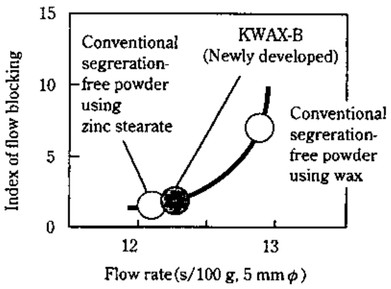


Fig. 5 Flowability of newly developed segregation-free iron based powder with wax lubricant compared with those of conventional segregation-free iron based powders

between lubricant particles. The van der Waals force increased as the surface of the lubricant and/or binder became smoother, and was in the range of 10^{-11} to 10^{-7} N per particle. These calculated results indicated the largest van der Waals forces act between the binder and lubricant and between particles of the lubricant.

3.1.3 Forces determining flowability

The calculated results in section 3.1.2, shows the forces comprising the adhesive force become smaller in the order of, liquid bridge force > van der Waals force > electrostatic force. The liquid bridge force occurs in a condition in which water molecules are adsorbed in multiple layers on the surface of particles (multimolecule adsorption). **Figure 4** shows the adsorption isotherm of water vapor of the segregation-free premixed iron powder with wax lubricant. This figure indicated that mono molecular layered water is adsored up to a relative humidity of 91%, whereas in a range of greater than 91%, multi molecule layered water is adsored. From this result, the liquid bridge force can be ignored under the working conditions which exist in mixing and compacting in ordinary powder metallurgy. Accordingly, the van der Waals force between the binder and lubricant and between particles of the lubricant is concluded to be the main factor determining flowability.

The measured value of the adhesive force of the segregation-free premixed iron powder with wax lubricant was 10.9×10^{-8} N/particle, and thus is in fairly good agreement with the calculated value of the van der Waals force. The segregation-free premixed iron powder with zinc stearate type, which is superior in flowability to the wax lubricant type, had a lower value than the wax lubricant type, at 3.2×10^{-8} N/particle. This is attributed to the fact that, although a constant value of the Hamaker coefficient of the lubricant was used in these calculations regardless of the kinds of lubricant, these values are actually different depending on the lubricant.

These results indicate that reducing the van der Waals force between the binder and the lubricant and between particles of the lubricant is effective in improving the flowability of segregation-free premixed iron powders.

3.2 Characteristics of KIP CLEANMIX (KWAX-B)

A new lubricant which reduces the van der Waals force, determining flowability, and new segregation-free iron powder; KIP CLEANMIX (KWAX-B), which simultaneously not only has high flowability but also eliminates the problem of lubricant sticking in the tube conveyor, was newly developed. The features of this new product are described below.

3.2.1 Powder characteristics

Figure 5 shows the relationship between the flowability (flow rate) and index of flow blocking (of segregation-free premixed iron powders); **Fig. 6** shows the

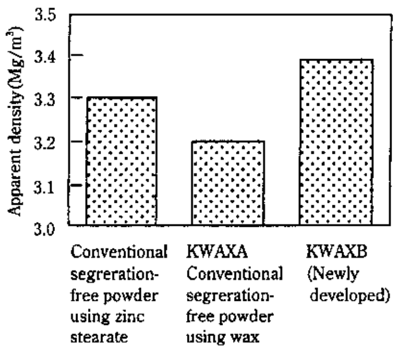


Fig. 6 Apparent density of the segregation-free iron based powder used

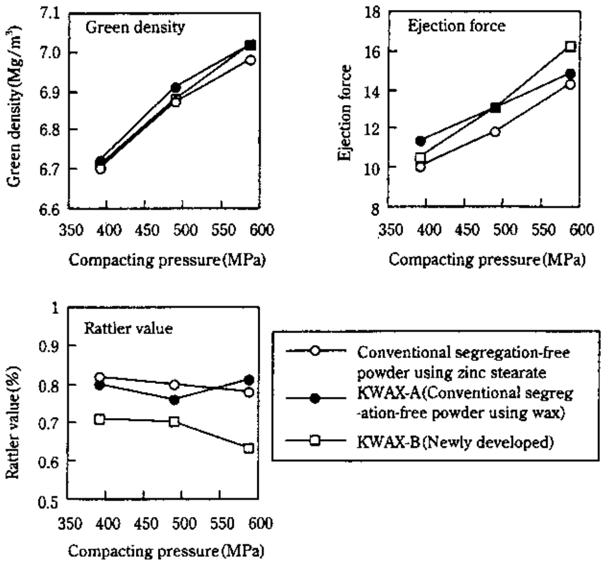


Fig. 7 Green density, ejection force and Rattler value of the segregation-free iron based powder used

apparent density. The flowability and index of flow blocking of the newly developed wax type KIP CLEANMIX (KWAX-B) have been greatly improved in comparison with the conventional product, wax type KIP CLEANMIX (KWAX-A), and values are close to those of zinc stearate type segregation-free premixed powder. Further, the apparent density of the new product increased by approximately 0.2 Mg/m^3 compared with that of the conventional wax type KIP CLEANMIX (KWAX-A)

The measured adhesive force of the wax type KIP CLEANMIX (KWAX-B) was 3.9×10^{-8} N/particle. This value is approximately 60% smaller than that of the conventional wax type KIP CLEANMIX (KWAX-A), as mentioned in section 3.1.3. This is maybe main reason to improve flowability.

Figure 7 shows the relationship between the compacting pressure and the green density, ejection force, and Rattler value. The newly developed product, wax type KIP CLEANMIX (KWAX-B), shows substantially the same compressibility, ejection force, and Rattler value as zinc stearate type segregation-free premixed iron powder and the conventional product, wax type KIP CLEANMIX (KWAX-A).

A mixture of lubricant and graphite adhered to the coil inside the conveyor, when the conventional KIP CLEANMIX (KWAX-A) segregation-free premixed iron powder was transported by a tube conveyor. In contrast, no lubricant was observed to stick with the zinc stearate type segregation-free premixed iron powder or with the newly developed wax type segregation-free premixed iron powder, KIP CLEANMIX (KWAX-B).

As described above, the lubricant does not adhere to the tube conveyor, and flowability and the index of flow blocking are both superior to those of the conventional product, KIP CLEANMIX (KWAX-A), with the newly developed wax type segregation-free premixed iron powder, KIP CLEANMIX (KWAX-B). Moreover, the newly developed product shows virtually the same compressibility, ejection force, and rattler value as the conventional product.

Table 4 Tensile strength and Charpy impact value of sintered body made of the segregation-free iron based powder used and dimentional change during sintering

	Tensile strength (MPa)	Impact value (J)	Dimentional change (%)
Conventional segreration-free powder using zinc stearate	445	10	0.34
KWAX-A(Conventional segreration-free powder using wax)	422	10	0.38
KWAX-B(Newly developed)	430	11	0.38

3.2.2 Mechanical properties

Table 4 shows the tensile strength, Charpy impact value, and dimensional change during sintering of sintered steel made from the segregation-free premixed iron powders. The newly developed wax type KIP CLEANMIX (KWAX-B) shows substantially the same tensile strength and Charpy impact value as zinc stearate type segregation-free premixed iron powder and the conventional product, wax type KIP CLEANMIX (KWAX-A). The dimensional change of sintered steel made from the newly developed wax type KIP CLEANMIX (KWAX-B) is as same as that made from the conventional product, KIP CLEANMIX (KWAX-A). And the sintered steel tends to slightly swell by approximately 0.04% in comparison with the zinc stearate type segregation-free premixed iron powder.

4 Conclusion

(1) The van der Waals forces which act between the binder and lubricant at the iron powder surface and between particles of the lubricant are thought to determine the flowability of wax type segregation-free premixed iron powders.

(2) The newly developed wax type segregation-free premixed iron powder, KIP CLEANMIX (KWAX-B), is superior in flowability (flow rate) and the index of flow blocking (flowability from the hopper) in comparison with the conventional product, KIP CLEANMIX (KWAX-A). The new product shows substantially the same level of compressibility, ejection force, and Rattler values as the conventional product.

(3) With the newly developed wax type segregation-free premixed iron powder, KIP CLEANMIX (KWAX-B), the lubricant does not adhere to the inner parts of the conveyor during transportation with the tube conveyor.

(4) The newly developed wax type, KIP CLEANMIX (KWAX-B) gives virtually the same tensile strength, Charpy impact value, and dimensional change of sintered steel after sintering as zinc stearate type segregation-free premixed iron powder and the conventional product, wax type KIP CLEANMIX (KWAX-A).

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