Newly Developed Iron-based Powder Mixture, High-density SEGLESS®, for High Density Compaction

Hironori SUZUKI[*1], Satoshi NISHIDA[*2], Takayasu FUJIURA[*3]
[*1] Takasago Steel Powder Plant, Steel Powder Division, Iron & Steel Business
[*2] Steel Powder Sales Department, Steel Powder Division, Iron & Steel Business
[*3] Research & Development Planning Department, Technical Development Group

The newly-developed lubricant Kobelco Polyhydroxyl Amide (KPA) consists of two different functional materials. Based on this feature, an iron powder mixture with KPA lubricant has better powder flow at the powder stage and better ejection performance at the green stage. Since KPA lubricity is superior to that of conventional lubricants, it is possible to reduce lubricant content without increasing the ejection pressure. By utilizing this process, high-density powder metallurgical parts can be made at a production cost relatively lower than that of conventional methods.

Introduction

Iron-based sintered parts are produced by a powder metallurgy process which comprises steps of mixing iron powder with additives, such as alloying powder and lubricant, compacting the mixture and sintering the compact (**Fig. 1**). It is a low-cost high-volume process for manufacturing complex-shaped products. Various efforts have been made to increase the strength of iron-based sintered parts, particularly for automotive engines, in response to the recent need for reducing automotive weight and enhancing engine performance. Among them, techniques such as warm compaction and die-wall lubricated compaction, which increase strength by decreasing porosity and thus densifying the sintered bodies, are effective because they are widely applicable to various sintered parts. These compaction techniques, however, require special ancillary equipment, such as heating and lubricant spray apparatuses. Thus, there has been a need for a more convenient method of high density compaction.

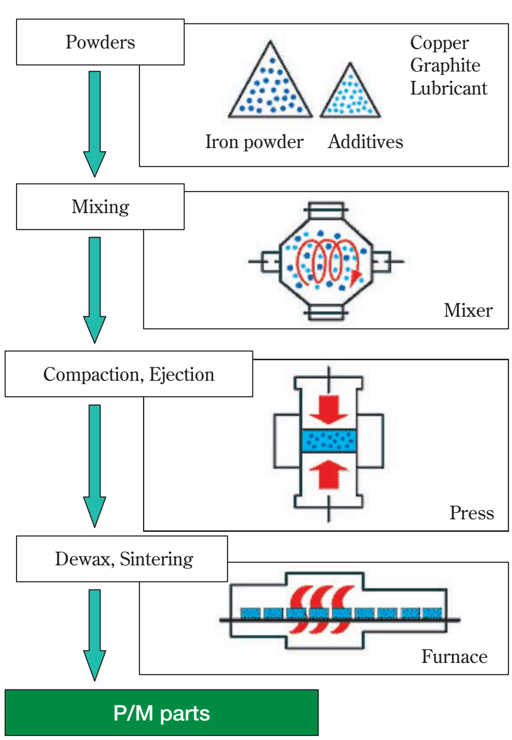

Fig. 1 Production process of powder metallurgy

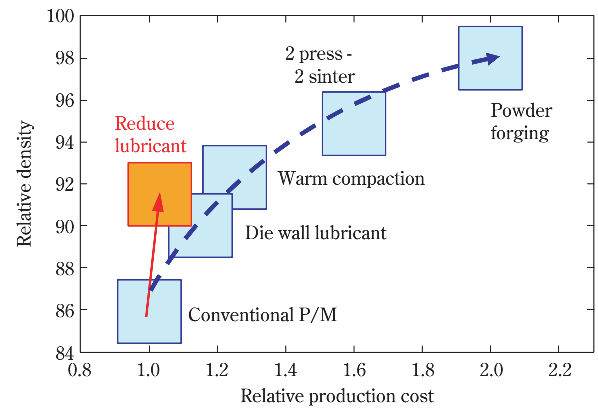

Fig. 2 Production cost and typical density for various high density processes

Fig. 2 depicts the relationship between the relative production costs and typical densities achieved by various powder metallurgical processes. Conventional high-density processes such as warm compaction, die-wall lubricated compaction and powder forging tend to cost more as the product density increases. Kobe Steel has found that a functional lubricant can decrease the amount of internal lubricant additive and achieve high density at relatively low cost without changing the existing equipment. This paper introduces the features of the functional lubricant, which exhibits excellent mold releasability. Also introduced is an iron-based powder mixture, containing the functional lubricant and designed for high-density compaction.

1. Role of lubricant in powder metallurgy

A lubricant for powder metallurgy is used for reducing frictional forces among powder particles, and between a compact and mold, particularly at the time of the powder compaction and releasing of the compact from the mold. Internal lubrication is a common approach for using lubricant, in which metal soap, such as zinc stearate (hereinafter referred to as "Zn-st"), and/or wax resin, such as ethylene bisstearamide (hereinafter referred to as "EBS") are/is added in powder form to the base powder. **Fig. 3** shows the relationship between lubricant content and the theoretical maximum density achieved by a commonly-used composition consisting of 2% copper powder and 0.8% graphite powder with the balance of iron powder, and also the same relationship found in the case of iron powder alone. This clearly shows that increasing the amount of lubricant significantly decreases the density. It should be noted that the lubricant becomes useless once the compaction is completed and must be removed from the powder compact. Thus, a smaller amount of additive is preferable. To ensure lubricity, lubricant is typically added to iron powder in the amount of about 0.75 wt. percent.

Lubricant is known to significantly affect the flowability of the mixture consisting of iron powder and lubricant. It also affects the mold releasability, or the ejection force required to eject the compact of the powder mixture from a mold after compaction. The flowability is evaluated by a unique Kobe Steel method and is represented by the minimum bore diameter, known as the "critical discharging diameter," which allows the discharge of subject powder from a test container. After the comprehensive evaluation of a variety of commercially available lubricants, flowability and mold releasability have been found to be in a trade-off relation, as shown in **Fig.4** [1].

A lubricant with a low melting point accumulates easily on the wall surfaces of a mold and facilitates the release of compacts from the mold after compaction; however, it adheres strongly to iron powder during the powder mixing and may cause problems such as agglomeration and decreased flowability. A lubricant with a high melting point, on the other hand, remains solid during the powder mixing and exhibits excellent flowability; however, it does not accumulate sufficiently on the wall surface and may exhibit poor mold releasability. Thus, conventional lubricants have been unable to satisfy both flowability and mold releasability at the same time.

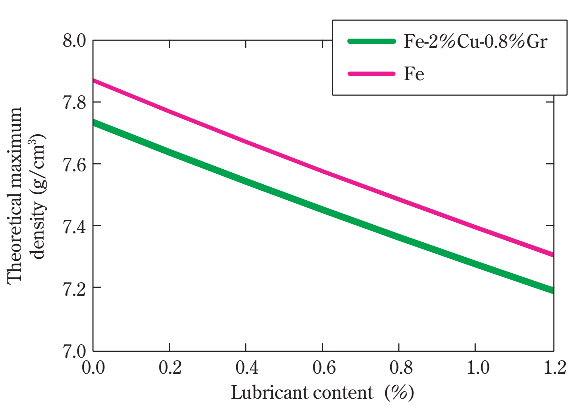

Fig. 3 Effect of lubricant content on theoretical maximum density

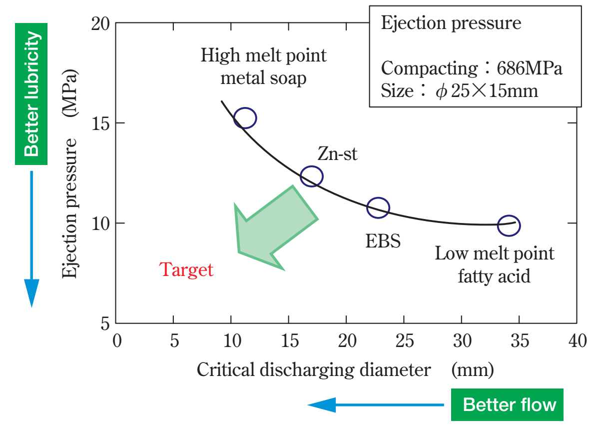

Fig. 4 Relation between flowability and ejection pressure for various lubricant mixture [1]

Targeting the development of a lubricant that can achieve both flowability and mold releasability, as shown by the arrow in Fig. 4, Kobe Steel has newly formulated a functional lubricant, Kobelco Polyhydroxyl Amide (hereinafter referred to as "KPA"), that selectively exerts the functions of assuring flowability during the powder mixing step and satisfying mold releasability at the time of compact ejection.

2. Concept of functional lubricant, KPA

KPA comprises two types of lubricants, both polyhydroxylamides, but having different melting points[2] (**Fig. 5**). A lubricant with a high melting point (no lower than 150℃), which contributes to flowability, is mixed with another lubricant with a low melting point (no higher than 100℃), which contributes to mold releasability, at an optimum ratio. These materials are also optimized for their qualities, such as particle size, to improve both characteristics, flowability and mold releasability. It should be noted that KPA is a wax-type lubricant consisting only of organic materials, which decrease the contamination of the sintering furnace and leave less smearing on the surfaces of sintered bodies.

A mixture of iron powder and KPA was compacted to evaluate dewaxing ability. The powder compact was heated in a thermobalance to measure its weight loss during heating (**Fig. 6**). The result indicates that the lubricant decomposes and gasifies as the temperature rises and is fully removed from

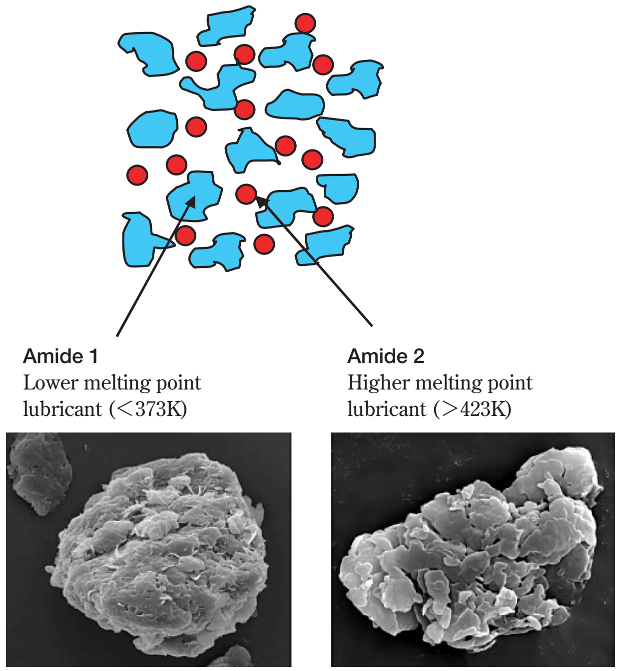

Fig. 5 Conceptual diagram of functional lubricant KPA

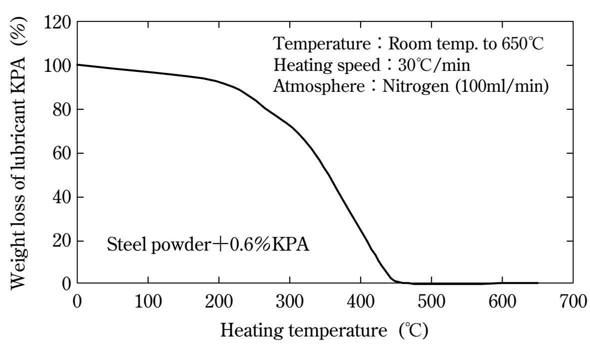

Fig. 6 Weight loss of KPA lubricant during heating

the powder compact in nitrogen atmosphere as the temperature approaches 450℃. Almost no residue of lubricant was found after the dewaxing.

3. Characteristics of KPA

3.1 Basic properties of powder mixture containing KPA[3), 4)]

Three different mixtures were prepared, each consisting of Kobe Steel's atomized iron powder, 300M (average particle size, 70μm), 2% of copper powder (average particle size about 30μm), and 0.8% of graphite powder (average particle size 5μm), plus 0.75% of one of three lubricants, i.e., the newly developed KPA, Zn-st (average particle size 10μm) and EBS (average particle size 25μm). The latter two lubricants, both commercially available and added in the same amount, were used as reference materials. These compositions, called "SEGLESS", were designed to prevent the segregation of graphite.

Table 1 summarizes the typical properties of these three powder mixtures, as well as those of their green compacts. The apparent densities and flow rates were measured according to JIS 2504 and JIS 2502, respectively. Each powder mixture was compacted into a cuboid (31.8mm×12.7mm×height 6.3mm) with a compacting pressure of 490MPa. The green density was measured on each powder compact. Also measured was its three-point bending strength according to JIS Z2511. The mold releasability was evaluated by the ejection force (peak value) required to release each compact of the powder mixture from the mold. The compacts were made into solid cylinders (ϕ25×25mm, ea.) under three different levels of compacting pressures; i.e., 490, 588 and 686MPa. **Fig. 7** shows the results of the releasability evaluations. The mixture containing KPA requires a significantly smaller ejection force compared with the conventional mixture and exhibits superior mold releasability. This effect is enhanced at higher compacting pressures. The ejection force is decreased by about 20% at a compacting pressure of 686MPa.

3.2 Lubrication mechanism of KPA

Several experiments were conducted to clarify the lubrication mechanism of KPA, which exhibits a superior mold releasability compared with Zn-st, or with EBS, both of which are commercially available. The viscoelasticity of the newly developed KPA and conventional EBS lubricants was measured

Table 1 Powder and green properties of KPA and other lubricant mixture

Lubricant	KPA	Zn-st	EBS
Apparent density (g/cm^3)	3.27	3.49	3.24
Flow rate (s/50g)	24.1	22.1	28.3
Critical flow diameter (mm)	22.5	22.5	30.0
Green density (g/cm^3)*	6.92	6.90	6.91
Green strength (MPa)*	12.0	10.0	10.6

*Compacting pressure : 490MPa

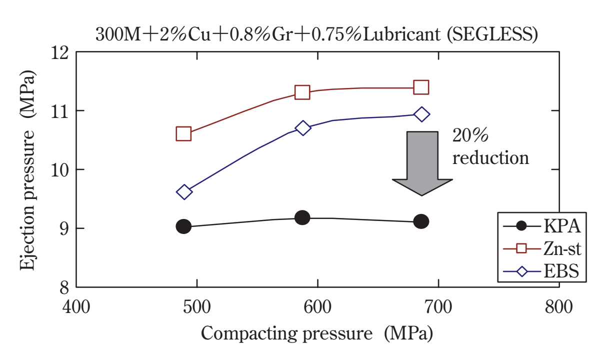

Fig. 7 Ejection pressure of KPA and conventional mixture

at different temperatures, using an instrument for measuring molten viscosity. The measuring instrument comprises a pair of disks, rotating in opposite directions, and a layer of lubricant placed between them, the layer being maintained at a thickness of about 1mm. The viscoelasticity is determined from the torque values obtained by rotating the disks at 62.5 rad/s (10Hz). **Fig. 8** compares the viscoelasticity of KPA to that of EBS. This figure indicates that the viscoelasticity of KPA gradually lessens as the temperature comes to exceed around 60℃, the temperature not exceeding the melting point of KPA, and remains lower than that of EBS. This suggest that, when ejecting the powder compact from a mold, KPA, which has a lower viscosity, can migrate more easily from the inside of the powder compact and accumulate on the mold wall surfaces to function better as a lubricant [5), 6)].

Next, the static friction coefficients of KPA and EBS were measured using a sliding tester, HEIDON. Each lubricant was applied on the surface of the steel plate in a thickness of 10 to 30 μm. Each sliding test was conducted with a load of 2 to 3MPa at a sliding speed of 100mm/min. The static friction coefficient was determined from the peak value of the resistance force during sliding for a specified distance. As shown in **Fig. 9**, the static friction coefficient of EBS increases with the rise in the surrounding temperature, while that of KPA appears to be independent of the temperature. This difference becomes more significant at temperatures above around 60℃. A static friction coefficient represents the lubricity of the lubricant itself. Even during compaction at room temperature, temperatures in some parts of the compact may exceed 60℃ due to the friction heat caused by the compacting pressure. This is considered to be the reason for the more effective lubricity of KPA.

Also studied were the behaviors of lubricants which migrate during compaction and ejection step to accumulate on the surfaces of the powder compacts. Three powder mixtures, each containing iron powder and 0.75% of the three respective lubricants, were compacted under three different pressures. For each compact, the surface that was in contact with a punch during the compaction was observed. **Fig.10** shows the results of energy dispersive X-ray (EDX) analyses performed on the surfaces of the powder compacts.

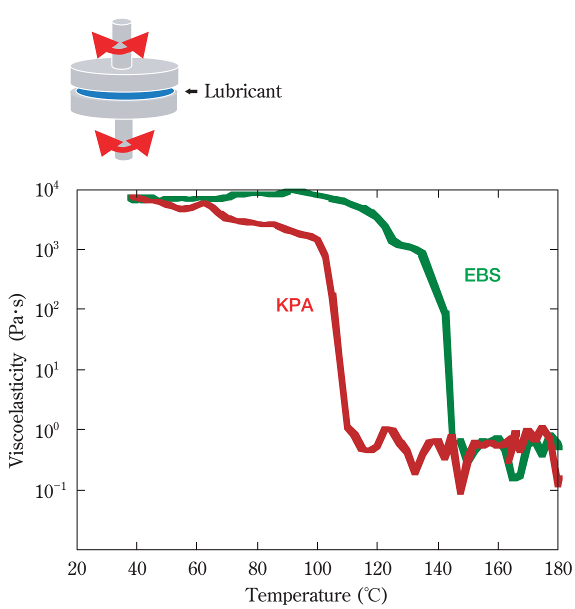

Fig. 8 Viscoelasticity of KPA and conventional lubricant

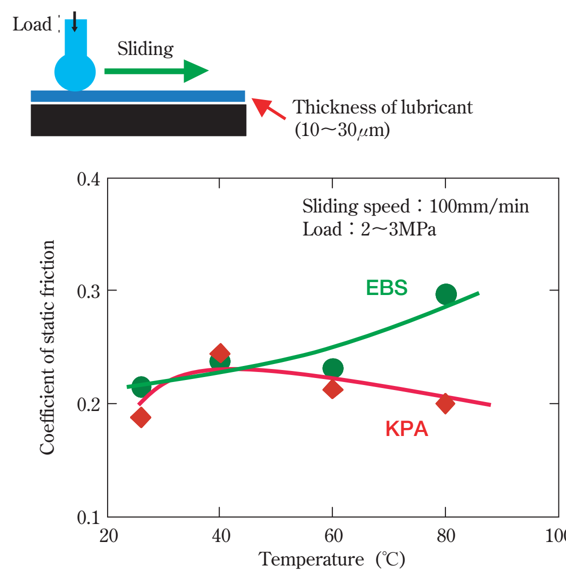

Fig. 9 Coefficient of static friction of KPA and conventional lubricant

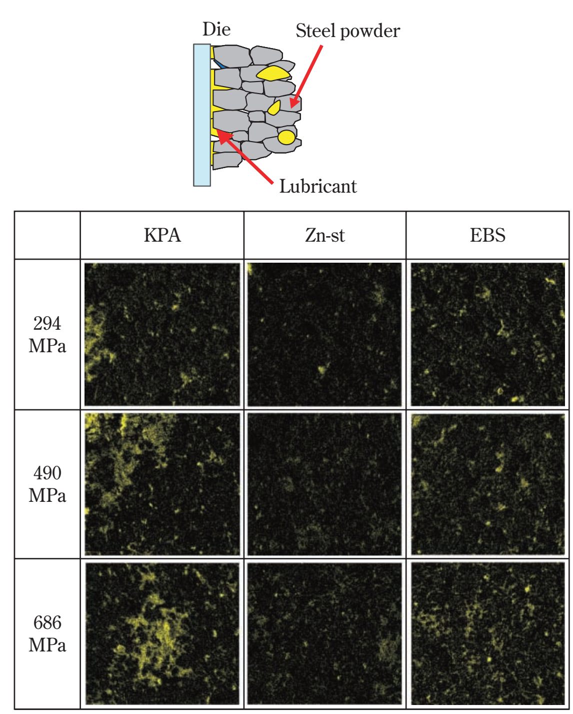

Fig.10 Lubricant distribution on surface of green compact by EDX

The yellow portions are where carbon, contained in each lubricant, is detected. These results indicate that, for all of the compacting pressures, larger quantities of KPA accumulate on the compact surfaces than in the case with the other two lubricants.

It has been concluded from the above experiments that KPA not only has a low static friction coefficient, which improves its lubricity, but also has a low viscosity that enables the lubricant to effectively seep to the compact surfaces. The resulting synergistic effect provides KPA with a mold releasability that is superior to that of other lubricants.

3.3 Characteristics of High-density SEGLESS containing KPA

KPA, with its excellent mold releasability, is expected to reduce the burden imposed on molds. This will extend the life of the molds and enable it to be applied to parts having complicated shapes. One way of using KPA is to decrease the amount added to a level that attains the ejection force achieved by conventional lubricants, while increasing the density of compacts. The high-density process with a reduced amount of lubricant additive does not call for additional equipment and, thus, can be established with relative ease. Such a process emits much less gas during dewaxing and is more environmentally friendly. This section introduces the features of a newly developed powder mix, "High-density SEGLESS", which contains KPA and halves the amount of lubricant additive compared with conventional technology.

Two types of powder mixtures were prepared: i.e., High-density SEGLESS, consisting of Kobe Steel's high-compressibility iron powder, 300NH, 2% of copper powder (average particle size, about 30μm), 0.8% of graphite powder (average particle size, 5μm) and 0.4% of KPA; and a conventional SEGLESS, containing 0.8% of conventional lubricant, EBS (average particle size, 25μm). **Table 2** summarizes the powder properties of High-density SEGLESS and the conventional SEGLESS. Both have a similar apparent density, however, High-density SEGLESS has a smaller amount of lubricant added and thus has a flow rate improved by 3s/50g compared with that of the conventional SEGLESS.

Fig.11 compares High-density SEGLESS with the conventional SEGLESS, in terms of the relationship between the compacting pressure, required to compact the mixture into a solid cylinder of ϕ25×25mm, and the resulting compact (green) density. High-density SEGLESS exhibits an improved compact density, 7.30g/cm^3 (compacting pressure, 686MPa), which is a result of the reduced amount of lubricant and is higher by 0.2g/cm^3 than that achieved by the conventional SEGLESS. **Fig.12** shows the forces required to eject the powder compacts of the various mixtures. In both the cases, the ejection force increases with increasing compact density. When comparing the two mixtures at a given compact density, High-density SEGLESS with reduced lubricant exhibits an ejection force comparable to that of conventional SEGLESS, proof that the amount of lubricant additive can be decreased without sacrificing ejection force.

Finally, both of the powder mixtures were compacted under different pressures into a ring shape with an outer diameter of 64mm, inner diameter of 24mm and height of 10mm. The ring-shaped compacts were sintered at 1,120℃ for 30 minutes in a nitrogen atmosphere containing 5% of hydrogen to produce sintered bodies. The characteristics of the sintered bodies were compared. Compressional load was applied by a press in the radial direction of each ring-shaped sample. The radial crushing strength was determined from the load that caused fracturing in each ring. The results are shown in **Fig.13**. In the case of High-density SEGLESS, the strength of the sintered body increases

Table 2 Powder properties of high density SEGLESS and conventional SEGLESS

	High density SEGLESS	Conventional SEGLESS
Apparent density (g/cm^3)	3.49	3.46
Flow rate (s/50g)	23.1	26.5

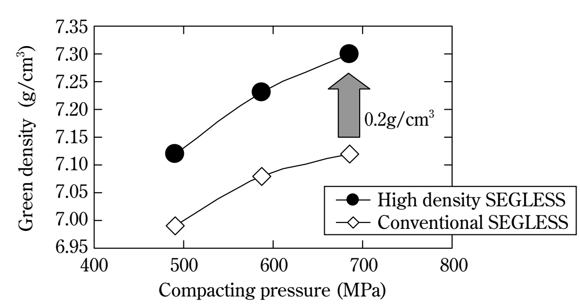

Fig.11 Compressibility curve of high density SEGLESS

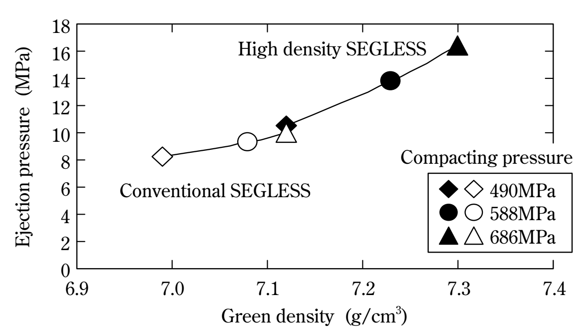

Fig.12 Ejection pressure of high density SEGLESS

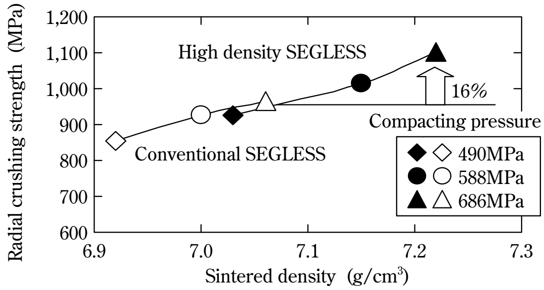

Fig.13 Radial crushing strength of sintered high density SEGLESS

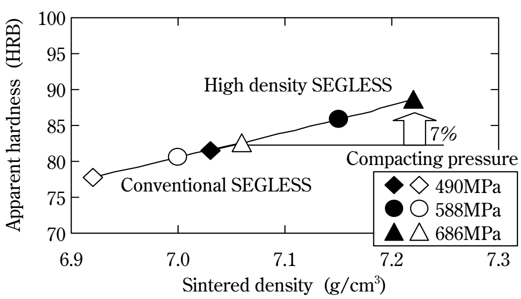

Fig.14 Apparent hardness of sintered high density SEGLESS

with increasing density, and, for a given compacting pressure, it increases by about 16% compared with conventional SEGLESS. **Fig.14** shows the apparent hardness, measured on the Rockwell Hardness B-Scale, of each sintered body. The hardness was determined from the average of the values measured at three points on the upper surface and another three points on the lower surface. The hardness also increases with increasing density in the same manner as in the case of strength; at a compaction pressure of 686MPa, it increased by about 7% as compared with conventional SEGLESS.

Conclusions

The following summarizes the characteristics of a newly developed functional lubricant, Kobelco Polyhydroxyl Amide (KPA), developed for powder metallurgy, and the features of the powder mixture comprising iron powder and the lubricant:

1) KPA, consisting of two types of lubricants with different melting points, can improve both flowability and mold releasability at the same time. Such an improvement has conventionally been difficult to achieve.
2) The iron-based mixed powder containing KPA exhibits an excellent mold releasability, which reduces the burden imposed on molds and is suitable for use in parts having complicated, thin-walled shapes.
3) High-density SEGLESS, containing an amount of KPA decreased to a level that exhibits the same ejection force as exhibited by conventional SEGLESS, enables the production of higher density parts without additional equipment. This responds to the needs for parts with higher strength and lower weight, features receiving much attention in recent years.
4) The newly developed lubricant is applicable to a wide variety of products, such as pure-iron based machine parts, high-strength parts made from alloy powder with inferior compactability and magnetic iron powder requiring higher density.

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