JFE TECHNICAL REPORT
No. 16 (Mar. 2011)

Molybdenum Hybrid-Alloyed Steel Powder for High Fatigue Strength Sintered Parts Using Mesh-Belt Sintering Furnace†

UNAMI Shigeru[*1] OZAKI Yukiko[*2]

Abstract:

The fatigue strength of the sintered and carburized compact made of the Mo hybrid-alloyed steel powder, which is based on a 0.6 mass% Mo prealloyed steel powder to which 0.2 mass% Mo powder particles have been diffusion bonded, is higher than that of the 0.6 mass% Mo prealloyed steel powder. The sintered compact made of the Mo hybrid-alloyed steel powder has a finer pore structure than that of the Mo prealloyed steel powder, because the Mo-rich region near the surface of the Mo hybrid-alloyed steel powder should exist as the α-iron phase with a high diffusion coefficient at high temperature and result in enhanced sintering. Improvement in the fatigue strength of the sintered and carburized compact made of the Mo hybrid-alloyed steel powder should result from not only the fine pore structure but also the strengthening of the sintering neck by Mo concentration around pores.

1. Introduction

The major applications of iron powder metallurgy products center on automobile and transportation equipment parts, which account for approximately 90% of domestic production[1]. High fatigue strength (contact and bending fatigue) is required in some automotive sintered parts which are used under severe high stress conditions due to downsizing of automotive engines.

It is known that reduction of the amount and size of pores, which cause fatigue crack initiation and propagation, is the most effective way to improve fatigue strength in iron-based sintered parts[2]. Technologies for reducing the amount of pores by densification, such as double-pressing and double-sintering[3] or warm compaction with die wall lubrication[4], and pore size reduction, such as high temperature sintering at over 1200°C[5] by a tray pusher furnace, have been adopted for production of special types of heavy-duty automotive sintered parts. However, the production cost of such methods is high due to their low productivities, and this has inhibited their widespread use. Therefore, a raw steel powder which makes it possible to attain a fine pore structure and high fatigue strength in sintered compacts produced by a conventional belt furnace whose sintering temperature is less than 1 200°C has been strongly desired.

Molybdenum (Mo) is more difficult to oxidize in manufacturing processes for powder materials and sintered parts than chromium and manganese because it has a lower affinity for oxygen than those elements. Mo also has the advantage of being easy to harden in heat treatment due to its high hardenability. For these reasons, Mo is conventionally applied to many sintered parts as an alloying elements added to raw powders for ferrous sintered materials[6,7].

On the other hand, iron also contains about 2.9 mass% of Mo or more (about 1.7 at%) (hereinafter, % indicates mass%), and the alpha iron phase is generated even at the sintering temperature of 1 130°C[8]. It is known that alpha iron phase sintering is a method of promoting sintering diffusion, making use of the fact that the self-diffusion coefficient of the alpha iron phase is about 100 times larger than that of the gamma iron phase[9–11].

For example, it has been reported that sintering of Fe-3.5%Mo prealloyed powder at 1 100–1 200°C proceeds more rapidly in comparison with Fe-1.5%Mo

† Originally published in *JFE GIHO* No. 26 (Aug. 2010), p. 48–53

*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

prealloyed powder[10]. It has also been reported that sintering proceeds more rapidly with Mo contents of more than 4% in a system consisting of pure iron powder with added Fe-60%Mo fine powder[11]. However, with prealloyed steel powder containing 4% Mo, the green density of the powder compact decreases and it is difficult to reduce the amount of pores due to reduction of plastic deformability by solid solution hardening of the powder particles. On the other hand, with mixed powders of pure iron powder and Mo-containing powder, regions of high Mo concentration remain in the sintered compacts, and the microstructure is remarkably inhomogeneous. The fatigue strength of these sintered compacts also decreases.

Therefore, JFE Steel developed an Mo hybrid-alloyed steel powder[12] for attaining a good compressibility with a low Mo content in a prealloyed powder and good sinterability with an Mo-rich region on the surface of the prealloyed powder. The developed powder is based on an Mo prealloyed steel powder, to which Mo powder was diffusion-bonded. A schematic illustration of the particle structure of the Mo hybrid-alloyed steel powder JIP® AH6020 is shown in **Fig. 1**. JIP® AH6020 is based on a 0.6% Mo prealloyed steel powder, to which 0.2% of Mo powder was diffusion-bonded.

This paper describes the properties of sintered and heat-treated compacts made of the Mo hybrid-alloyed steel powder using a mesh-belt sintering furnace.

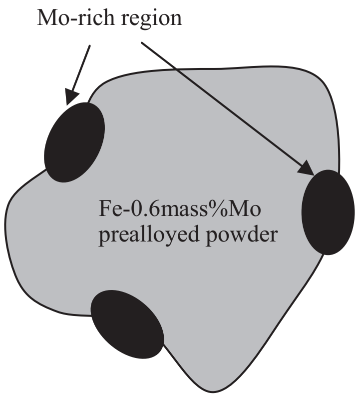


Fig. 1 Schematic illustration of particle structure of the Mo hybrid-alloyed steel powder “JIP® AH6020”

2. Experimental procedure

2.1 Raw Materials

Mo hybrid-alloyed powders JIP® AH6020 or JIP® AH4515 (hereinafter, AH6020 or AH4515) or a 0.6 mass% Mo prealloyed steel powders (hereinafter, 0.6Mo), as shown in **Table 1**, were used as the base materials. A natural graphite powder (Nippon Graphite Industries, Ltd., J-CPB, average diameter: 4 μm) and three types of internal lubricants JWAXB (KWAXB)[13], HDX[14], and JW-WAX (KW-WAX)[15] were added in the base powders. JWAXB with excellent flowability is suitable for conventional compaction at room temperature. HDX with good lubricity even in small amounts is suitable for heated die compaction with a powder at room temperature and a heated die at 60°C. JW-WAX is suitable for warm compaction with a heated powder and die at 130°C. The chemical compositions of the powders are shown in **Table 2**.

2.2 Processing Conditions

The base powders shown in Table 1 were mixed with graphite and a lubricant, as shown in Table 2. The mixed powders were compacted under the conditions shown in Table 2. The green compacts were sintered at 1 130°C for 20 min in endothermic gas by a mesh-belt furnace. The samples were carburized at 900°C for 60 min under a carbon potential of 0.8%, followed by quenching in oil

Table 1 Chemical compositions of Mo hybrid-alloyed and the prealloyed steel powders

Grade	Mo content (mass%)		
	Prealloy	Diffusion alloy	Total
Hybrid (AH6020)	0.59	0.20	0.79
Hybrid (AH4515)	0.45	0.15	0.60
Prealloy (0.6 mass% Mo)	0.59	—	—

Table 2 Compositions and compacting conditions

Symbol	Composition			Compacting condition		
	Graphite content (mass%)	Lubricant	Lubricant content (mass%)	Compacting temperature		Compacting pressure (MPa)
				Powder	Die	
CC	0.3	JWAXB	0.8	R.T.	R.T.	690
HD	0.3	HDX	0.5	R.T.	60℃	690
WC	0.3	JW-WAX	0.6	130℃	130℃	690

CC: Cold compaction HD: Heated die compaction
WC: Warm compaction R.T.: Room temperature

at 60°C and tempering at 180°C for 60 min.

2.3 Property Evaluation Methods

The densities of the sintered compacts were measured by the Archimedes method. The tensile strength and Charpy impact values of the sintered and carburized compacts were evaluated in accordance with JIS Z 2241 and 2202, respectively. The specimens for the tensile test were prepared by machining to a dimensions of 5 mm in diameter and 15 mm in length after sintering.

Unnotched specimens 10 mm in width, 10 mm in thickness, and 55 mm in length were used for Charpy impact tests. The rotating bending fatigue strength of the sintered and carburized compacts was evaluated in accordance with JIS Z 2274 using 8 specimens. The dimensions of the specimens for the fatigue test were 8 mm in diameter and 15.4 mm in length. The test was performed at a load ratio R of –1 and a rotation speed of 3 000 rpm. Fatigue strength was defined as the stress at which failure did not occur in more than half of the specimens at 10^7 cycles. The microstructures were observed with an optical microscope after etching the cross-section in 3% nital. The pore structures of non-etched cross sections were also observed with an optical microscope.

3. Results

The relationship between the sintered density and tensile strength of the sintered and carburized compacts made of AH6020, AH4515 or 0.6Mo is shown in **Fig. 2**. The sintered density of the hybrid-alloyed steel powder AH6020 increases about 0.1 Mg/m^3 when using the heated die at 60°C and about 0.15 Mg/m^3 when using the heated powder and die at 130°C in comparison with conventional compaction at room temperature. The tensile strength of the sintered and carburized compacts made of the hybrid-alloyed steel powder AH6020 (hereinafter, AH6020 compact) increases with increases in density by densification. The tensile strength of the AH6020 compact reaches 1 300 MPa at 7.3 Mg/m^3 and is slightly higher than that of the sintered and carburized compacts made of the hybrid-alloyed steel powder AH4515 (hereinafter, AH4515 compact) or the prealloyed steel powder (hereinafter, 0.6Mo compact).

The relationship between the sintered density and rotating bending fatigue strength of the AH6020,

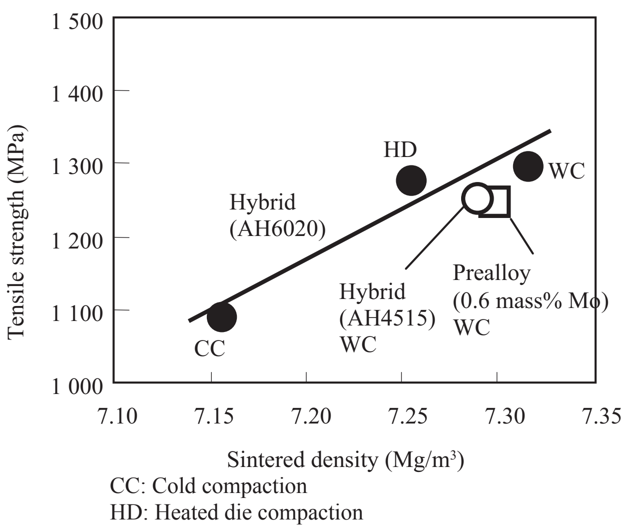


Fig. 2 Relationship between sintered density and tensile strength of the sintered and carburized compacts made of Mo hybrid-alloyed and prealloyed steel powders

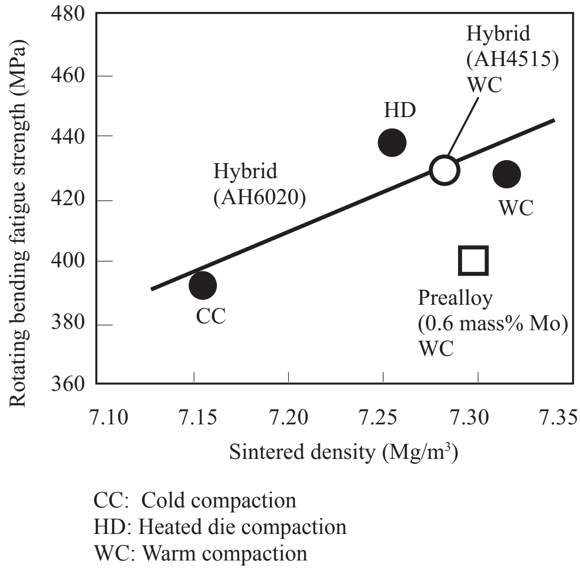


Fig. 3 Relationship between sintered density and rotating bending fatigue strength of the sintered and carburized compacts made of Mo hybrid-alloyed and prealloyed steel powders

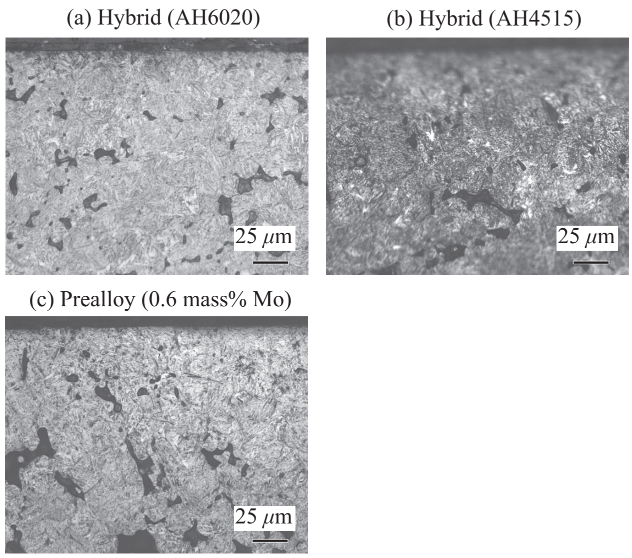


Photo 1 Cross-sectional microstructure of the sintered and carburized compacts made of Mo hybrid-alloyed and prealloyed steel powders

AH4515, and 0.6Mo compacts are shown in **Fig. 3**. The fatigue strength of the AH6020 compact tends to increase with increases in density and reaches 430 MPa at 7.3 Mg/m^3. The fatigue strengths of the AH4515 and AH6020 compacts are substantially the same, and are higher than the 410 MPa of the high-temperature sintered and carburized compacts made of the conventional 4% Ni diffusion alloyed steel powder[16]. The AH4515 and AH6020 compacts display remarkably higher fatigue strength than the 0.6Mo compact at the same density.

The cross-sectional microstructures of the surfaces of the AH6020, AH4515, and 0.6Mo compacts are shown in **Photo 1**. These compacts have an almost homogeneous tempered martensite structure and there are no large differences among the materials.

4. Discussion

Figure 3 indicates that the sintered and carburized compacts made of the hybrid alloyed steel powders (AH6020 and AH4515) show higher fatigue strength than compacts of the prealloyed steel powder (0.6Mo). The mechanism responsible for improved fatigue strength with AH6020 is discussed in the following.

Figure 4 shows X-ray diffraction patterns of the green compacts of AH6020 and 0.6Mo at 900°C. The γ-iron phase is detected in the 0.6Mo green compact. In contrast, not only the γ-iron phase but also the α-iron phase is detected in the AH6020 green compact. The Mo-rich region near the surface of the AH6020 powder particles is considered to exist as the α-iron phase even at high temperature.

Sintering behavior at 1 130°C and 20 min in endothermic gas was measured with a contact-type thermal dilatometer using green compacts of the AH6020 and 0.6Mo powders. The results are shown in **Fig. 5**. Figure 5(a) is at full range and Fig. 5(b) is in the high temperature range >950°C. There is no difference at low temperature. The AH6020 sintered compact shows lower dimensional change at 900°C accompanying the α-γ transformation than 0.6Mo. This is attributable to the residual α-iron phase remaining at 900°C. The interparticulate Mo-rich regions, which form the diffusive α-iron phase in the early sintering stage of 1 000–1 130°C, enhanced sintering diffusion.

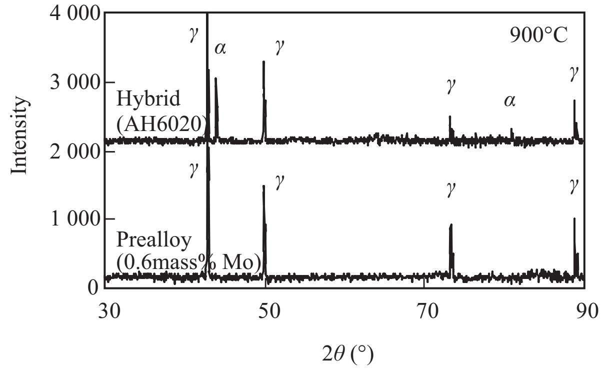


Fig. 4 Results of high temperature X-ray diffraction of the green compacts made of Mo hybrid-alloyed and prealloyed steel powders

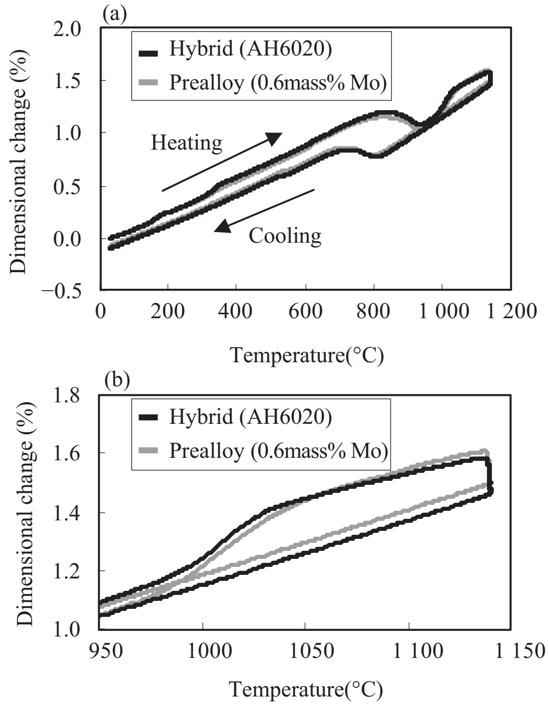
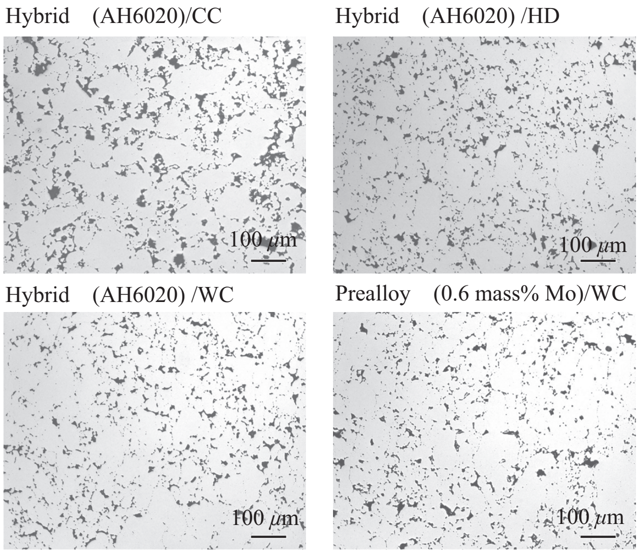


Fig. 5 Sintering behavior of Mo hybrid-alloyed and prealloyed steel powders((a)Full range, (b)High temperature range)

The slope of the expansion curve of AH6020 is about 18% smaller from 1 000 to 1 130°C and shrinkage during keeping time is about 14% larger than with 0.6Mo. This indicates that sintering of AH6020 is enhanced in the early stage.

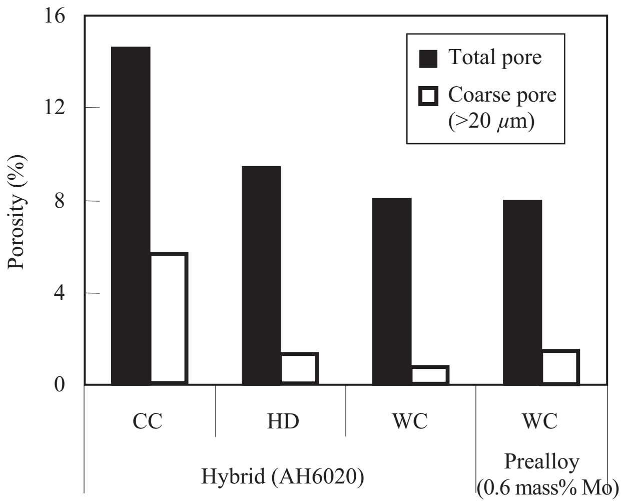
Cross-sectional pore structures are shown in **Photo 2**. The amounts of total and coarse pores over 20 μm of equivalent circle diameter with areas of 0.48 mm^2, which were quantified by image analysis, are shown in **Fig. 6**. The sintered and carburized compact of AH6020 has fewer total pores as its density increases. The AH6020 compact has the same total porosity but fewer coarse pores compared to the 0.6Mo compact. The AH6020 compact has a finer pore structure than the 0.6Mo compact, because the Mo-rich region near the surface of the Mo hybrid-alloyed steel powder exists as the α-iron phase, which has a high diffusion coefficient at high temperature, and this accelerates sintering diffusion.

The relationship between the amount of coarse pores over 20 μm and rotating bending fatigue strength is shown in **Fig. 7**. The rotating bending fatigue strength of the AH6020 compact tends to increase as the amount of coarse pores decreases. The AH6020 compact has higher rotating bending fatigue strength than the 0.6Mo



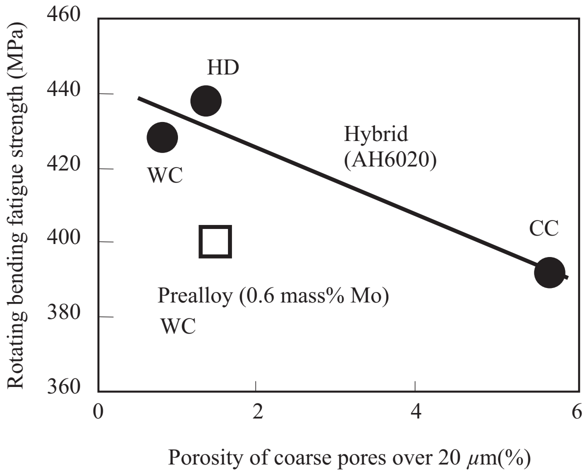
CC: Cold compaction
HD: Heated die compaction
WC: Warm compaction

Photo 2 Cross-sectional pore structure of the sintered and carburized compacts made of Mo hybrid-alloyed and prealloyed steel powders



CC: Cold compaction
HD: Heated die compaction
WC: Warm compaction

Fig. 6 Amount of total and coarse pores over 20 μm of the sintered and carburized compacts made of Mo hybrid-alloyed and prealloyed steel powders



CC: Cold compaction
HD: Heated die compaction
WC: Warm compaction

Fig. 7 Relationship between rotating bending fatigue strength and porosity of coarse pores over 20 μm of the sintered and carburized compacts made of Mo hybrid-alloyed and prealloyed steel powders

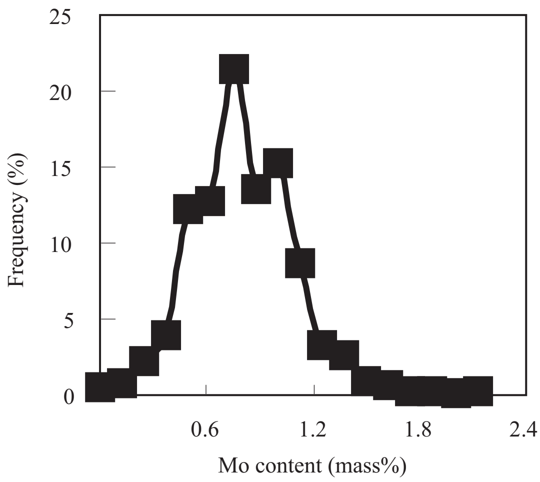


Fig. 8 Molybdenum content distribution in the sintered compact made of the Mo hybrid-alloyed steel powder calculated by electron probe micro analyzer (EPMA) area analysis

compact at the same amount of coarse pores. This means the increase in fatigue strength with the hybrid material cannot be explained only by the decrease in the amount of coarse pores.

The Mo content distribution of a sintered compact of AH6020, as calculated by EPMA area analysis, is shown in **Fig. 8**. The area fraction of Mo content has the maximum point at 0.8%, which is the average Mo content in AH6020. The Mo-rich area contains a maximum of 2% Mo. The area fraction of the Mo-rich area containing more than 0.8% Mo exceeds 40%. The Mo-rich area around the sintering neck should be strengthened in comparison with the matrix, which has a relatively low Mo content. Therefore, the improvement in the fatigue strength of the AH6020 compact is considered to result from suppression of fatigue crack initiation and propagation due to strengthening of the sintering neck.

5. Conclusions

An Mo hybrid-alloyed steel powder with an Mo-rich region on the surface of the prealloyed powder for attaining high fatigue strength in sintered parts was developed. The fatigue strength of sintered and carburized compacts made of the Mo hybrid-alloyed steel powder JIP® AH6020, which is based on a 0.6% Mo prealloyed steel powder to which 0.2% of Mo powder particles were diffusion-bonded, was investigated in comparison with a 0.6% Mo prealloyed steel powder. The major results are summarized as follows.

(1) The fatigue strength of sintered and carburized compacts made of the Mo hybrid-alloyed steel powder is higher than that of compacts of the prealloyed steel powder and reaches 430 MPa at 7.3 Mg/m^3.

(2) Improvement in the fatigue strength of sintered and carburized compacts made of the Mo hybrid-alloyed steel powder is considered to result from not only the fine pore structure of the compacts, but also strengthening of the sintering neck by Mo concentration around pores.

The Mo hybrid-alloyed steel powder has been highly evaluated for providing high fatigue strength at relatively low sintering temperatures, and has been adopted in several automotive parts. This Mo hybrid-alloyed steel powder is making a major contribution to reducing the manufacturing cost of high fatigue strength sintered parts. A further expansion of applications is expected in the future.

References

1) Annual Report 2009 of Japan Powder Metallurgy Association. 2009, p. 8.
2) Lindqvist, Bjorn. Metal Powder Report. 1989-06, p. 443.
3) Ogura, Kuniaki; Takajo, Shigeaki; Maeda, Yoshiaki; Katsuki, Junichi; Sakurada, Kazuo. Kawasaki Steel Giho. 1989, vol. 21, no. 3, p. 250. (Japanese)
4) Unami, Shigeru; Ozaki, Yukiko; Uenosono, Satoshi. JFE Giho. 2005, no. 7, p. 6. (Japanese)
5) Unami, Shigeru; Uenosono, Satoshi. Advances in Powder Metallurgy & Particulate Materials. MPIF, part 10, 2004, p. 10. 254.
6) Lindqvist, Bjorn. Proceedings of World Congress on Powder Metallurgy. 1990, no. 2, p. 170.
7) Milligan, Dave; Engstrom, Ulf; Lingenfelter, Jim; Dizdar, Senad; Nyberg, Ingalill. SAE Paper. 2003-01-0338, p. 1.
8) Kubaschewski, Ortrud. Iron-Binary Phase Diagrams. Springer-Verlag, 1982, p. 66.
9) Buffington, F. S.; Hirano, K.; Cohen, M. Acta Metallurgica. 1961, vol. 9, p. 434.
10) Schoeler, Annett; Bleck, Wolfgang; Link, Rainer. Steel Research. 2000, vol. 71, no. 1+2, p. 44.
11) Unami, Shigeru; Hayashi, Koji. Journal of the Japan Society of Powder and Powder Metallurgy. 1997, vol. 44, p. 765. (Japanese)
12) Unami, Shigeru; Ozaki, Yukiko. Materia. 2007, vol. 46, p. 93. (Japanese)
13) Uenosono, Satoshi; Sugihara, Hiroshi; Ogura, Kuniaki. Kawasaki Steel Giho. 1999, vol. 31, no. 2, p. 139. (Japanese)
14) Ozaki, Yukiko; Ono, Tomoshige; Unami, Shigeru. JFE Giho. 2005, no. 7, p. 1. (Japanese)
15) Ozaki, Yukiko; Unami, Shigeru; Uenosono, Satoshi. Kawasaki Steel Giho. 2001, vol. 33, no. 4, p. 170. (Japanese)
16) Furukimi, Osamu; Yano, Koji; Takajo, Shigeaki. Journal of the Japan Society of Powder and Powder Metallurgy. 1991, vol. 38, p. 18. (Japanese)