JFE TECHNICAL REPORT
No. 16 (Mar. 2011)

Ni-Free Alloyed Steel Powder “FM Series” for High Strength Sintered Compacts with Excellent Machinability†

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Abstract:

Ni-free alloyed steel powder “FM Series” for high strength sintered compacts with excellent machinability has been developed. FM Series are pre-mixed powder based on a low Mo prealloyed (0.45 mass%) steel powder added with copper, graphite and lubricant for high density compaction. The sintered compacts of FM series provide tensile strength of 600 MPa with mesh-belt sintered condition and tensile strength of 1 000 MPa with mesh-belt sintered and carburized condition, which are equivalent to those of the conventional 4 mass% Ni diffusion-alloyed steel powder. The sintered compacts of FM series provide less than one fifth the tool wear of that of the conventional 4 mass% Ni diffusion-alloyed steel powder. The high strength and excellent machinability are caused by microstructure homogenization of the sintered compacts due to low Mo prealloyed steel powder.

1. Introduction

Sintered compacts produced by a conventional mesh-belt sintering furnace using Fe-4mass%Ni-1.5mass%Cu-0.5mass%Mo diffusion-alloyed steel powder[1)], in which fine nickel, copper, and molybdenum (Mo) powders are diffusion-bonded to the surface of the iron powder particles, are widely adopted for tensile strength 600–1 000 MPa grade iron-based sintered parts. This powder has features of high compressibility due to the use of high purity iron powder as the base material, and the formation of a heterogeneous microstructure due to insufficient diffusion of the alloying elements during sintering.

Problem with the powder include the following:

(1) In spite of the high content of alloying elements, the tensile strength of the sintered compact is limited to 600–1 000 MPa due to the low strength microstructure.

(2) Machinability is reduced and machining costs increase due to the mixture of high and low strength microstructures and large differences in microhardness.

Therefore, a new low-cost alloyed steel powder with equivalent properties and excellent machinability has been desired.

In order to solve the above-mentioned problems, JFE Steel investigated a low alloy steel powder without Ni addition with sintered compact strength equivalent to that of the conventional 4 mass% Ni alloyed steel powder, and developed the segregation-free premixed powders in its FM Series.

The chemical compositions of the FM Series products and 4 mass% Ni alloyed steel powder (SIGMALOY®415S) mixtures, together with the recommended processes, are shown in **Table 1**. In the FM Series, FM600 has a tensile strength of 600 MPa as-sintering using a mesh-belt sintering furnace, and FM1000 has a tensile strength of 1000 MPa with case-hardening after mesh-belt sintering.

The concept of development of the FM Series was as follows:

(1) A molybdenum prealloyed steel powder was selected

† Originally published in *JFE GIHO* No. 26 (Aug. 2010), p. 54–59


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Table 1 Chemical compositions of FM Series and the 4Ni alloyed steel powder mixture and recommended process

Code	Chemical composition (mass%)								Recommended process
	Iron powder				Pre-mixed powder		Lubricant		
	Prealloy	Diffusion alloy							
	Mo	Ni	Cu	Mo	Cu	Gr	HDX	ZnSt	
FM600	0.45				2	0.8	0.5		As-sintering
FM1000	0.45				1	0.5	0.5		Sintering +Heat-treatment
SIGMALOY® 415S-0.5C		4	1.5	0.5		0.6		0.8	As-sintering
SIGMALOY® 415S-0.3C		4	1.5	0.5		0.3		0.8	Sintering +Heat-treatment

as the base powder because Mo has the advantages of high hardenability and resistance to oxidization at the conventional sintering temperature due to its low oxygen affinity. Molybdenum prealloyed steel powder has a uniform concentration of molybdenum, and the absence of the soft microstructure associated with insufficient diffusion of alloying elements contributes to increased strength of the matrix and improved machinability.

(2) Copper powder addition was selected for strengthening of the sintering neck. Copper powder gives a liquid phase at a relatively low temperature and short sintering time, and quickly diffuses into the sintered compact. As a result, strengthening can be obtained even at low alloying contents.

(3) In comparison with pure iron powder, the plastic deformability of the base powder particles was reduced by prealloying of Mo. Decreased green density was suppressed by using the lubricant HDX[2], which was developed previously for high-density compaction.

This paper discusses the mechanism responsible for the mechanical properties of sintered compacts made of the Ni-free alloyed steel powders in the FM Series.

2. Properties of Sintered Compacts of FM600 for As-sintered Use

2.1 Experimental Procedure

2.1.1 Raw materials

JIP® 4MOA (Fe-0.45mass%Mo prealloyed steel powder) was mixed with 2mass% of atomized Cu powder (average particle diameter: 35 μm), 0.8 mass% of a natural graphite powder (average particle diameter: 4 μm), and 0.5 mass% of the lubricant HDX, and given segregation-free treatment (hereinafter, this material is referred to as FM600). HDX is a newly-developed lubricant which makes it possible to achieve high green density without a powder heating unit[2]. As a comparison material, JIP SIGMALOY® 415S[1] (Fe-4mass%Ni-1.5mass%Cu-0.5mass%Mo diffusion-alloyed steel powder) was mixed with 0.6 mass% of a natural graphite powder and 0.8 mass% of zinc stearate as a lubricant (hereinafter, 4Ni alloyed steel powder).

2.1.2 Processing conditions

The mixed powders were compacted at 490, 590, and 690 MPa at room temperature. The green compacts were ring specimens (outer diameter: 38 mm, inner diameter: 25 mm, thickness: 10 mm), dog-bone type specimens (width: 5.7 mm, thickness: 5 mm, parallel portion: 32 mm, in accordance with JIS Z 2550), and bar specimens (width: 10 mm, thickness: 10 mm, length: 55 mm). The green compacts were sintered at 1 130°C for 10 min in an endothermic gas using a mesh-belt furnace.

For the rotating bending fatigue test, the mixed powders were compacted at 590 MPa at room temperature to bar specimens (width: 15 mm, thickness: 15 mm, length: 85 mm). The green compacts were sintered at 1 130°C for 10 min in an endothermic gas using a mesh-belt furnace. The sintered compacts were machined to dimensions of 8 mm in diameter, 15.4 mm in length in the parallel portion, and 80 mm in total length.

For the machining test, the mixed powders were compacted at 590 MPa at room temperature to ring specimens (outer diameter: 60 mm, inner diameter: 20 mm, thickness: 25 mm). The green compacts were sintered at 1 130°C for 20 min in an endothermic gas using a mesh-belt furnace.

2.1.3 Property evaluation methods

The densities of the sintered compacts were measured by the Archimedes method. Dog-bone type specimens were used for the tensile tests. Unnotched bar specimens (width: 10 mm, thickness: 10 mm, length: 55 mm) were used for the Charpy impact tests. The Rockwell hardness was measured using the B scale. The rotating bending fatigue test was performed using a load ratio R of –1 and rotation speed of 3 000 rpm. Fatigue strength was defined as the endurance limit at

10^7 cycles. The microstructures were observed with an optical microscope after etching the cross-section of the sintered compacts in 3% nital (mixture of 100 m*l* ethanol to 3 m*l* nitric acid).

The machinability of the sintered materials was evaluated by measuring the cutting tool wear during turning tests. Machining of the ring specimens was performed using a cermet insert (Grade T1200A, Sumitomo Electric Hardmetal Corp.). The machining conditions were cutting speed: 200 m/min, cutting depth: 0.5 mm, feed rate: 0.1 mm/rev, without coolant. The flank wear of the inserts was measured after 1 000 m of turning.

2.2 Results

The mechanical properties of the sintered compacts made of FM600 and 4Ni alloyed steel powder mixtures are shown in **Fig. 1**. The tensile strength and hardness of the FM600 material are approximately equivalent to those of the 4Ni alloyed steel. The tensile strength of both materials is 600 MPa at 7.1 Mg/m³. The Charpy impact value of the FM600 material is lower than that of the 4Ni alloyed steel.

The results of rotating bending fatigue tests of the sintered compacts made of FM600 and 4Ni alloyed steel powder mixtures are shown in **Fig. 2**. The FM600 material shows a higher fatigue strength of 195 MPa than the 4Ni alloyed steel.

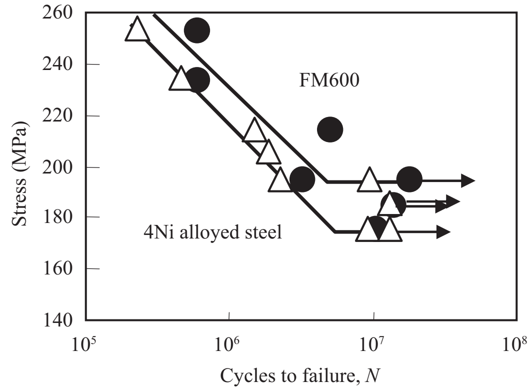

Fig. 2 Results of rotating bending fatigue tests of the sintered compacts made of FM600 and the 4Ni alloyed steel powder mixture

Optical micrographs of the cross-sectional microstructures of the sintered compacts made of FM600 and 4Ni alloyed steel powder mixtures are shown in **Photo 1**. The microstructure of the 4Ni alloyed steel consists of ferrite, pearlite, martensite, and austenite. Due to insufficient diffusion during sintering of the alloying elements Ni, Cu, and Mo, which are diffusion-bonded to the iron powder particles, this material comprises various microstructures, depending on the alloying content distribution. That is, the ferrite and pearlite

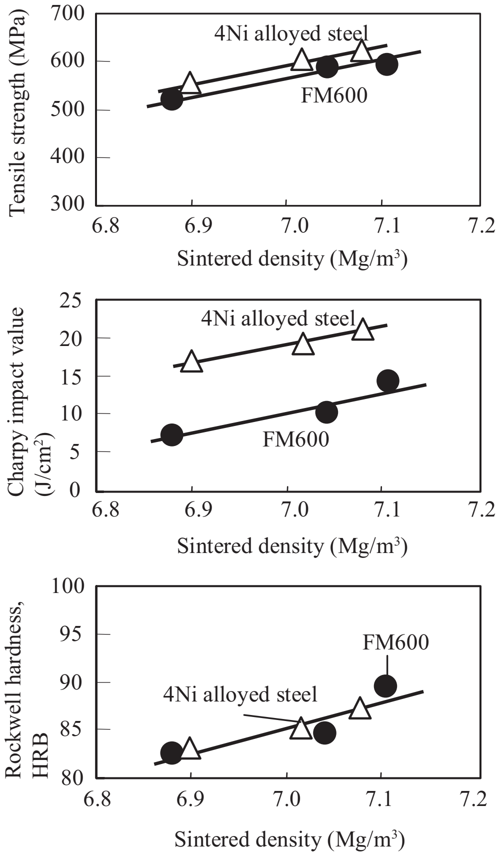

Fig. 1 Mechanical properties of the sintered compacts made of FM600 and the 4Ni alloyed steel powder mixture

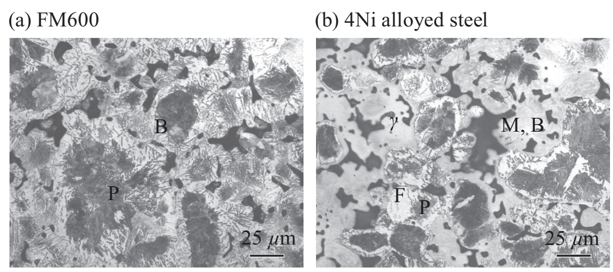

Photo 1 Microstructures of the sintered compacts made of FM600 (a) and the 4Ni alloyed steel powder mixture (b)

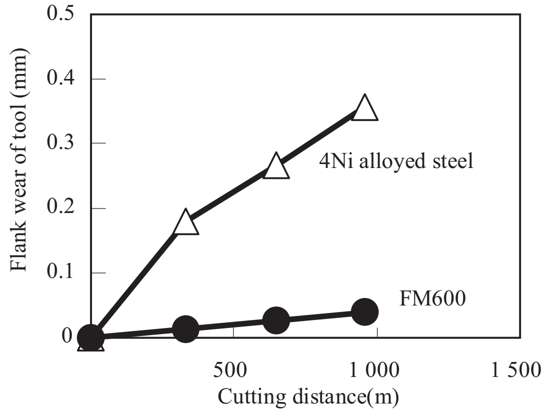

Fig. 3 Machinability of the sintered compacts made of FM600 and the 4Ni alloyed steel powder mixture

structures correspond to low alloying content regions which are prior iron powder particles, the martensite structure corresponds to high alloying content regions, and austenite corresponds especially to Ni-rich regions.

The microstructure of the FM600 material consists of white bainite structures surrounding gray fine pearlite structures.

The machinability of the sintered compacts made of FM600 and 4Ni alloyed steel powder mixtures are shown in **Fig. 3**. With the FM600 material, tool wear is reduced to less than one-fifth that with the 4Ni alloyed material.

2.3 Discussion

The effect of differences in the microstructures of the FM600 and 4Ni alloyed materials on their properties are discussed in the following.

A nickel-rich phase remains in the 4Ni alloyed steel, as shown in Photo 1 It is known that the Ni-rich phase, which forms due to incomplete diffusion of nickel during sintering, increases ductility and toughness. Therefore, the lower toughness of the FM600 material, as shown in Fig. 1, is attributed to the absence of the Ni-rich phase.

The fatigue crack propagation paths observed at the cross-section of fracture surfaces of the fatigue test specimens are shown in **Photo 2.** In the 4Ni alloyed steel, the fatigue crack passes through the boundaries of each microstructure or ferrite-pearlite structure, which is indicated by the arrows in Photo 2. The fatigue crack is considered to avoid the martensite structures with high alloy contents at sintering necks and to pass through the weak ferrite-pearlite microstructure, which consists of prior iron powder particles. In contrast, in the FM600 material, the fatigue crack propagates almost entirely through the bainite phases in the sintering neck, which is a stress concentration area, and does not pass through the fine pearlite structures.

In the FM600 material, fine pearlite structures, and not the low strength ferrite and pearlite structure, are generated due to molybdenum prealloying. In contrast, copper is distributed surrounding the prior molybdenum prealloyed steel powder particles, corresponding to bainite structures. The sintering necks in which the fatigue crack has propagated are strengthened by sintering enhancement and solution hardening due to copper addition. The fact that the FM600 materials exhibits high fatigue strength in spite of its lower alloy content is attributed to strengthening of the inner particle and interparticle regions (sintering neck).

The microstructure of the FM600 shows less difference in hardness and is more homogenous than that of the 4Ni alloyed steel because FM600 does not contain hard martensite and soft austenite structures, as shown in Photo 1. Therefore, the excellent machinability of the FM600 material, as shown in Fig. 3, is attributed to reduced intermittent shock during machining due to the more homogeneous hardness of the FM600 material.

(a) FM600 (b) 4Ni alloyed steel

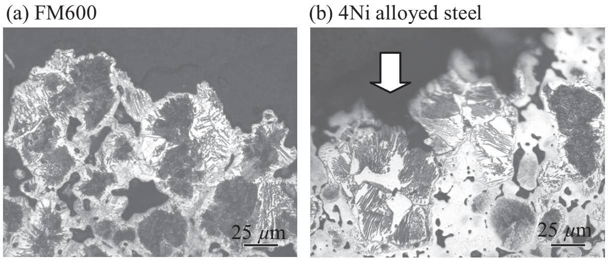

Photo 2 Fatigue crack propagation path observed at the cross-section of fatigue fracture surfaces of the sintered compacts made of FM600 (a) and the 4Ni alloyed steel powder mixture (b)

3. Properties of Sintered Compacts of FM1000 for Sintering and Heat Treatment Use

3.1 Experimental Procedure

3.1.1 Raw materials

JIP® 4MOA was mixed with 1mass% of atomized Cu powder (average particle diameter: 35 μm), 0.5 mass% of a natural graphite powder (average particle diameter: 5 μm) and 0.5 mass% of the lubricant HDX, and given segregation-free treatment (hereinafter, this material is referred to as FM1000). HDX is a newly-developed lubricant which provides high green density without use of a powder heating unit[2]. As a comparison material, JIP SIGMALOY® was mixed with 0.3 mass% of a natural graphite powder and 0.8 mass% of zinc stearate as a lubricant (hereinafter, 4Ni alloyed steel powder).

3.1.2 Processing conditions

The mixed powders were compacted at 590 MPa at room temperature. The green compacts were two types of bar specimens with different dimensions (width: 10 mm, thickness: 10 mm, length: 55 mm and width: 15 mm, thickness: 15 mm, length: 85 mm). The green compacts were sintered at 1 130°C for 20 min in an endothermic gas using a mesh-belt furnace. The samples were carburized at 900°C for 60 min under a carbon potential of 0.8%, quenched in oil at 60°C, and tempered at 180°C for 60 min.

For the machining test, the mixed powders were compacted at 590 MPa at room temperature to ring specimens (outer diameter: 60 mm, inner diameter: 20 mm, thickness: 25 mm). The green compacts were sintered at 1 130°C for 20 min in an endothermic gas using a mesh-belt furnace.

3.1.3 Property evaluation methods

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 dimensions of the specimens for the tensile test were 5 mm in diameter and 15 mm in length. The specimens were prepared by machining after sintering. Unnotched specimens 10 mm in width, 10 mm in thickness, and 55 mm in length were used for the Charpy impact tests. Rockwell and Vickers hardness was measured in accordance with JIS Z 2245 and 2244, respectively. The rotating bending fatigue strength of the sintered and carburized compacts was evaluated in accordance with JIS Z 2274. The dimensions of the specimens for the fatigue test were 8 mm in diameter and 15.4 mm in length. The fatigue test was performed using a load ratio R of –1 and 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 specimen at 10^7 cycles. The microstructures were observed with an optical microscope after etching the cross-section of the sintered compacts in 3% nital.

The machinability of the sintered materials before carburizing was evaluated by measuring the cutting tool wear during turning tests. Machining of the ring specimens was performed using a P type carbide insert (Grade ST10P, Sumitomo Electric Hardmetal Corp.). The machining conditions were cutting speed: 200 m/min, cutting depth: 0.5 mm, feed rate: 0.1 mm/rev, without coolant. The flank wear of the inserts was measured after 1 000 m of turning.

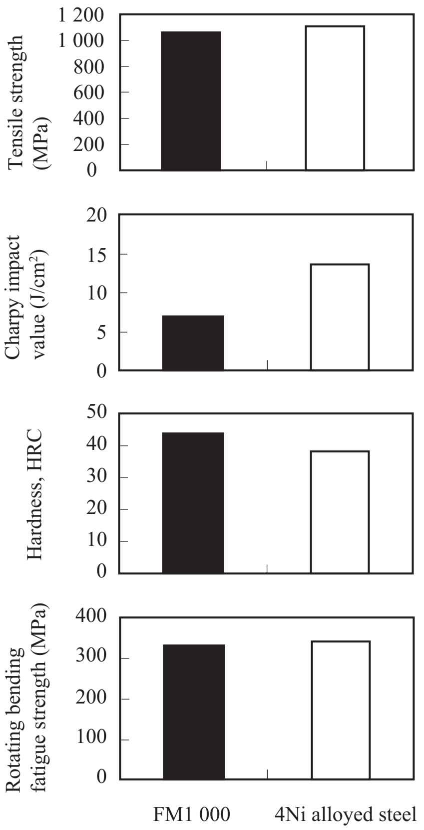

Fig. 4 Mechanical properties of the sintered and case-hardened compacts made of FM1000 and the 4Ni alloyed steel powder mixture

3.2 Results

The mechanical properties of the sintered and case-hardened compacts made of FM1000 and 4Ni alloyed steel powder mixtures are shown in **Fig. 4**. The tensile strength and rotating bending fatigue strength of the FM1000 material are substantially equivalent to those of the 4Ni alloyed steel. However, the FM1000 material displayed a lower impact value and higher hardness than the 4Ni alloyed steel.

The microstructures of the cross section of the surface of the sintered and case-hardened compacts made of FM1000 and the 4Ni alloyed steel powder mixture are shown in **Photo 3**. The microstructure of the FM1000 material consists of a uniform tempered martensite structure. In contrast, the 4Ni alloyed steel contains white Ni-rich phases in a martensite structure.

The machinability of the sintered compacts made of FM1000 and 4Ni alloyed steel powder mixtures is shown in **Fig. 5**. The FM1000 material reduces tool wear to less than one-fifth that with the 4Ni alloyed

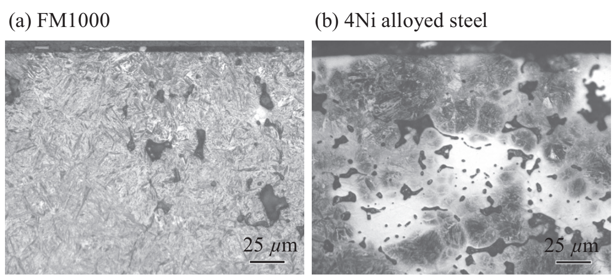

Photo 3 Microstructures of the sintered and case-hardened compacts made of FM1000 (a) and the 4Ni alloyed steel powder mixture (b)

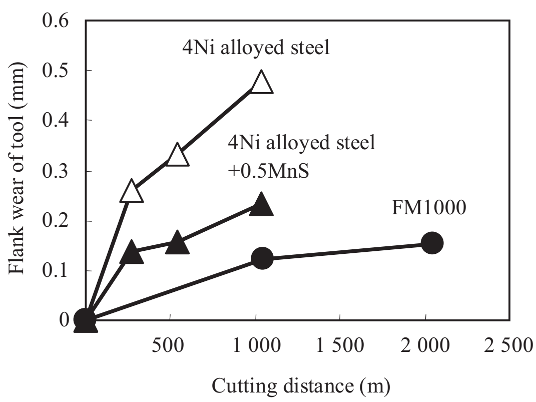

Fig. 5 Machinability of the sintered compacts made of FM1000 and the 4Ni alloyed steel powder mixture

steel. Moreover, the FM1000 material also shows lower tool wear than 4Ni alloyed steel with manganese sulfide as a machinability improvement additive, and thus has extremely high machinability.

3.3 Discussion

The effect of copper on the strength of the sintered and case-hardened compacts made of the molybdenum prealloyed steel powder are discussed in the following.

The effects of the amount of copper addition on the tensile and fatigue strength of the sintered and case-hardened compacts made of the molybdenum prealloyed steel powder are shown in **Fig. 6**. Tensile strength increases by about 100 MPa and achieves 1 070 MPa with 1% copper powder addition, but reaches saturation with 2% copper powder addition.

Rotating bending fatigue strength increases from 300 MPa to 330 MPa when copper addition is increased from 0% to 1%. However, fatigue strength decreases when copper addition increases from 1% to 2%.

Copper has the effects of enhancing sintering and strengthening particle connection due to the generation of a liquid phase during sintering. Other factors also affect the properties of sintered and case-hardened compacts, including the carburizing depth, pore distribution, amount of retained austenite, and fatigue crack propagation behavior.

The hardness distributions of the sintered and case-hardened compacts made of the molybdenum prealloyed steel powder are shown in **Fig. 7**. There are no large differences in the hardness distribution related to the amount of copper addition. This means that copper does not have a significant effect on carburizing behavior and hardenability in this case-hardening condition.

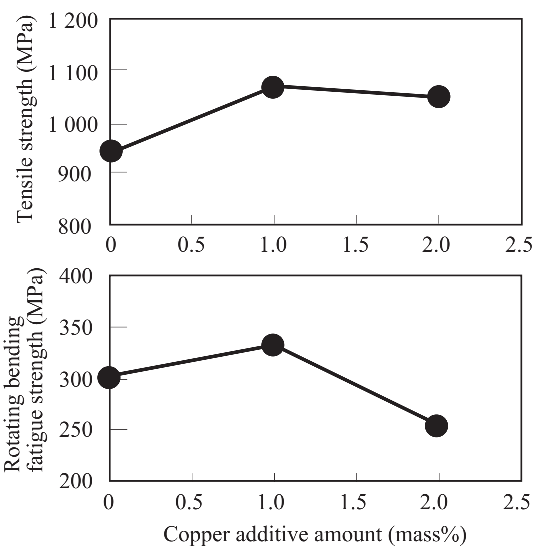

Fig. 6 Effects of copper addition on mechanical properties of the sintered and case-hardened compacts made of the low molybdenum prealloyed steel powder

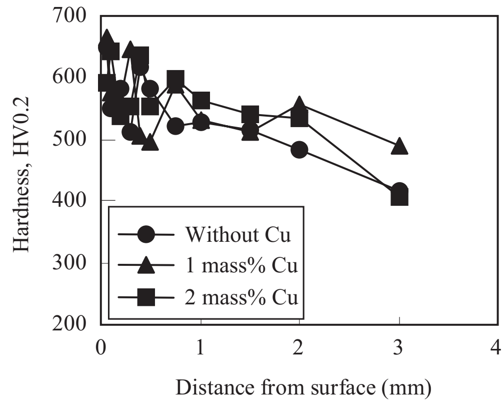

Fig. 7 Microhardness profiles in the surface layer of the sintered and case-hardened compacts made of the low molybdenum prealloyed steel powder with copper addition

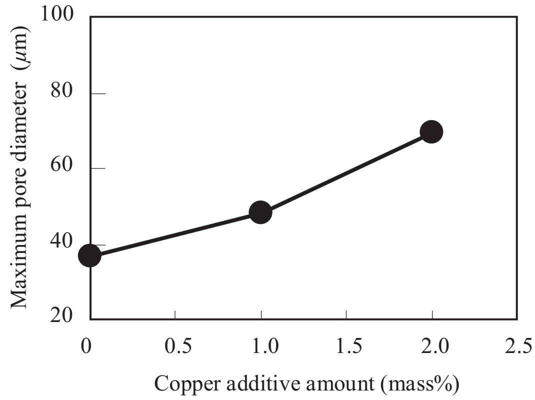

Fig. 8 Maximum pore size of the sintered and case-hardened compacts made of the low molybdenum prealloyed steel powder with copper addition

The amount of retained austenite increased very slightly with increasing copper addition. However, it would be extremely difficult to attribute the sudden drop in fatigue strength to this small increase in retained austenite.

Next, the pore size distribution in the sintered and case-hardened compacts was quantified by image analysis (0.96 mm^2) of photographs of the cross section. **Figure 8** shows the relationship between the maximum pore diameter and copper addition.

The maximum pore size increases with increasing copper addition. Coarse pores are thought to be generated by copper liquid phase outflow and become an origin of fatigue fracture due to stress concentration.

The stress concentration at coarse pores in the quenched and tempered materials should increase, because these materials are more sensitive to stress concentration due to their higher hardness in comparison with as-sintered material. Therefore, the coarse pores should have a stronger effect on fatigue strength than on tensile strength.

It has also been reported that 2% copper addition increased the fatigue crack propagation rate by 12% in a sintered and quenched material made of a low molybdenum prealloyed steel powder[3]. Thus, as a factor in the reduction of fatigue strength when copper addition was increased to 2%, with no corresponding change in tensile strength, as observed in the present research, the effect of the fatigue crack propagation rate on reduced fatigue strength cannot be considered insignificant.

Based on these results, fatigue strength should increase by 1% copper addition, because the effect of sintering enhancement outweighs the effect of the increased maximum pore size due to the copper liquid outflow. However, there is a high possibility that the decreased fatigue strength when copper addition was increased to 2% copper was influenced by an increase in the rate of fatigue crack growth, in addition to an increase in maximum pore size.

4. Conclusions

JFE Steel developed the FM Series of nickel-free alloyed powders for high strength sintered compacts with excellent machinability based on a low Mo prealloyed (0.45%) steel powder with added copper and graphite. The mechanical properties of sintered compacts made of premixed powders in the FM Series were investigated. The major results are summarized as follows:

(1) Sintered compacts of FM600, based on low Mo prealloyed (0.45%) steel powder with added copper (2%), graphite (0.8%), and the lubricant HDX for high green density (0.5%), provide tensile strength of 600 MPa and rotating bending fatigue strength of 195 MPa, which are equivalent to those of the conventional 4%Ni diffusion-alloyed steel powder.

(2) Sintered and case-hardened compacts of FM1000, based on low Mo prealloyed (0.45%) steel powder with added copper (1%), graphite (0.5%) and HDX (0.5%), provide tensile strength of 1 070 MPa and rotating bending fatigue strength of 330 MPa, which are equivalent to those of the conventional 4% Ni diffusion-alloyed steel powder.

(3) Sintered compacts of both FM600 and FM1000 reduce tool wear to less than one-fifth that with the conventional 4% Ni diffusion-alloyed steel powder, and show excellent machinability.

Powders in the FM Series can be sintered using conventional sintering methods and conditions and have excellent machinability. FM Series products are expected to be applied to automobile engine and drive train parts, because the machining costs of sintered parts can be reduced significantly.

References

1) Ogura, Kuniaki; Abe, Teruyoshi; Makiishi, Yukio; Takajo, Shigeaki; Minegishi, Toshiyuki; Hatsugai, Eiji. Kawasaki Steel Giho. 1987, vol. 19, no. 3, p. 202.
2) Ozaki, Yukiko; Ono, Tomoshige; Unami, Shigeru. JFE Giho. 2005, no. 7, p. 1.
3) Saritas, Suleyman; Causton, Robert; James, W. Brien; Lawley, Alan. Advances in Powder Metallurgy and Particulate Materials. 2002, part 5, p. 136.