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Ni-Free Alloyed Steel Powder for Manufacturing Heat-Treated Compacts with Tensile Strength of 1 300 MPa and 1 500 MPa Grades†

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Abstract:

JFE Steel has developed new grades of Ni-free alloyed steel powders, "JIP™ FM1300" and "JIP™ FM1500", with tensile strengths of 1 300 MPa and 1 500 MPa, respectively, after sintering and heat-treatment. "FM1300" is a Mo-prealloyed steel powder with diffusion bonded Mo fine particles on the steel particle surfaces for acceleration of inter-particle atomic diffusion during sintering and improvement of mechanical strength of sintered products. "FM1500" includes Cr and Mn as well as Mo for further improvement of the hardenability effect. The newly developed powders show almost homogeneous microstructures after sintering at high temperatures (≧1 200˚C), and give the tensile strengths mentioned above after appropriate heat-treatments. In conjunction with the products developed previously, JFE Steel has lined up Ni-free alloyed steel powders suitable for a wide range of applications requiring tensile strength from 600 MPa to 1 500 MPa.

1. Introduction

Fe-based sintered parts are used in numerous applications, beginning with the automotive field, and in recent years, high strength materials have been adopted in order to respond to the trends toward weight reduction and downsizing. In applications in which high strength is required, alloyed steel powders with diffusion bonded Ni particles on the steel particle surfaces have long been used with Ni as an alloying element[1], but development of alloyed steel powders which meet the need for Ni-free compositions is also underway[2–4]. JFE Steel has already completed the development of the Ni-free alloyed steel powders "JIP™ FM600" and "JIP FM1000" in the tensile strength of 600–1 000 MPa grades[2].

This paper describes newly-developed alloyed steel powders which make it possible to manufacture Ni-free sintered materials of 1 300 MPa and 1 500 MPa grades. For example of sintered materials of these strength grades, it has been reported that tensile strength of 1 380 MPa was obtained with a 4 Ni type diffusion bonded alloyed steel powder (Fe-4 mass%Ni-1.5 mass%Cu-0.5 mass%Mo) by high temperature sintering at 1 250˚C, followed by carburizing, case-hardening and tempering[1]. Strength of 1 500 MPa has also been obtained with a 2 Ni type diffusion bonded alloyed steel powder (Fe-2 mass%Ni-1 mass%Mo) in materials manufactured by high temperature sintering equally at 1 250˚C and carburizing, case-hardening and tempering[1]. The newly-developed "JIP FM1300" and "JIP FM1500" reported here are segregation-free premixed powders based on Ni-free alloyed steel powders mixed with the optimum amounts of submaterials and lubricants. With these new powders, it is possible to obtain sintered materials of 1 300 MPa and 1 500 MPa grades, respectively, by sintering and heat treatment under the proper conditions. This paper describes the mechanical properties of sintered and heat-treated compacts manufactured from these alloyed steel powders.

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2. Properties of Ni-Free Ultra-High Strength Alloyed Steel Powder FM1300

2.1 Concept of Material Design

(1) Base Alloyed Steel Powder

As the base alloyed steel powder of "FM600" and "FM1000," 0.45 mass% Mo prealloyed steel powder was used[2]. In contrast, the based alloyed steel powder of "FM1300" is a hybrid alloyed steel powder (Prealloyed + Diffusion bonded alloyed steel powder) in which 0.15 mass% of Mo is diffusion bonded to 0.45 mass% Mo prealloyed steel powder in order to further increase strength. As shown in **Fig. 1**, the Mo particles which are diffusion bonded to the surface of the prealloyed steel powder maintaining the ferrite phase during sintering, and then, a high diffusion rate is kept and sintering is effectively accelerated. Hardenability improvement by high Mo content[5] as well as sintering acceleration leads to increase of strength.

(2) Submaterials and Sintering Conditions

The graphite powder used in "FM1300" is a natural graphite powder with a mean particle size of 4 μm. The amount of graphite addition was set at 0.5 mass% based on preliminary experiments. The applied lubricant is the previously-developed HDX™ providing high green densities[6]. The powder mixture was processed by JIP Cleanmix™ which contained segregation-free treatment for suppressing graphite powder segregation. As in the example[1] in which 1 300 MPa grade material was obtained with the conventional 4% Ni alloyed steel powder, the sintering conditions were premised on high temperature ones around 1 250°C followed by heat treatment of carburizing, case-hardening, and tempering.

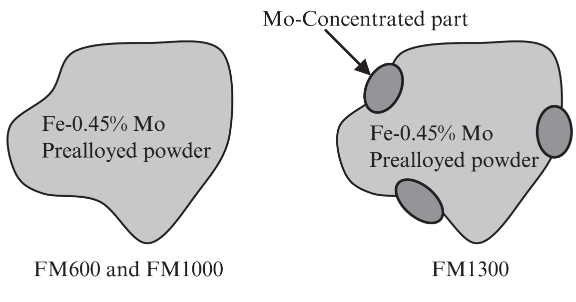

Fig. 1 Particle structure of the base alloyed steel powders

2.2 Test Material and Experimental Method

2.2.1 Raw material powder

The base powder was the hybrid Mo steel powder JIP™ AH4515, in which 0.45 mass% of Mo was alloyed by prealloying and 0.15 mass% of Mo was alloyed by diffusion bonding. The test material (hereinafter, "FM1300") was prepared by adding 0.5 mass% of natural graphite powder (Mean particle size: 4 μm) and 0.5 mass% of HDX™ [6] for high green densities to this base powder and conducting segregation-free treatment to suppress graphite powder segregation. A comparison material (hereinafter, "4Ni") was prepared by adding 0.3 mass% of natural graphite powder and 0.6 mass% of ethylene bis stearamide to a 4 mass% Ni diffusion bonded alloyed steel powder. **Table 1** shows a comparison of the compositions of the "FM1300" and "4Ni" powder mixtures.

2.2.2 Test specimen preparation conditions and evaluation method

Type A bar-shaped test specimens with dimensions of 55 mm × 10 mm × 10 mm, type B bar-shaped specimens with dimensions of 80 mm × 15 mm × 15 mm and ring-shaped test specimens with an outer diameter of 60 mm, inner diameter of 20 mm and height of 6 mm were prepared by compacting the respective powder mixtures at compaction pressures between 429 and 686 MPa. These test specimens were sintered at 1 250°C × 60 min in a 90 vol% N_2 + 10 vol% H_2 atmosphere.

For the tensile test, small-scale round-bar test specimens with a parallel part diameter of 5 mm were machined from the type A bar-shaped test specimens after sintering. After carburizing heat treatment (Carburizing: 900°C × 60 min, Carbon potential: 0.8 mass%, Hardening: In oil at 60°C, Tempering: 180°C × 60 min), tensile strength was evaluated based on JIS Z 2241 (JIS: Japanese Industrial Standards) . For hardness, the Rockwell hardness was measured on the surface of the type A

Table 1 Nominal composition of FM1300 and 4Ni steel powder mixture

(mass%)

Code	Steel powder				Pre-mixed powder	Lubricant	
	Prealloy	Diffusion alloy bonded					
	Mo	Ni	Cu	Mo	Gr	HDX™	EBS
FM1300	0.45	—	—	0.15	0.5	0.5	—
4Ni	—	4	1.5	0.5	0.3	—	0.6

Gr: Graphite, EBS: Ethylene-bisstearamide bar-shaped specimens after carburizing heat treatment under the above-mentioned conditions. Microstructural observations with an optical microscope were performed after etching the cut and polished surface of the type A specimens after carburizing heat treatment with 3% Nital.

For the rotating bending fatigue test, round bar-shaped test specimens with a parallel part diameter of 8 mm were machined from the type B bar-shaped specimens after sintering. Carburizing heat treatment was then performed under the above-mentioned conditions, and the surface of the specimens was finished smoothly by polishing. The rotating bending fatigue test was conducted with a rotation speed of 3 000 min^{-1} and stress ratio $R = -1$ with an Ono-type fatigue testing machine. The fatigue limit was determined as the maximum endurance stress at 10^7 cycles. For the contact fatigue test, the pressed surface of the ring-shaped test specimens after sintering was smoothed by mechanical polishing, and after carburizing heat treatment under the above-mentioned conditions, additional mirror polishing was performed. The contact fatigue test was conducted by the 6-ball method using a Mori-type testing machine. The fatigue limit was determined as the maximum contact stress (Hertz stress) at 10^7 cycles. The wear test was conducted using an Ogoshi-type abrasion tester. As the test specimens, the type A bar-shaped specimens after sintering and carburizing heat treatment under the above-mentioned conditions were used. As the opposite material, a cylindrical material made of SUJ2 (JIS G 4805, High carbon chromium bearing steels) was used. The side face of this cylindrical material was placed in contact with the test specimen under a load of 124 N, and this opposite material was rotated at a friction velocity of 4.21 m/s while dripping oil (Dexron III) in the contact part at a rate of 1 drop per second. Wear resistance was evaluated by the wear volume of the contact part. It was calculated from the width of the wear track on the test specimen surface and the cylinder diameter of the opposite material. The friction distance was obtained from (the circumference of the opposite material cylinder) × (the number of rotations). A smaller wear volume at the same friction distance could be seen as higher wear resistance.

2.3 Experimental Results and Discussion

Figure 2 shows the dependence of green density and sintered density on the compaction pressure of the type A bar-shaped specimens prepared using "FM1300" and the comparison material "4Ni. " Both the green density and the sintered density of "FM1300" and "4Ni" are approximately the same. **Figure 3** shows the relationship of tensile strength and sintered density. "FM1300" shows behavior approximately equal to that of "4Ni." If the density and compaction pressure for obtaining tensile strength of 1 300 MPa with "FM1300" and "4Ni" are interpreted from Fig. 2 and Fig. 3, the results are basically the same; concretely, the necessary conditions are a sintered density of 7.18 Mg/m^3, a green density of 7.03 Mg/m^3 and a compaction pressure of 570 MPa.

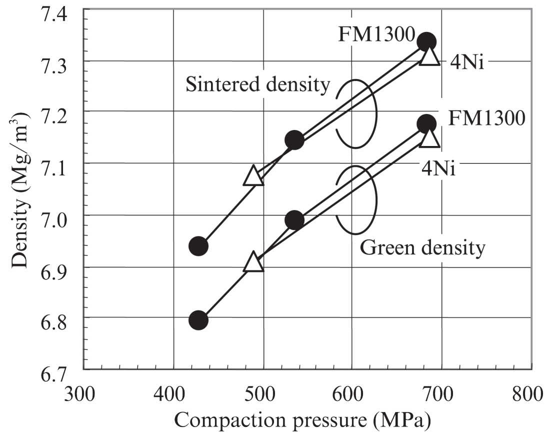

Fig. 2 Dependence of green and sintered density on compaction pressure

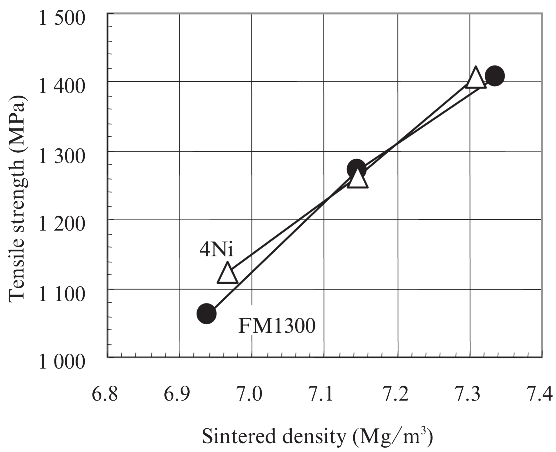

Fig. 3 Tensile strength of the case-hardened materials made from FM1300 and 4Ni

Figure 4 shows the *S-N* curves obtained in the rotating bending fatigue test and the contact fatigue test. The rotating bending fatigue limits of "FM1300" and "4Ni" are approximately the same, and the contact fatigue limit of "FM1300" is larger than that of "4Ni." **Figure 5** shows the results of the wear test. The wear volume of "FM1300" is about 2 orders smaller than that of "4Ni," showing that "FM1300" has extremely high wear resistance. Concerning these mechanical properties, it can be understood that "FM1300" has potential equal or superior to that of "4Ni."

The cross-sectional microstructures of the surface layer of the test materials used in the evaluation of properties are shown in **Photo 1**. In both "FM1300" and "4Ni," many parts have a tempered martensite micro-

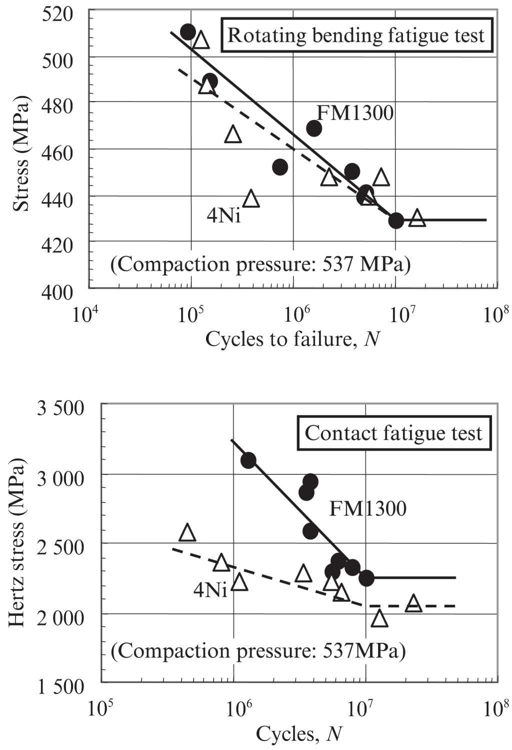

Fig. 4 Fatigue properties of the case-hardened compacts made from FM1300 and 4Ni

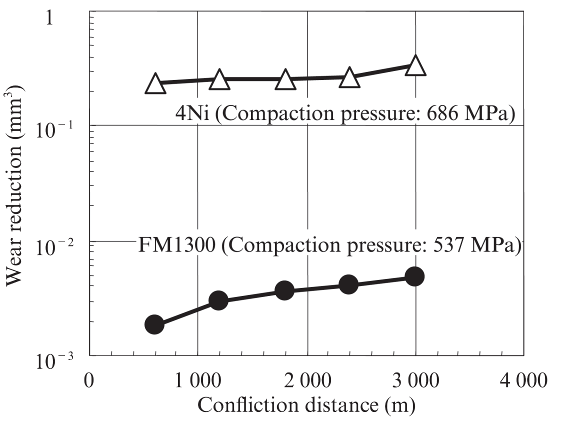

Fig. 5 Wear resistance of the case-hardened compacts made from FM1300 and 4Ni

structure; however, a large amount of retained austenite, which is shown in white, is distributed in “4Ni.” As the reason, it is thought that the Ni which was diffusion bonded to the surface of the iron powder particles was not adequately diffused, and Ni-rich parts that remained in the microstructure formed retained austenite. The results of measurements of the surface hardness of the specimens are shown in **Fig 6**. The surface hardness of “FM1300” is higher than that of “4Ni,” and at the sintered density of 7.18 Mg/m^3, at which both materials achieve a tensile strength of 1 300 MPa, the hardness of “FM1300” is approximately 10% higher. Because “4Ni”

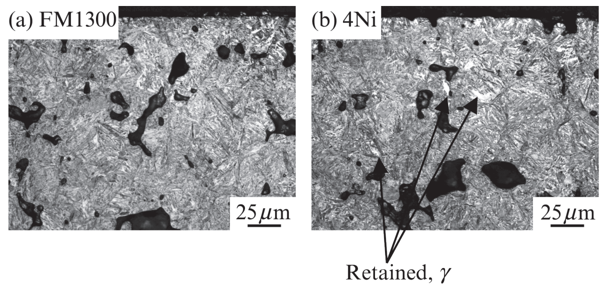

Photo 1 Microstructures of the case-hardened compacts made from FM1300 (a) and 4Ni (b)

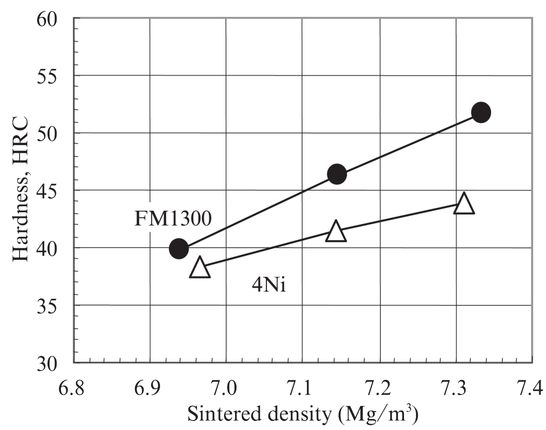

Fig. 6 Surface hardness of the case-hardened materials made from FM1300 and 4Ni

contains a large amount of retained austenite, which is a softer phase than martensite, both results of the microstructural observations and the hardness measurements are in agreement. The fact that “FM1300” is superior to “4Ni” in terms of the contact fatigue limit and wear properties appears to be due to the effect of the hard microstructure of “FM1300,” which contains little retained austenite.

3. Properties of Ni-Free Ultra-High Strength Alloyed Steel Powder FM1500

3.1 Concept of Material Design

(1) Base Alloyed Steel Powder

In order to obtain high strength of 1 500 MPa grade, which is even higher strength than that of “FM1300,” an increase in hardenability was studied in “FM1500” by adding Cr and Mn as well as Mo and by using synergistic effect of these elements. In the selection of the composition, avoiding a reduction in the compressibility of the alloyed steel powder was also considered. Therefore, a composition with Cr as the main element was investigated, because Cr has the lowest solution hardening ability for ferrite among these elements[7]), and an prealloyed iron powder with the composition of 0.5 mass%Cr-0.2 mass%Mn-0.2 mass%Mo was selected.

(2) Submaterials and Sintering Conditions

The graphite powder used in “FM1500” is a natural graphite powder with a mean particle size of 4 μm. As the amount of addition, 0.7–0.8 mass% becomes a precondition. Like “FM1300,” the previously-developed HDX™ for high green densities was adopted as the lubricant, and the JIP Cleanmix™ segregation-free treatment was applied to suppress graphite powder segregation. Regarding the sintering conditions, high temperature and long time conditions of, for instance, 1 200˚C × 150 min to secure high strength by promoting sintering, and bright quenching heat treatment become preconditions.

3.2 Test Material and Experimental Method

The base powder was JIP™ 5CRA (Fe-0.5 mass%Cr-0.2 mass%Mn-0.2 mass%Mo), in which Cr, Mn, and Mo were prealloyed as the alloying elements. In order to make the 5CRA powder mixture, 4 level amounts of natural graphite powder (Mean particle size: 4 μm), namely, 0.6 mass%, 0.7 mass%, 0.8 mass%, and 0.9 mass% of graphite powder, and 0.5 mass% of HDX™ for high green densities were added to this base powder, and mixed by segregation-free treatment.

The 5CRA powder mixtures with the above-mentioned 4 levels of graphite addition were compacted into type A bar-shaped test specimens with dimensions of 55 mm × 10 mm × 10 mm at a compaction pressure of 700 MPa (Green density: 7.13–7.15 Mg/m^3), and were then sintered at 1 200˚C × 150 min in a 90 vol% N_2 + 10 vol% H_2 atmosphere. After sintering, heat treatment was conducted under the following two conditions: (1) Gas carburizing (Methanol instillation-type gas carburizing: 870˚C × 60 min, Carbon potential: 0.8 mass%, Hardening: In oil at 60˚C, Tempering: 180˚C × 60 min) and (2) Bright quenching (900˚C × 30 min, Ar atmosphere; Hardening: In oil at 60˚C, Tempering: 180˚C × 60 min) .

The tensile test and microstructural observation were conducted in the same manner as described in Section 2.2.3. Carbon contents were analyzed with some of the test specimens after sintering. The Charpy impact test was conducted using unnotched type A bar-shaped test specimens in accordance with JIS Z 2550, and the Charpy impact values were measured.

3.3 Experimental Results and Discussion

Figures 7 and **8** show the tensile strength and Charpy impact values of the test specimens prepared as described in Section 3.2, arranged by the C contents of the sintered compacts. For comparison, as an example of

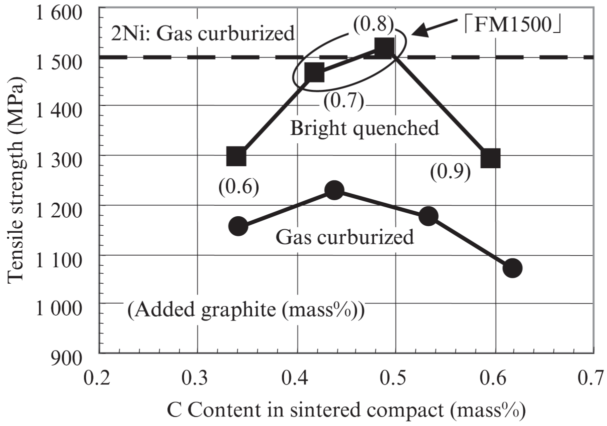

Fig. 7 Tensil estrength of the case-hardened compacts made from JIP™ 5CRA mixed with graphite and lubricant

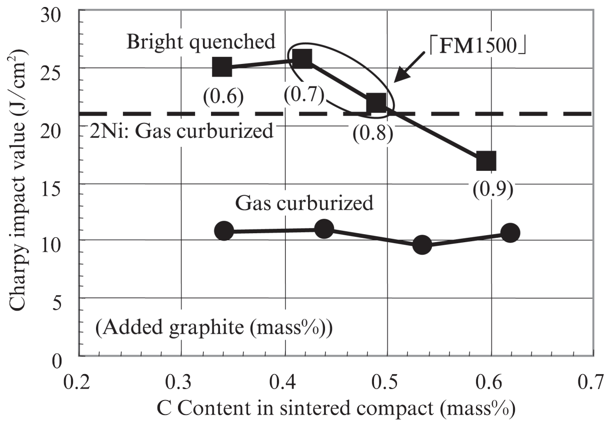

Fig. 8 Charpy impact value of the case-hardened compacts made from JIP™ 5CRA mixed with graphite and lubricant

a conventional powder with which tensile strength of 1 500 MPa grade has been obtained, the figure also shows the measured values of the gas carburized material (hereinafter, “2Ni”) of the 2Ni diffusion bonded steel powder (Fe-2 mass%Ni-1 mass%Mo) described in Reference 1). Tensile strength of 1 520 MPa, which exceeded the target of 1 500 MPa, can be obtained with the bright quenched material 0.8 mass% graphite was added in. The Charpy impact value of this bright quenched material with 0.8 mass% graphite addition is on roughly the same level as the “2Ni” gas carburized material. In applications where the impact value has a priority, the Charpy impact value can be increased by approximately 20%, while limiting the decrease in tensile strength to about 4%, by using a bright quenched material with 0.7 mass% addition of graphite. On the other hand, the tensile strength of the gas carburized materials did not achieve the target tensile strength level at any of the graphite addition, and the Charpy impact values of the gas carburized materials were also limited

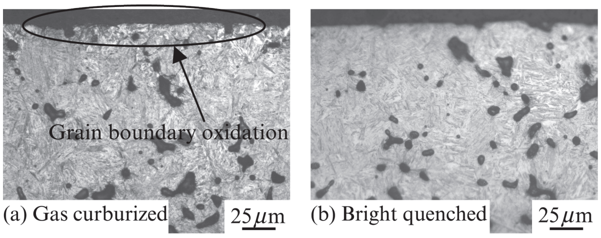

Photo 2 Microstructures of the sintered and case-hardened compacts made from FM1500

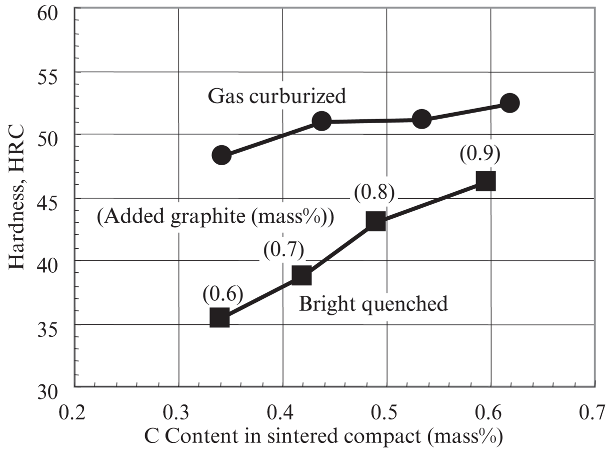

Fig. 9 Surface hardness of the case-hardened compacts made from JIP™ 5CRA mixed with graphite and lubricant

to approximately 1/2 of that of the "2Ni" gas carburized material.

Based on these results, it is possible to obtain tensile strength of 1 500 MPa grade by using a powder mixture in which 0.7–0.8 mass% of graphite is added to JIP™ 5CRA and segregation-free treatment is applied in combination with bright quenching as the heat treatment process. And then, the Charpy impact values of the obtained materials are equal or superior to that of "2Ni." Accordingly, the powder mixture in which 0.7–0.8 mass% of graphite and 0.5 mass% of the lubricant for high green densities is added to the 5 CRA and segregation-free treatment is applied is defined as "FM1500."

Among the test specimens evaluated above, **Photo 2** shows the cross-sectional microstructures of the surface layer of specimens 0.8 mass% graphite added in. Under both heat treatment conditions, the specimens have a generally homogeneous tempered martensite structure. However, slight grain boundary oxidation could be observed in the surface layer of the gas carburized material. Furthermore, similar grain boundary oxidation was also observed in all of the four gas carburized materials. As the reason why the tensile strength of the gas carburized materials is lower than that of the bright quenched materials in Fig. 7, it is estimated that the grain boundary oxidation which is seen in the surface layer of the specimens can be the generating point of fracture in the tensile test.

As the cause of grain boundary oxidation, the atmosphere in gas carburizing is more oxidizing than the dry Ar atmosphere (Dew point: Lower than −60°C) in bright quenching. In the methanol instillation-type gas carburizing adopted in this experiment, the main components of the atmosphere are CO and H_2, which are formed by the reaction $CH_3OH = CO + 2H_2$. In this process, the carbon potential is controlled by controlling the partial pressure of O_2 based on the reaction $CO = [C] + (1/2)O_2$. Because O_2 and H_2 exist in the atmosphere, H_2O (water vapor) is generated by the reaction $H_2 + (1/2)O_2 = H_2O$. The equilibrium partial pressure of water vapor in this reaction at 870°C corresponds to a dew point of approximately 14°C[8,9], which is a higher dew point than that of the atmosphere in bright quenching.

Furthermore, the results of the tensile strength in Fig. 7 show that there are peaks at a certain C content of the sintered compact in both the bright quenched material and the gas carburized material. In wrought steel, in case the material has a homogeneous tempered martensite structure, its hardness increases with the carbon content up to a C content of approximately 0.6 mass% after heat treatment[10], and tensile strength also shows a similar tendency[11]. However, in sintered materials, it has been reported that tensile strength decreases when the carbon content exceeds a certain value, even though hardness increases with the C content[12]. In actuality, as shown in **Fig. 9**, surface hardness increases monotonously with the C content in these sintered materials, whereas tensile strength displays a peak value, as shown in Fig. 7. This is explained by the fact that, when the hard martensite phase increases, notch sensibility increases, and brittle fracture occurs more easily due to the notch effect of internal pores.

On the other hand, the following can be considered regarding the C content dependence of the Charpy impact value shown in Fig. 8. First, in the bright quenched material, the Charpy impact value decreases with increasing C content in the sintered compact. In addition to the above-mentioned point that notch sensibility increases and brittle fracture occurs more easily with the hard martensite microstructure[12], the decreasing tendency of the Charpy impact value can be understood from the fact that increased C contents cause embrittlement in general steel materials[13].

Next, in the gas carburized materials, the Charpy impact values are lower than those of the bright quenched material, and virtually there is no dependence on the C content of the sintered compact. The fact that the Charpy impact values are lower than those of the bright quenched materials is considered to be due to the grain boundary oxidation which was seen in the specimen surface layer of the gas carburized material. This can be the initiation point of crack in the impact test as in the tensile test, and it can cause the decrease in the Charpy impact values. Regarding the low dependence on the C content of the sintered compact, in the gas carburized specimens, the carbon content in the surface layer part reached approximately the same 0.8 mass% as the carbon potential of the carburizing atmosphere irrespective of the C content of the sintered compact. And as a result, it is thought that the carbon content of the specimen surface layer, where cracks easily initiate due to grain boundary oxidation, determined the brittleness of the material.

4. Conclusion

Two new steel powders, "FM1300" (Tensile strength: 1 300 MPa grade) and "FM1500" (Tensile strength: 1 500 MPa grade) were developed for high strength application as JFE Steel's "FM Series" of Ni-free alloyed steel powders. The properties of these newly-developed alloyed steel powders after sintering and heat treatment were investigated. The main results are summarized below.

(1) "FM1300"

"FM1300" is a segregation-free powder produced by mixing 0.5 mass% of graphite powder and 0.5 mass% of the lubricant for high green densities with a prealloyed + diffusion bonded alloyed steel powder, in which an additional 0.15 mass% of Mo is diffusion bonded on a 0.45 mass% Mo prealloyed steel powder. Its green density when compacted at a compaction pressure of 570 MPa or higher is 7.03 Mg/m^3 or higher. Tensile strength of 1 300 MPa or higher can be obtained by sintering at 1 250°C × 60 min followed by carburizing, case-hardening and tempering.

(2) "FM1500"

"FM1500" is a segregation-free powder produced by mixing 0.7–0.8 mass% of graphite powder and 0.5 mass% of the lubricant for high green densities with a Fe-0.5 mass%Cr-0.2 mass%Mn-0.2 mass%Mo steel powder, in which Cr, Mn, and Mo are alloyed by prealloying. Green densities of 7.13 to 7.15 Mg/m^3 are achieved by compacting at a compaction pressure of 700 MPa. When sintered at 1 200°C × 150 min followed by bright quenching, tensile strength of 1 500 MPa grade can be obtained, and the Charpy impact value is also equal or superior to that of the conventional Ni-containing iron powder.

Together with the previously-developed "FM600" and "FM1000," the lineup of JFE Steel's "FM Series" have been expanded to include strength levels from 600 MPa to 1 500 MPa by the newly-developed products mentioned above. The "FM Series" is a new type of alloyed steel powders which contains absolutely no Ni. In the future, application of these "FM Series" powders to automobile engine and drive train parts is expected to expand.

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