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# Characteristics of Chromium-Containing Alloyed Steel Powders with High Wear Resistance<sup>\*</sup>



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#### **1** Introduction

The use of alloyed steel powders in sintered auto parts is increasing in response to higher auto performance requirements.<sup>1)</sup> Appropriate sintering materials are also required by the greater use of parts with both high wear resistance and strength in oil pumps and similar components in order to enhance stability and operability. In the past, carburized Cr-containing steels were frequently used as ingot materials for automotive parts in locations where both wear resistance and strength were required. In powder metallurgy, Ni-containing alloyed steel powders have been used in applica-

#### Synopsis:

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tions of this type, because the strong affinity of Cr for oxygen makes it difficult to obtain low-oxygen steel powders with high compressibility by the water atomizing method. However, nickel is an inadequate alloying element in several respects, including the fact that it reduces the amount of solid solution C in the austenite phase and leaves a soft austenite phase on the surface of parts, which is detrimental to wear resistance.

This report describes a low-oxygen Cr-containing pre-alloyed steel powder KIP 4100 V (1%Cr-0.7%Mn-0.3%Mo) which is produced by a water atomizing/ vacuum-annealing process and offers high compressibility and excellent wear resistance, and a composite-type alloyed steel powder with enhanced compressibility. The compressibility, static strength of heat-treated materials, fatigue strength, and wear resistance of both powders, and the sintering behavior of the composite-type alloyed steel powder are also discussed.

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#### 2 Low-Oxygen Cr-Containing Alloyed Steel Powder

The mechanical properties of sintered parts fundamentally depend on the sintered density of the part in question. Moreover, improved compressibility is essential not only to increase the sintered density of products, but also from the viewpoint of the life of the compacting die and the forming of parts of complex shape.<sup>2)</sup> Methods of enhancing the compressibility of alloyed steel powders include (1) reducing the content of impurities and (2) reducing solid solution hardening due to the use of alloying elements.

The compressibility of Cr-containing pre-alloyed steel powders increases as the contents of O, C and N impurities in the powder decrease. On the other hand, Cr is among the most commonly used elements for enhancing hardenability after Mn and Mo, and together with Mn, is widely employed in ingot materials. Figure 1 shows the ideal critical diameter  $D_{\rm I}$  which expresses the hardenability of sintered and forged materials obtained by controlling the contents of oxygen and added graphite in Cr-containing alloyed steel powders. The value of  $D_1$  increases as the oxygen content decreases regardless of the carbon content, thus providing better hardenability, which indicates that it is necessary to reduce the oxygen content of Cr-containing steel powders for heavy-duty applications from the viewpoint of hardenability.<sup>3)</sup> Kawasaki Steel has succeeded in commercially producing a Cr-containing alloyed steel powder with low contents of O, C, and N and excellent compressibility and hardenability, using a water atomizing/vacuumannealing process developed by the company's researchers.<sup>3)</sup>



Fig. 1 Effect of C and O contents on ideal critical diameter  $D_1$  of Cr-containing powder-forged steels

#### 3 Features of Carburized Materials Made from Cr-Containing Pre-Alloyed Steel Powder

Although Cr-containing steel powders are frequently used in applications requiring wear-resistance with carburizing treatment, a special atmosphere is required because Cr has a strong affinity for both oxygen and carbon. Moreover, the conventional hydrogen-based sintering atmosphere has in recent years been increasingly replaced by a nitrogen-based atmosphere to achieve energy savings. This chapter will therefore discuss the relationship of sintering and heat treatment atmospheres to the static strength of sintered compacts with respect to two types of sintering atmospheres and three levels of carburizing atmospheres, as detailed in Sec. 3.1 below.

#### 3.1 Experimental Method

The O, C, and N contents of KIP 4100V steel powder (1%Cr-0.7%Mn-0.3%Mo) are compared with those of a conventional gas-reduced steel powder in **Table 1**. Samples were produced by blending the steel powder with 0.15% graphite and 1% zinc stearic acid, forming under a pressure of 686 MPa, and sintering for 60 min at 1 523 K in atmospheres of  $75\%H_2-25\%N_2$  and  $90\%N_2-10\%H_2$ . Carburizing was performed for 30 min at 1 203 K in atmospheres with carbon potentials of 0.7, 0.9, and 1.1%. After quench hardening in oil, the specimens were tempered for 60 min at 453 K.

Table 1 C, O and N contents of KIP 4100V (%)

	0	С	Ν		
KIP 4100V	0.10	0.02	0.001		
KIP 4100	0.58	0.05	0.006		

#### 3.2 Results and Discussion

#### 3.2.1 Compressibility of KIP 4100V steel powder

The compressibility of KIP 4100V steel powder is shown in Fig. 2. In comparison with the conventional gas-reduced steel powder (KIP 4100), the O, C, and N contents of KIP 4100V have been reduced, and its compressibility has improved by  $0.14 \text{ Mg/m}^3$ .

## 3.2.2 Effect of carbon potential in carburizing atmosphere

**Table 2** shows the tensile strength and absorbed energy values of carburized compacts with a sintered density of 7.11 Mg/m<sup>3</sup> after sintering in a 75%H<sub>2</sub>-25%N<sub>2</sub> atmosphere. Both tensile strength and absorbed energy show higher values when the carbon potential of the carburizing atmosphere is reduced, reaching maxi-

Table 2 Tensile strength and absorved energy of the sintered, carburized and tempered compacts

Compacting and sintering conditions		Carbon potential	Tensile strength	Absorbed energy	
Graphite addition (%)	Compacting pressure (MPa)	Sintering atmosphere	(%)	(MPa)	(J)
0.15	686	$75\% H_2 - 25\% N_2$	0.7 0.9 1.1	1100 1090 990	8 7 6
0.15	686	10%H2 - 90%N2	0.7 0.9 1.1	1180 1160 970	8 7 7



Fig. 2 Compressibility of KIP 4100V

Table 3 Core hardness of the sintered, carburized and tempered compacts (compacted with 0.15% graphite at 686 MPa and sintered in 75%H<sub>2</sub>-25%N<sub>2</sub>)

Carbon potential (%)	Core hardness (HRC)
0.7	27
0.9	28
1.1	30

mum respective values of 1 100 MPa and 7.6 J at a carbon potential of 0.7%. The fact that carburizing is more effective at lower carbon potentials is attributed to the strong affinity of Cr for C. **Table 3** shows the core hardness of the carburized compacts. Hardness increases at higher carbon potentials, and carburizing proceeds more easily to the core. For this reason, absorbed energy values increase as the carbon potential is decreased.

#### 3.2.3 Effect of sintering atmosphere

Table 2 also shows the tensile strength and absorbed energy values of carburized compacts with a sintered density of 7.11 Mg/m<sup>3</sup> after sintering in a  $90\%N_2$ -10%H<sub>2</sub> atmosphere. Both the tensile strength and absorbed energy values are comparable to or better than those of the previously mentioned compacts sin-

tered in the  $75\%H_2-25\%N_2$  atmosphere, demonstrating that KIP 4100V can be effectively sintered in an energy-saving  $90\%N_2-10\%H_2$  atmosphere.

#### 4 High-Compressibility Cr-Containing Composite-Type Alloyed Steel Powder

To enhance the compressibility of Cr-containing steel powders, the contents of O, C, and N impurities in the powder were lowered by vacuum annealing. Practical methods of increasing compressibility by reducing the solid solution hardening caused by alloying elements have been developed with Cr-containing low-alloy steel powders. These include (1) reduction in the content of pre-alloy<sup>4)</sup> and (2) use of the composite alloying technique, in which alloying components are made to adhere to the surface of pure iron powder with high compressibility.<sup>5)</sup> Cr composite-type alloyed steel powders make use of the high compressibility of Cr prealloyed steel powder produced by water atmizing and vacuum annealing, and add to this material Mo as a composite alloying element.

This chapter will discuss a Cr-containing compositetype alloyed steel powder which offers significantly higher compressibility than existing low-oxygen pre-alloyed steel powders and shows excellent tensile strength and wear resistance in sintered compacts. The compressibility, features of heat-treated specimens, and sintering behavior of this material are also described.

#### 4.1 Principle of Cr-Containing Composite-Type Alloyed Steel Powder

Low-alloy sintered steels containing Cr have been produced by blending ferrochrome powder with pure iron powder<sup>6,7</sup>, or using the Cr-containing pre-alloyed steel powders produced by water atomizing and vacuum reduction method described in the previous chapters. However, the Cr in high-Cr ferrochrome is susceptible to oxidation during sintering, and with pre-alloyed steel powders, there is an upper limit to improved compressibility due to the solid solution hardening associated with alloying elements. A new Cr-containing compositetype alloyed steel powder was developed to solve these

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Fig. 3 Alloying methods for steel powders

problems.

In this composite-type alloyed steel powder, as shown in **Fig. 3**, pre-alloying is used with elements having little solid solution hardening effect relative to ferrite, while composite alloying is used with the other alloying elements. This selection of techniques makes it possible to obtain significantly higher compressibility than is possible with conventional pre-alloyed powders.

Chromium is superior to Ni and Mn as a pre-alloying element with a small solid solution hardening effect. Moreover, Cr contributes to better hardenability when the sintered steel is heat treated for high strength. While offering high hardenability, following Mn and Mo, Cr is also desirable for preventing oxidation during sintering and heat treatment, as it has a weaker oxygen affinity than Mn. Pre-alloying is also desirable for preventing oxidation, because when alloying elements are pre-alloyed in an iron powder, their activity decreases at lower concentrations, reducing their affinity for oxygen.

The content of pre-alloyed Cr is determined by the compressibility and reducibility of the steel powder. The compressibility of Cr-containing pre-alloyed steel powders produced by the water atomizing/vacuum annealing process, as shown in Fig. 4, increases as the Cr content decreases, making it possible to obtain a density of 7.18 Mg/m<sup>3</sup> or higher when the Cr content is held to no more than 1%. The content of pre-alloyed Cr was set at 1% in the composite-type alloyed steel powder under discussion here.

On the other hand, as a composite-type alloying ele-



Fig. 4 Compressibility of Cr prealloyed powders

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ment, Mo shows a weaker affinity for oxygen than Cr and is superior in improving hardenability. When Mo is composite alloyed, the compressibility of the material shows no change in the range of 1-2% Mo. The content of composite-alloyed Mo was therefore set at 1% in consideration of the mechanical properties of sintered compacts.

#### 4.2 Experimental Method

Table 4 shows the chemical composition of the steel powders used in experiments. Powder A is a compositetype alloyed steel powder in which 1% Mo was composite-alloyed to a 1% Cr pre-alloyed steel powder produced by the water atomizing/vacuum annealing process. The comparison materials were powder B (the previously discussed KIP 4100V), which was also produced by the water atomizing/vacuum annealing process, and powder C, a high-compressibility Ni-containing composite-type alloyed steel powder in which 4% Ni, 0.5% Mo, and 1.5% Cu were composite alloyed onto the surface of particles of pure iron powder. As shown in Fig. 5, the 1%Cr-1%Mo composite-type alloyed steel powder A showed a compressibility 0.10 Mg/m<sup>3</sup> or more higher than that of the pre-alloyed powder B, and was thus comparable to the high-compressibility Ni-containing composite-type alloyed steel powder in this regard.

Set amounts of graphite and 1% zinc stearic acid were added to the steel powders, which were compacted at 686 MPa and sintered for 60 min at 1 523 K in a

 Table 4 Chemical compositions and methods of the studied powders
 alloying (%)

Powder	Cr	Mo	Mn	Ni	Cu	Alloying method
A	1	1	_	—		Modified composite-type al- loying (Cr prealloying and Mo composite-type alloying)
В	1	0.3	0.7			Prealloying
С	-	0.5		4	1.5	Composite-type



Fig. 5 Compressibility of 1%Cr-1%Mo modified composite-type alloyed steel powder

75%H<sub>2</sub>-25%N<sub>2</sub> atmosphere. Tensile strength and absorbed energy tests were conducted in the as-sintered condition with compacts containing 0.8, 1.0, and 1.2% added graphite. The same tests were also performed with compacts containing 0.4, 0.6 and 0.8% added graphite after sintering, bright quenching in oil from 1 143 K, and tempering. Tensile strength, absorbed energy, fatigue, and wear tests were conducted with specimens containing 0.15% graphite after sintering, carburizing, and tempering. Carburizing was performed for 30 min at 1 203 K, at a carbon potential of 0.7%. The tempering temperature was 453 K in all cases in which heat treatment was applied.

The Ono rotating bending fatigue test and Mori contact fatigue test were conducted to determine the endurance fatigue strength of the compacts. After a wear test using an Ohgoshi type wear testing apparatus, the wear volume relative to the wear distance was obtained as a basis for evaluating wear resistance.

The dimensional change behavior of a test piece with 0.6% added graphite was measured using an optical thermal expansion meter. The microstructure of sintered compacts cooled immediately after heating to a temperature of 1 423 or 1 523 K, and sintered compacts bright-quenched and tempered after sintering for 60 min at 1 523 K were examined using an optical microscope and microbeam X-ray diffraction. The microstructure of the carburized and tempered materials was also examined by ordinary X-ray diffraction.

#### 4.3 Results and Discussion

### 4.3.1 Features of bright-quenched materials of composite-type alloyed steel powder

Figure 6 shows the tensile strength of sintered compacts and bright-quenched and tempered specimens of composite-type alloyed steel powder A and prealloyed steel powder B. Figure 7 shows the toughness of the same materials. In both the sintered compacts and the heat-treated specimens, a higher tensile strength was obtained with powder A than with powder B at the same graphite addition, while the absorbed energy values were substantially the same with the two powders. A tensile strength of 1 420 MPa was obtained with the heat-treated material, which was 160 MPa higher than the value with KIP 4100V. Table 5 shows the oxygen content, C content of sintered compacts with a 0.6% graphite addition, green density, and sintered density with steel powders A and B. The sintered density of the powder A compacts was high due to the good compressibility of this material. Moreover, the steel powder oxygen content of powder A was lower than that of powder B. Consequently, the oxygen content of the powder caused only a slight decrease in C during sintering of the powder A compacts, making it possible to obtain a higher C yield with the same amount of added graphite. The microstructure of the bright-quenched



Fig. 6 Relationship between the graphite addition and tensile strength with and without brightquenching and tempering



Fig. 7 Relationship between the graphite addition and absobed energy with and without brightquenching and tempering

Table 5	Green density, sintered density, oxygen con-
	tent of the original powders, and carbon con-
	tent of the sintered compacts (compacted at
	686 MPa with 0.6% graphite)

Powder	Green density (Mg/m³)	Sintered density (Mg/m³)	O content of powder (%)	C content of sintered compact (%)
A (1% Cr-1% Mo)	7.05	7.05	0.10	0.47
B (1% Cr-0.3% Mo-0.7 Mn)	6.94	7.02	0.16	0.40

and tempered materials is shown in **Photo 1**. The structure obtained by applying an optical microscope to a nital-etched specimen shows some shading, but the results of microbeam X-ray diffraction showed a martensitic structure which was uniform and without variation in all cases.

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Photo 1 Photomicrograph of the compact of powder A sintered at 1 523 K with 0.6% graphite and bright-quenched and tempered

Thus, it may be concluded that powder A provides higher tensile strength than powder B at the same addition of graphite due to the high compressibility, higher yield of added C, and more uniform sintered structure with powder A.

#### 4.3.2 Features of carburized materials of compositetype alloyed steel powder

As can be seen in **Table 6**, the carburized and tempered specimen produced from powder A showed higher tensile strength and absorbed energy values than the powder B specimen. The carburized and tempered specimen of powder A was also superior to that of powder B in rotating bending fatigue strength and contact fatigue strength, as shown in **Table 7**. These results were attributed to the fact that the sintered density of

Table 6 Mechanical properties of sintered, carburized and tempered compacts

Powder	Tensile strength (MPa)	Absorbed energy (J)
A (1% Cr-1% Mo)	1120	9.4
B(1% Cr-0.3% Mo-0.7% Mn)	1100	7.6

 Table 7 Fatigue properties of sintered, carburized and tempered compacts

Pourder	Sintered	Surface	Fatigue endurance limit (MPa)		
1 Uwder	(Mg/m <sup>3</sup> )	Mg/m <sup>3</sup> ) (HRC)		Contact	
A (1% Cr-1% Mo)	7.15	36	440	2370	
B (1% Cr-0.3% Mo-0.7% Mn)	7.11	48	410	2260	

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Fig. 8 Abrasive wear resistance of sintered, carburized and tempered compacts of powders A, B and C

high-compressibility powder A is higher than that of powder B, which is significant because contact fatigue strength is more strongly dependent on density than on surface hardness.<sup>8)</sup>

The wear resistance of Cr-containing steel powder A is comparable to that of powder B, and more than 100 times better than that of the Ni-containing steel powder C, as shown in Fig. 8. This difference is explained by the 9% residual content of austenite in the carburized and tempered microstructure of the powder C specimen, in contrast to the smaller 5-6% austenite content with powders A and B. Austenite is soft and inferior in wear resistance, and forms as a high-Ni residual austenite phase with powder C, which contains 4% composite-alloyed Ni. (This problem does not occur with powders A and B, which do not contain Ni.) Moreover, because the affinity of Cr for C is stronger than that of Ni, some improvement in wear resistance can be attributed to the presence of Cr carbides.

## 4.3.3 Sintering behavior of composite-type alloyed steel powder

**Table 8** shows the dimensional changes in sintering with composite-type powder A, a mixed powder of the same composition as powder A, and powder B, together with the standard deviation of such dimensional changes, which is an index of the degree of variation. In contrast to the dimensional change with powder B com-

Table 8	Dimensional	change	of	sintered	compacts
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Powder	Alloying method	Dimensional change (%)	Standard deviation of dimensional change (%)
A (1% Cr-1% Mo)	Modified composite-type	0.08	0.02
A (1% Cr-1% Mo)	Mixing	0.09	0.05
B (1% Cr-0.3% Mo-0.7% Mn)	Pre-alloying	-0.52	0.03



(a) Quenched from 1423K
 (b) Quenched from 1523K
 Photo 2 Photomicrograph of compacts made from powder A with 0.6% graphite quenched from 1 423 K(a) and 1 523 K(b)

pacts, which show a considerable shrinkage of -0.53%, the compacts of powder A and the mixed powder of the same composition were extremely stable, showing dimensional change of virtually 0%. Because powder A is a composite alloyed powder, the deviation in dimensional change in sintered compacts of this material is small, being comparable to that of the pre-alloved steel powder B and less than 1/2 that with the mixed powder of the same composition. It should be emphasized that this kind of stability in dimensional changes is an extremely advantageous feature for the manufacture of parts of complex shape. Photo 2 shows the microstructure of specimens produced by heating and then quenching green compacts of powder A with 0.6% added graphite powder from temperature of 1 423 K and 1 523 K. In Photo 2 (a), the specimen quenched from 1 423 K shows the growth of a white phase, which was detected as M<sub>6</sub>C type carbide by microbeam X-ray diffraction (Fig. 9). M<sub>6</sub>C type carbides were absent in the specimen quenched from 1 523 K, which is shown in Photo 2 (b), and an Mo-rich white structure has penetrated the grain boundaries, causing the pores to spheroidize. The results of an X-ray diffraction analysis of the white and black structures in Photo 2 (b) are shown in Fig. 9 (b) and (c). Although no austenite was observed in the black structures, martensite and austenite were found in the white structures,

From these finding, it can be inferred that  $M_6C$  type carbides and a liquid phase which forms in the temperature range of 1 423 to 1 523 K transform into  $\gamma$ Fe, and transient liquid-phase sintering occurs.<sup>9)</sup> However, because the amount of expansion in the vicinity of 1 523 K is smaller with the 1.5%Mo-0.7%C composition than the previously reported figure of approximately 0.6%,<sup>10)</sup> it may be inferred that only a small amount of liquid phase is formed in the present case. For this reason, the amount of dimensional change in the sintered compact was a very small 0.08%. To summarize the preceding discussion, the appearance of a transient liquid phase in the Cr-containing composite-type alloy steel powder A promotes diffusion of Mo and spheroidizing



Fig. 9 Microbeam X-ray diffraction patterns of the white phase shown in Photo 2(a) and of the white and black areas shown in Photo 2(b)

of the pore, which suppresses shrinkage during sintering. On the other hand, because the amount of the liquid phase is slight, dimensional change in sintering is extremely small.

#### 6 Conclusions

An investigation was made of the features of carburized compacts made from low-oxygen Cr-containing

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pre-alloyed steel powder KIP 4100V, which is produced by the water atomizing/vacuum annealing process and offers excellent compressibility. A study was also made of the features of heat-treated compacts and the sintering behavior of a Cr-containing composite-type alloy steel powder developed for higher compressibility.

- (1) The tensile strength of carburized specimens of KIP 4100V sintered in a hydrogen-based atmosphere increased when the carbon potential of the carburizing atmosphere was decreased, and a value of 1 100 MPa was obtained at a carbon potential of 0.7%. Higher values were obtained when the compacts were sintering in a nitrogen-based atmosphere.
- (2) With a 1%Cr-1%Mo composite-type alloyed steel powder, a green density of 7.18 Mg/m<sup>3</sup> was obtained at a compacting pressure of 686 MPa; this value was more than 0.10 Mg/m<sup>3</sup> higher than that with the pre-alloyed material KIP 4100V.
- (3) A tensile strength of 1 420 MPa was obtained by bright-quenching specimens produced from the composite-type alloyed steel powder.
- (4) The wear resistance of the composite-type alloyed steel powder specimens was comparable to that of KIP 4100V specimens, and more than 100 times better than that of Ni-containing composite-type steel powder samples.
- (5) With the composite-type alloyed steel powder containing 0.6% added graphite, a small amount of transient liquid-phase sintering occurred between the temperatures of 1 423 and 1 523 K, suppressing shrinkage during sintering and limiting dimensional change to a very small value.

The authors believe that the development of the new Cr-containing alloyed steel powder discussed herein will contribute to the expanded use of materials of this type in sintered parts, particularly in heavy-duty, wear-resistant applications.

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