Abridged version

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Properties of High Compressibility Composite-Type Alloy Steel Powders, "KIP SIGMALOY"*

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1 Introduction

Ferrous powder metallurgy has been extensively applied to heavy duty mechanical parts mainly for the automobile industry[1]. These applications essentially require increased addition of alloying elements for strengthening, and improved compressibility of powders to increase the sintered density[2].

Two alloying methods have been employed to date: mixing elemental powders and prealloying. The mixing method is favored for its simplicity, since a single iron powder can be used in various alloy compositions, giving the mixed powders high compressibility. The method is, however, unfavorable in that the alloying elements are liable to segregate and remain unalloyed after sintering, hence degrading the mechanical properties of sintered compacts products. Decreased particle size of alloying elements may improve diffusion during sintering, but often worsens the consistency of sintered properties due to further segregation and poor flowability of the mixed powders during filling into compacting dies. The prealloying method, in contrast, is superior in the homogeneity of alloying elements in sintered compacts but generally decreases compressibility of the powders. Although the compressibility of prealloyed steel powders has recently been improved to a great extent[3, 4], the amount of alloying elements should be limited in order to maintain compressibility.

Composite-type alloy steel powders **KIP SIGMALOY**, developed by Kawasaki Steel to solve the problems encountered with conventional powders, combine the compressibility of simple mixed powders and the homogeneity of prealloyed powders. The improved characteristics are based on a new alloying method in which fine particles of alloying elements are metallurgically bonded

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to the surface of soft iron particles.

The present paper compares the characteristics of composite-type alloy steel powders containing Cu, as well as Ni, Cu, and Mo, with those of mixed and prealloyed powders, and describes the effect of alloying methods and alloying elements on the properties of sintered compacts made from these powders.

2 Principle of Composite-Type Alloying

Composite-type alloy steel powders are produced by annealing mixtures of iron powders and powders of alloying elements in a reducing atmosphere. In this process the alloying elements are metallugically bonded to the surface of the iron particles. An optimal selection of raw materials and annealing conditions is essential if both compressibility and homogeneity requirements are to be satisfied. Starting iron powders should be highly compressible. Subsieve sized powders of alloying elements are used in order to assure high diffusivity and homogeneity in sintered compacts. Annealing conditions should be precisely controlled in accordance with alloy composition. An excess degree of diffusion decreases compressibility, while insufficient diffusion lowers flowability and homogeneity.

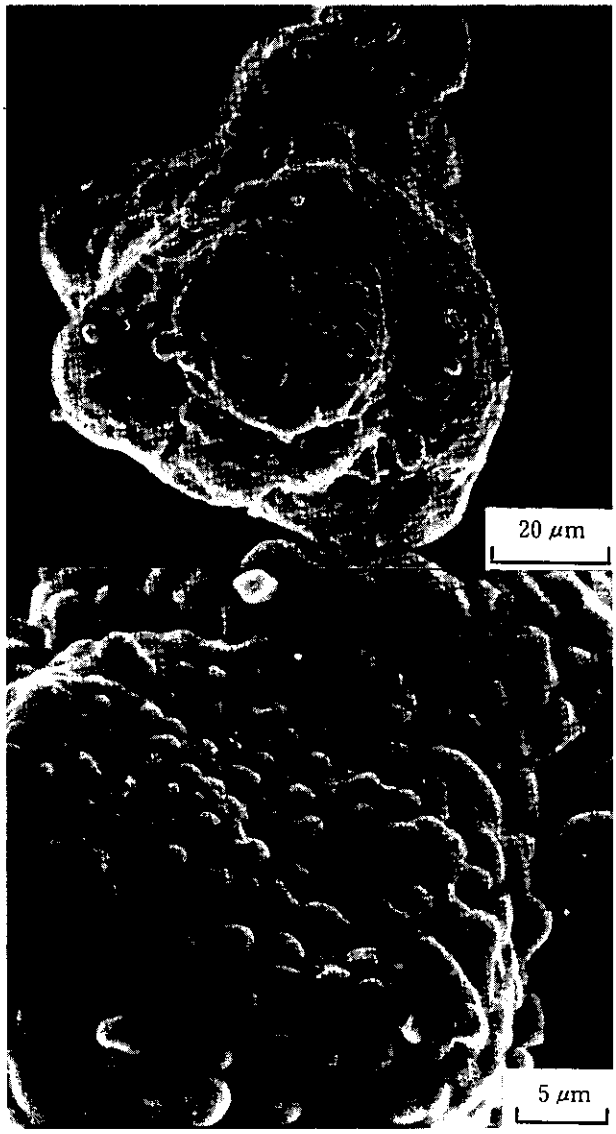


Photo 1 Scanning electron micrographs of particle of SIGMALOY Cu (2%Cu)

Table 1 Chemical composition of SIGMALOY powders (%)

	Ni	Cu	Mo
SIGMALOY Cu	—	2~20	—
SIGMALOY 215	2	1.5	0.3
SIGMALOY 315	3	1.5	0.5
SIGMALOY 415	4	1.5	0.5

Photo 1 shows, as an example, the surface of a particle of composite-type alloyed KIP SIGMALOY Cu(2%Cu). The Cu particles which are diffusion-bonded to the surface of iron particles are about 2μm in size, and are much finer than the Cu powders sized several tens of micrometers usually used for conventional mixing. The KIP SIGMALOY product series consists of KIP SIGMALOY Cu as well as KIP SIGMALOY 215, 315 and 415, with the respective chemical compositions shown in **Table 1**.

3 Properties of Composite-Type Alloy Steel Powders Containing Copper

3.1 Objectives

Copper is the most widely used alloying element in ferrous powder metallurgy. Copper is usually added to iron powders by a simple mixing method, but this often results in poor dimensional accuracy and mechanical properties due to segregation of Cu and so-called "copper growth"[5] during sintering.

These problems can be solved by the composite-type alloying of Cu, as demonstrated by products of KIP SIGMALOY Cu, which contain up to 20%Cu. In the following, the properties of a composite-type alloyed Fe-2%Cu powder will be compared with those of a conventional powder mixture in terms of powder characteristics, sintering behaviors, and properties of sintered compacts, and the factors resulting in the improvements attained by the composite-type alloying will be discussed.

3.2 Experimental Procedures

A composite-type powder SIGMALOY Cu, containing 2%Cu and based on a water atomized iron powder, was used. A conventional mixture of Fe-2%Cu from the same base iron powder and an electrolytic Cu powder sized under 150 mesh were examined for comparison. The powders were mixed with 1% zinc stearate, compacted at 490 MPa, and sintered at 1120°C for 30 min in dissociated ammonia. Dimensional changes during sintering and transverse rupture strength of sintered compacts, as well as the standard deviations of these properties, were measured. The sintering behavior was examined by observing the distribution of Cu and pores in compacts fully sintered or heated only to 950°C or 1050°C, as well as by measuring the dilatometric curves for compacts of 55-mm length during heating.

3.3 Results and Discussion

Table 2 shows apparent density, flow rate, and green density of the Fe-2%Cu powders. The composite-type powder maintains good flowability even with the addition of a lubricant, although it includes fine Cu particles as shown in Photo 1. The lubricant is believed to be held tightly by the irregular particle surface of the composite-type powder. The superior compressibility of the composite-type powder can be attributed to the effect of annealing during composite-type alloying.

Table 2 Apparent density, flow rate, and green density of powders used for Fe-2%Cu compacts

Powder	Apparent density (Mg/m^3)	Flow rate (sec/50 g)	Green density* (Mg/m^3)
Composite-type Fe-2%Cu	2.95	20.6	—
Base Fe Powder	3.03	20.6	—
Composite-type Fe-2%Cu+1%Zn-St**	3.10	26.4	6.93
Mixed powder Fe-2%Cu+1%Zn-St**	3.07	29.0	6.87

* Compacted at 490 MPa

** Zn-St: Zinc stearate as lubricant

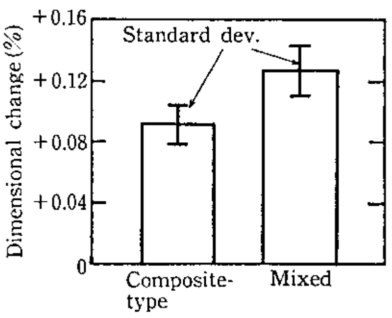


Fig. 1 Dimensional change of Fe-2%Cu compacts sintered at 1 120°C for 30 min

Figure 1 shows the dimensional change of Fe-2%Cu compacts during sintering. Composite-type alloying suppresses the growth and reduces the scatter of dimensional change down to 70% of that with simple mixing, hence improving dimensional accuracy. **Figure 2** compares the dilatometric curves during heating. The powder mix compact grows abruptly at the melting point of Cu(1083°C), while the composite-type powder compact begins to grow earlier at about 900°C, and dilatation at the melting point is kept smaller. **Photo 2** shows the distributions of Cu in Fe-2%Cu compacts heated to 950°C and 1050°C, both of which are lower than the melting point of Cu. In the composite-type powder compact, Cu has already begun to diffuse at 950°C and has reached fairly homogeneous distribution at 1050°C, while the powder mix compact still contains large Cu

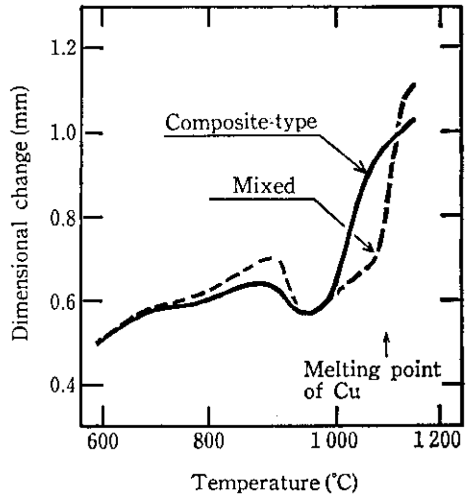


Fig. 2 Dilatometric curve of Fe-2%Cu compacts during heating

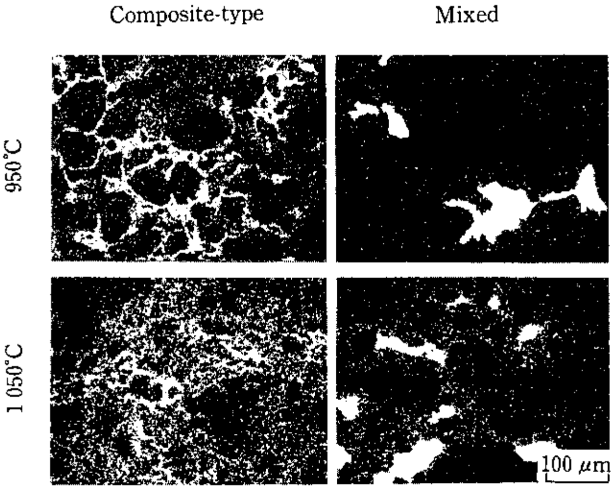
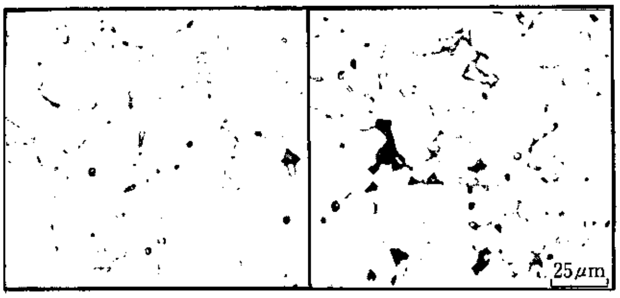


Photo 2 Copper distribution in Fe-2%Cu compacts heated up to 950°C and 1 050°C



Composite-type Mixed

Photo 3 Pore distribution in Fe-2%Cu compacts sintered at 1 120°C for 30 min

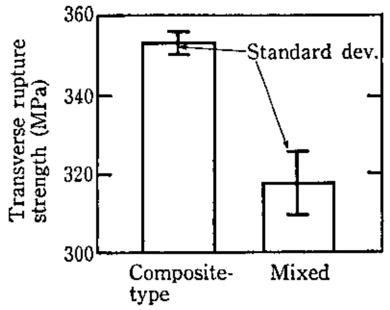


Fig. 3 Transverse rupture strength of Fe-2%Cu compacts sintered at 1 120°C for 30 min

particles even at 1050°C.

The phenomenon of Cu growth during sintering of Fe-Cu is considered a result of the penetration of liquid Cu into the particle boundaries and grain boundaries of the iron[6]. In the powder mix compacts, in which Cu hardly diffuses under the melting point of Cu, liquid Cu appears abruptly at the Cu melting point and rapidly penetrates into the particle boundaries and grain boundaries of the iron, causing sudden growth. In the composite-type powder compact, in which Cu diffuses to some extent below the melting point, abrupt growth is suppressed, in a way similar to that observed in the sintering of Cu-plated iron powders[7,8].

As shown in **Photo 3**, large pores remain in the powder mix compact as a result of Cu particles which have melted and flowed out. In contrast, fine pores are uniformly distributed in the composite type powder compact.

The difference in pore size distributions results in a difference in the mechanical properties of sintered compacts. As shown in **Fig. 3**, the composite-type powder improves transverse rupture strength by 10% when compared with the simple mixture. It also decreases the scatter of transverse rupture strength down to less than one third.

The above described composite-type alloy steel powders, SIGMALOY Cu, make possible the production of high precision sintered parts which were formerly difficult to produce from simply mixed powders. The production technology allows SIGMALOY Cu to contain up to 20% of Cu. Such powders of high Cu content can also be used as a mother alloy powder in mixing with iron powders as a substitute for the usual Cu powders.

4 Properties of Composite-Type Alloy Steel Powders Containing Ni, Cu, and Mo

4.1 Objectives

High strength sintered materials require further alloying elements such as Ni in addition to Cu. Nickel is more difficult to diffuse during sintering than Cu, since no melt forms at sintering temperatures. Therefore the strengthening effect of Ni cannot be attained effectively by a simple mixing method. Prealloying of Ni beyond 2% decreases compressibility and is not suitable for obtaining high density parts. SIGMALOY 215, 315, and 415 have been developed in order to solve these problems of Ni alloying. These composite-type powders contain Ni, Cu, and Mo as shown in Table 1. The compressibility of the powders represented in **Fig. 4** is considerably higher than that of a prealloyed powder (1.5%Ni-0.5%Cu-0.5%Mo, type 4600) although the former powders include higher contents of alloying elements, and is equal to or higher than that of pure iron powders.

The following describes the effect of chemical compositions and alloying methods on the properties of sintered and carburized compacts made from steel powders containing Ni, Cu, and Mo, and discusses the advantages of composite-type alloying.

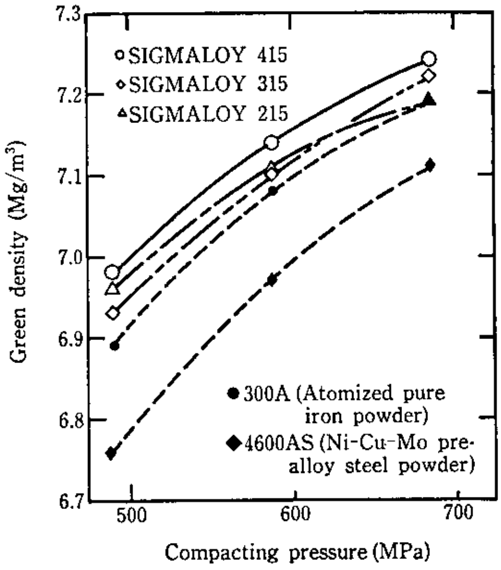


Fig. 4 Compressibility of Ni-Cu-Mo composite-type steel powders SIGMALOY 215, 315 and 415

Table 3 Chemical composition of powders used for Fe-Ni-Cu-Mo compacts (%)

Grade	Ni	Cu	Mo
Mixture	4	1.5	0.5
Composite-type	2~4	1.5~3.5	0.3~0.6
Prealloy	1.45	0.53	0.47

4.2 Experimental Procedures

Composite-type powders containing 2 to 4%Ni, 1.5 to 3.5%Cu and 0.3 to 0.6%Mo were prepared from a water atomized iron powder and powders of alloying elements. The operational condition for composite-type alloying remained the same for these powders. Additional composite-type powders with varied degrees of alloying were prepared for a constant composition of 4%Ni-1.5%Cu-0.5%Mo by altering operational conditions. Further powder samples containing 4%Ni-1.5%Cu-0.5%Mo were prepared, in which some elements were composite-type alloyed and others were simply mixed. A simply mixed powder of the same composition was made from the same base iron powder used for composite-type powders. A prealloyed powder KIP 4600(1.5%Ni-0.5%Cu-0.5%Mo) was examined for comparison. The chemical compositions of these powders are summarized in **Table 3**.

Steel powders thus prepared were mixed with 1% zinc stearate as a lubricant, compacted at 690 MPa, and sintered at 1250°C for 60 min in dissociated ammonia. The sintered specimens were carburized at 900°C with an atmospheric carbon potential of 0.9%, oil-quenched and tempered at 180°C, and subjected to measurements of mechanical properties, metallographic observation and quantitative analysis of microstructures by X-ray diffraction.

4.3 Results and Discussion

Figure 5 shows the mechanical properties of sintered and heat-treated compacts made from powders of a fully or partly composite-type alloyed or simply mixed type. Composite-type alloying of Ni increases both strength and toughness by more than 10% in comparison with the simply mixed type. Copper increases strength and toughness, while Mo increases only toughness.

Photo 4 shows the distribution of Ni-rich areas in sintered and heat-treated compacts made from mixed (4%Ni-1.5Cu-0.5%Mo), composite-type alloyed (4%Ni-1.5%Cu-0.5%Mo), and prealloyed (1.5%Ni-0.5%Cu-0.5%Mo) materials. The sintered compact made from the composite-type powder obviously gives better homogeneity when compared with the powder mix compact. The difference in mechanical properties shown in Fig. 5 is considered due to the difference in homogeneity after sintering9).

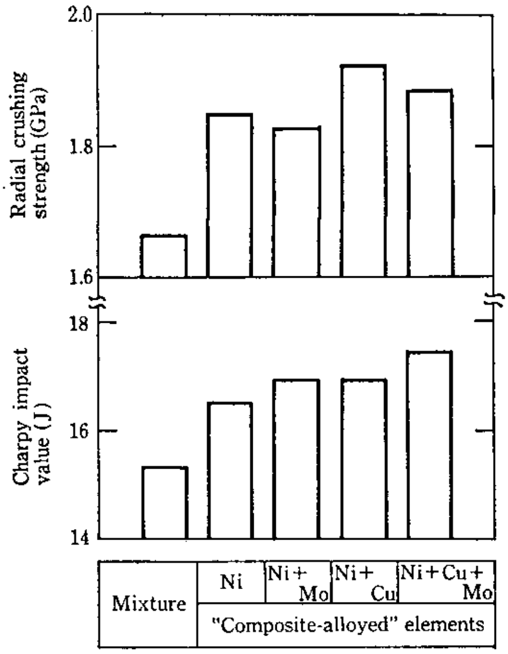
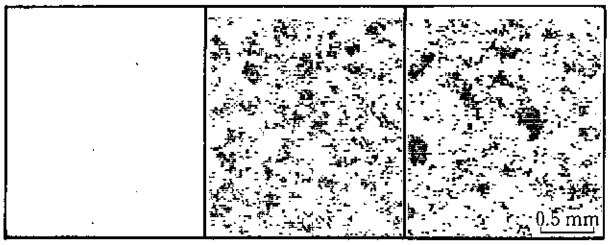


Fig. 5 Sintered and heat-treated properties of composite-type and mixed powders (4%Ni-1.5%Cu-0.5%Mo)



Prealloy (1.5%Ni-0.5%Cu-0.5%Mo)
Composite-type (4%Ni-1.5%Cu-0.5%Mo)
Mixture (4%Ni-1.5%Cu-0.5%Mo)

Photo 4 Electron-probe macro-analysis of sintered and heat-treated compacts (Black area corresponds to high concentration of Ni)

The degree of metallurgical bonding in composite-type powders can be varied by altering annealing conditions. A higher degree of bonding increases homogeneity in sintered compacts and hence improves mechanical properties. An excess degree of alloying, however, degrades the compressibility of powders. These relationships are represented in **Fig. 6.** It is essential to optimize and exactly control the conditions of composite-type alloying.

Figures 7 and 8 show the tensile strengths and impact values of sintered and heat-treated compacts made from composite-type powders containing 2 to 4%Ni, 1.5%Cu, and 0.5%Mo. Both properties increase with increased Ni

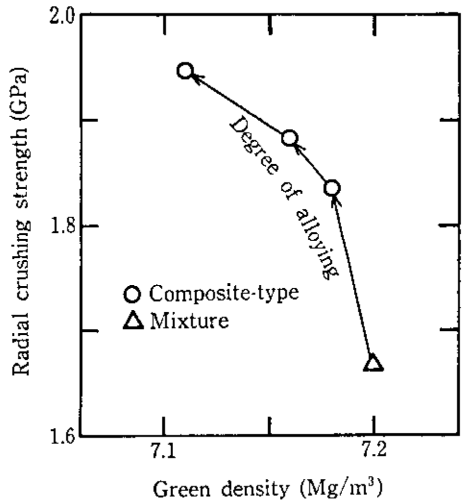


Fig. 6 Effect of degree of alloying on compressibility and radial crushing strength of sintered and heat-treated compacts (4%Ni-1.5%Cu-0.5%Mo)

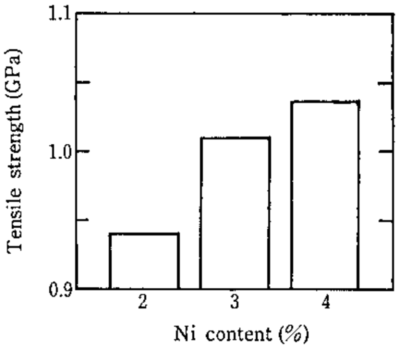


Fig. 7 Tensile strength of sintered and heat-treated compacts made from 2 to 4%Ni-1.5%Cu-0.5%Mo composite-type alloy steel powders

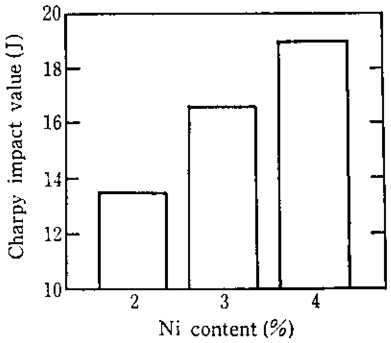


Fig. 8 Impact value of sintered and heat-treated compacts made from 2 to 4%Ni-1.5%Cu-0.5%Mo composite-type alloy steel powders

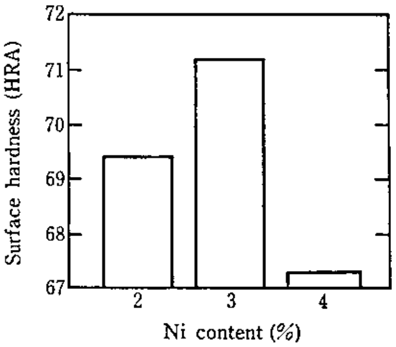


Fig. 9 Surface hardness of sintered and heat-treated compacts made from 2 to 4%Ni-1.5%Cu-0.5%Mo composite-type alloy steel powders

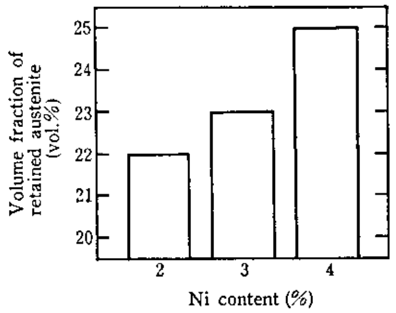


Fig. 10 Concentration of retained austenite in the surface area of sintered and heat-treated compacts made from 2 to 4%Ni-1.5%Cu-0.5%Mo composite-type alloy steel powders

content. However, hardness of the surface of sintered and heat-treated compacts shows a maximum at 3%Ni and decreases with further increase in Ni up to 4% (**Fig. 9**). This decrease in hardness is attributable to an increased amount of retained austenite near the surface of the carburized compacts, as seen in **Fig. 10**. **Photo 5** shows the microstructure and distributions of alloying elements in a sintered and heat-treated compact made from 4%Ni-1.5%Cu-0.5%Mo composite-type alloy steel powder. White areas correspond to Ni-rich retained austenite. Such retained austenite may affect fatigue properties, although it should be noted that a quantitative evaluation has not yet been obtained. It is considered that the amount of retained austenite can be reduced by choosing appropriate operational conditions for the process of composite-type alloying or heat treatment of the sintered compacts.

Table 4 summarizes the dependence of sintered and heat-treated properties on alloy compositions of composite-type powders with compositions 2 to 4%Ni, 1.5 to 3%Cu and 0.3 to 0.6%Mo. The desired composition corresponding to required properties for each application can be selected on the basis of these empirical relationships.

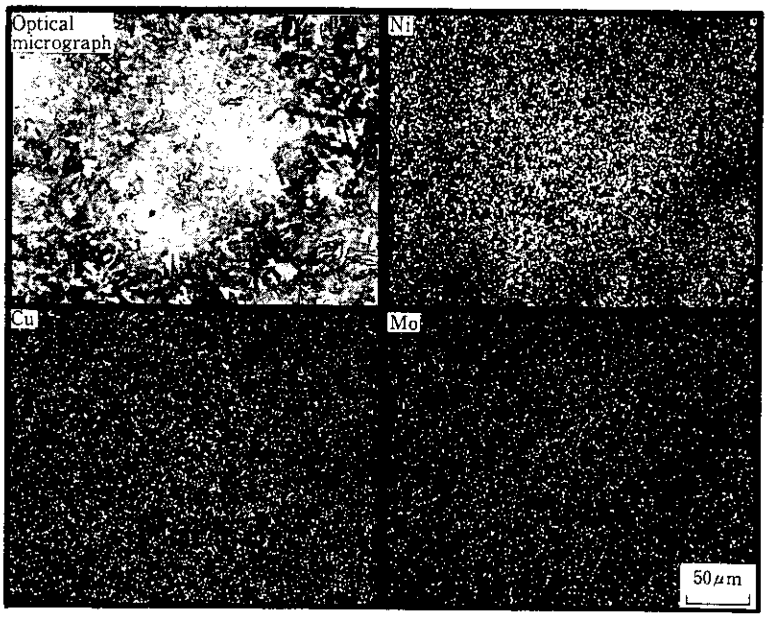


Photo 5 Microstructure and distribution of alloying elements in sintered and heat-treated compacts made from 4%Ni-1.5%Cu-0.5%Mo composite-type alloy steel powders

Table 4 Coefficients of regression equations*1 for the effect of alloying elements on sintered and heat-treated properties of Ni-Cu-Mo composite-type alloy steel powders

Property	α_{Ni}	α_{Cu}	α_{Mo}	Const.	Correlation coefficient
Dimensional chage*2 (%)	−0.0479	0.298	0.0961	−0.805	0.989
Radial crushing strength (MPa)	134	76.1	303	1110	0.814
Charpy impact value (J)	2.31	0.686	9.66	3.79	0.913

*1 Property$=\alpha_{(Ni)}(\%Ni)+\alpha_{(Cu)}(\%Cu)+\alpha_{(Mo)}(\%Mo)+$Const.

*2 Dimensional change from green to heat-treated compacts

5 Conclusions

Characteristics of composite-type alloy steel powders KIP SIGMALOY developed by Kawasaki Steel were investigated in comparison with those of conventional prealloyed and mixed powders. The effects of alloying methods and alloying elements were evaluated. The results are summarized as follows:

(1) KIP SIGMALOY powders satisfy both the conditions essential for application to heavy duty mechanical parts: high compressibility of powders and uniformity of alloying elements in sintered compacts. Compressibility is comparable with that of pure iron powders.

(2) SIGMALOY Cu containing 2%Cu increases the strength of sintered compacts by 10% and dimensional accuracy by 30% when compared with those of powder mix compacts.

(3) SIGMALOY 415 containing 4%Ni, 1.5%Cu and 0.5%Mo improves the strength and toughness of sintered and heat-treated compacts by more than 10% when compared with those of powder mix compacts.

The applications of powder metallurgy are certain to be enhanced by the use of these new powders in combination with improved compaction and sintering technologies.

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