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Characteristics and Strengthening Mechanism of Alloyed Steel Powder "KIP SIGMALOY 2010" for Ultra High Strength Sintered Materials*


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The chemical composition of Ni- and Mo-containing composite-type alloyed steel powder was optimized for the production of high strength sintered components via compaction, sintering and heat treatment. A powder with 2%Ni and 1%Mo, KIP SIGMALOY 2010, attained a high tensile strength of 1 920 MPa, a high unnotched Charpy absorbed energy of 53 J, a high endurance limit of rotating bending fatigue strength of 390 MPa and a high endurance limit of contact fatigue strength of 2 710 MPa, when double-pressed, double-sintered, bright-quenched and tempered. These values were much higher than those of a conventional composite-type alloyed steel powder. The stgrengthening was attributed to the increase of sintered density and the strain-induced martensitic transformation of the austenite phase. The sintered compacts made from the new powder remained soft before heat treatment and showed a ten-times longer tool life in the case of low cutting speed, compared with that of a conventional composite-type alloyed steel powder.

1 Introduction

With higher automotive engine outputs and increasingly stringent fuel efficiency requirements in response to environmental problems, the trend in mechanical parts for automobiles is toward higher performance and reduced size. The development of sintered materials with outstanding strength, toughness, and fatigue properties has been pursued against this background.[1] However, the current maximum value for tensile strength is on the order of 1 300 MPa. Effective means of achieving further increases in strength include (1) optimizing the method of alloying and composition in consideration of compressibility, (2) increasing the density of the sintered compact by repressing,[2] (3) applying heat treatment[3,4] and (4) reducing the content of impurity elements. When repressing and heat treatment are applied, it is necessary to select a composition which provides low hardness in the as-sintered state and high strength when heat treated.

A composite alloyed steel powder with a composition of 4%Ni-1.5%Cu-0.5%Mo, which is produced by blending pure iron powder and metal or metal oxide powders and applying diffusion annealing, has been developed as a high-compressibility alloyed steel powder,[5] but application in the as-sintered state is a precondition with this composition. On the other hand, no research has been done on compositions suitable for repressing and heat treatment. However, the existence of a densified layer of Ni and Mo in composite alloyed steel powders makes it necessary to give attention to the austenite formation behavior of sintered and heat treated compacts.

Although a considerable amount of research has been done on the effect of impurities such as P and S on the strength and toughness of iron and steel materials, the effect of the content of impurity elements remains unclear where it concerns sintered materials, which are porous, and in particular, heat-treated sintered materials.

This paper first discusses (1) the effect of Ni and Mo content on repressing characteristics, (2) the effect of P and S content on the strength and toughness of sintered and heat-treated compacts, and (3) the strengthening mechanism of sintered and heat-treated compacts from the viewpoint of austenite strain stability. The paper also describes the characteristics of sintered and

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heat-treated compacts of a composite alloyed steel powder (KIP SIGMALOY 2010) with a new 2%Ni-1%Mo composition, which was developed on the basis of these results.

2 Fundamental Experiments

2.1 Experimental Method

2.1.1 Effect of Ni and Mo content on compressibility, repressibility, and hardness of sintered compacts of composite alloyed steel powders

As sample steel powders, composite alloyed steel powders A through G were used, as shown in **Table 1**. The Ni content was between 1.5% and 4%, and the Mo content was between 0.5% and 1.5%. Zinc stearate (1%) was added as a lubricant, together with graphite (0.6%). Test pieces were fabricated by compacting at 690 MPa, presintering for 30 min at 1 123 K in a 75%H_2-25%N_2 atmosphere, repressing at 690 MPa, and resintering for 60 min at 1 523 K in a 75%H_2-25%N_2 atmosphere (double-pressing, double-sintering; 2P2S). The change in density in each processing step was measured by the Archimedes method, and the Rockwell hardness of the final sintered compacts were examined.

Table 1 Chemical compositions of composite-type alloyed steel powder studied (wt %)

Powder	Ni	Mo	P	S
A	1.56	0.99	0.006	0.003
B	2.09	1.02	0.007	0.003
C	2.04	0.52	0.007	0.003
D	2.10	1.48	0.006	0.002
E	2.50	0.99	0.007	0.003
F	2.99	0.99	0.006	0.002
G	3.92	1.02	0.005	0.003
H	2.00	0.98	0.013	0.012
I	2.10	0.98	0.011	0.012
J	1.92	0.93	0.001	0.002

2.1.2 Effect of P and S content on strength and toughness of sintered and heat-treated compacts

Using powder B and powders H-J with modified contents of P and S (Table 1), an examination was made of the effect of the P and S content on the strength of sintered and heat-treated compacts produced from 2%Ni-1%Mo steel powder. Double pressing and double sintering were carried out under the conditions described in Sec. 2.1.1. The specimens were heat treated for 60 min at 1 143 K in Ar gas, bright quenched, and tempered for 60 min at 453 K in oil (2P2S-BQT).

The strength of the sintered and heat-treated compacts was evaluated in terms of tensile strength using small round-bar specimens 5 mm in diameter and 15 mm long in the parallel section. Tensile fracture surfaces were observed with a scanning electron microscope at an acceleration voltage of 15 kV and inclusions were identified by energy dispersive X-ray analysis.

2.1.3 Effect of austenite content on strength of sintered and heat-treated compacts

Sintered and heat-treated compacts of 2%Ni-1%Mo steel powder and 4%Ni-1.5%Cu-0.5%Mo steel powder of conventional composition were produced under the same conditions as those described in Sec. 2.1.2. Compressive deformation was used to apply a strain of 0-15% in the height direction. The austenite content was measured from the integrated intensity of the austenite in the (200) and (220) planes using the MoKα characteristic X-ray method. Strain was applied to investigate strain-induced behavior in the transformation of austenite to martensite (strain-induced martensitic transformation of austenite).[6] In addition, the relationship between tensile strength and austenite content was examined using steel powders with differing Ni and Mo diffusion characteristics. Tensile strength was evaluated using small round-bar specimens 5 mm in diameter and 15 mm long in the parallel section.

2.2 Experimental Results and Discussion

2.2.1 Effect of Ni and Mo on density of green and sintered compacts and on strength of sintered compacts

The respective effects of Ni and Mo content on the density of green, presintered, and repressed compacts are shown in **Figs. 1** and **2**. The density of the green and presintered compacts was unchanged in the range of Ni and Mo contents studied in this work. However, when the Ni content exceeded 2% and the Mo content exceeded 1%, the effectiveness of repressing is increasing the density of the compact decreased. Adding the maximum amount of alloy possible without negatively

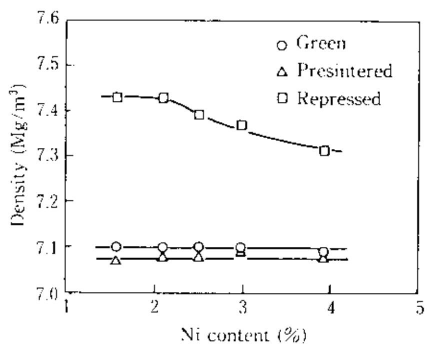

Fig. 1 Relationships between Ni content and green, presintered and repressed density for 1% Mo containing composite-type alloyed steel powders with 0.6% graphite

affecting density enhancement was effective in improving strength. The drop in density which occurred during presintering was attributed to volatilization and loss of the lubricant.

The respective effects of Ni and Mo content on the hardness of compacts sintered at 1 523 K are shown in **Figs. 3** and 4. The hardness of the sintered compacts rose to over HRB 90 when Ni exceeded 2% and when Mo exceeded 1%.

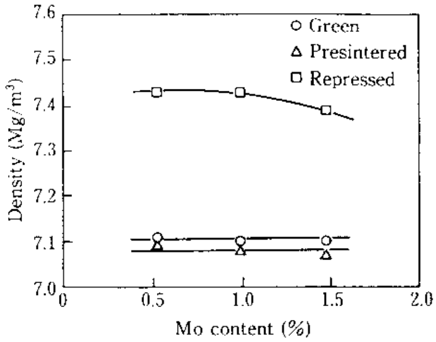

Fig. 2 Relationships between Mo content and green, presintered and repressed density for 2% Ni containing composite-type alloyed steel powders with 0.6% graphite

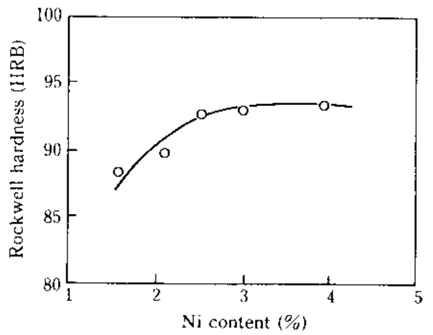

Fig. 3 Relationship between Ni content and Rockwell hardness for sintered compacts of 1%Mo containing composite-type alloyed steel powders with 0.6% graphite

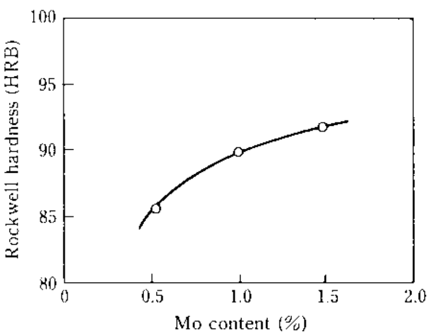

Fig. 4 Relationship between Mo content and Rockwell hardness for sintered compacts of 2%Ni containing composite-type alloyed steel powders with 0.6% graphite

From these results, it was concluded that a composite alloyed steel powder with a 2%Ni-1%Mo composition would be an appropriate material for heat-treated high-strength sintered parts, in consideration of the increase in mechanical workability attributable to the reduced hardness of the sintered compact and the higher density obtained by repressing.

2.2.2 Strength of double-pressed, double-sintered, bright-quenched compacts of 2%Ni-1%Mo material

Figure 5 shows the changes in austenite content when strain was applied to double-pressed, double-sintered, bright-quenched and tempered compacts produced from 2%Ni-1%Mo and 4%Ni-1.5%Cu-0.5%Mo steel powders. Such compacts differ in tensile strength. In addition, specimens with differing tensile strengths were obtained by changing the condition of Ni and Mo diffusion. The tensile strength was proportionate to the austenite content prior to the application of strain in the compacts of both materials. Although the austenite content decreased as the amount of strain increased, the austenite content showed a fixed value in the strain region of 10% and over. Moreover, austenite became stable to the strain at a higher content with the 4%Ni-1.5%Cu-0.5%Mo material than with the 2%Ni-1%Mo

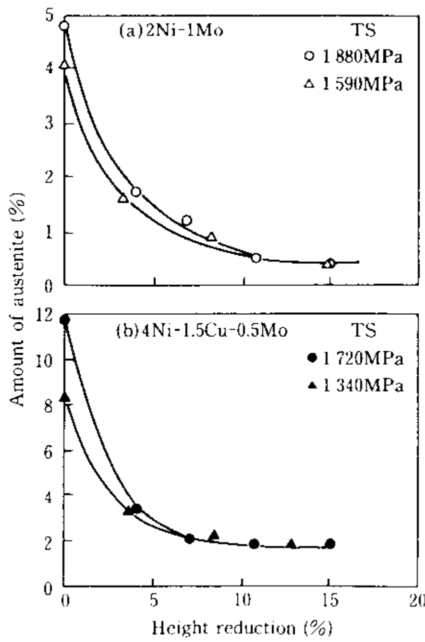

Fig. 5 Change in austenite amount of double-pressed, double-sintered and heat-treated compacts with 0.6% graphite

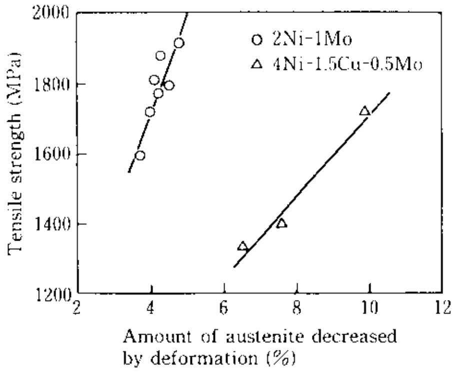

Fig. 6 Effect of amount of austenite decreased by deformation on tentile strength of double-pressed, double-sintered and heat-treated compacts with 0.6% graphite

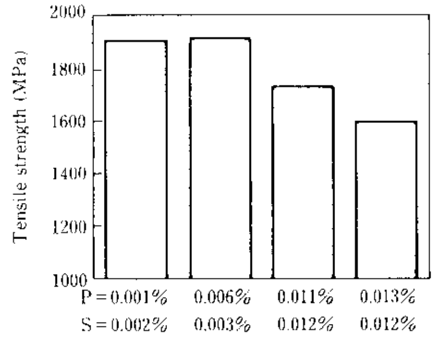

Fig. 7 Effects of P and S contents on tensile strength of double-pressed, double-sinterd and heat-treated 2%Ni-1%Mo compacts with 0.6% graphite

material.

Figure 6 shows the relationship between changes in austenite content and tensile strength in double-pressed, double-sintered, bright-quenched and tempered compacts of 2%Ni-1%Mo and 4%Ni-1.5%Cu-0.5%Mo steel powders. With the same steel powder, tensile strength increases as the change in austenite content becomes greater. Under these experimental conditions, a tensile strength of 1 960 MPa was achieved with the 2%Ni-1%Mo material by controlling the austenite content. On the other hand, the highest tensile strength obtained with the 4%Ni-1.5%Cu-0.5%Mo powder was low at 1 720 MPa. The high tensile strength obtained with the 2%Ni-1%Mo material was attributed to the facts that retained austenite stabilized to strain at a low content and the density of the sintered compact was high. The density of the sintered compact of 2%Ni-1%Mo steel powder was 7.43 Mg/m^3, while that of the 4%Ni-1.5%Cu-0.5%Mo powder was 7.34 Mg/m^3. However, an estimate based on the results presented by Ohara et al.[7] suggested that the difference in tensile strength which can be explained in terms of sintered density is no more than 90 MPa.

Figure 7 shows the effects of P and S contents on the tensile strength of double-pressed, double-sintered, bright-quenched compacts of 2%Ni-1%Mo steel powder. When P is reduced to 0.006% and S to 0.003%, the tensile strength will be greater than 1 900 MPa. **Photo 1**, which is the result of an examination of the fracture surface of a high-P, high-S specimen with respective P and S contents of 0.013% and 0.012%, shows a micrograph obtained by SEM observation and the content of the inclusions as determined by energy dispersive X-ray analysis. MnS and CaS were found at the fracture surface. In contrast, grain boundary fracture surfaces were not observed even at high P levels. The presence of P showed no relation to ductile fracture and reduced the deformation stress by grain boundary fracture.[8,9] These results suggest that the main reason for the increase in tensile strength as the P and S contents decrease is a reduction in MnS and CaS inclusions.

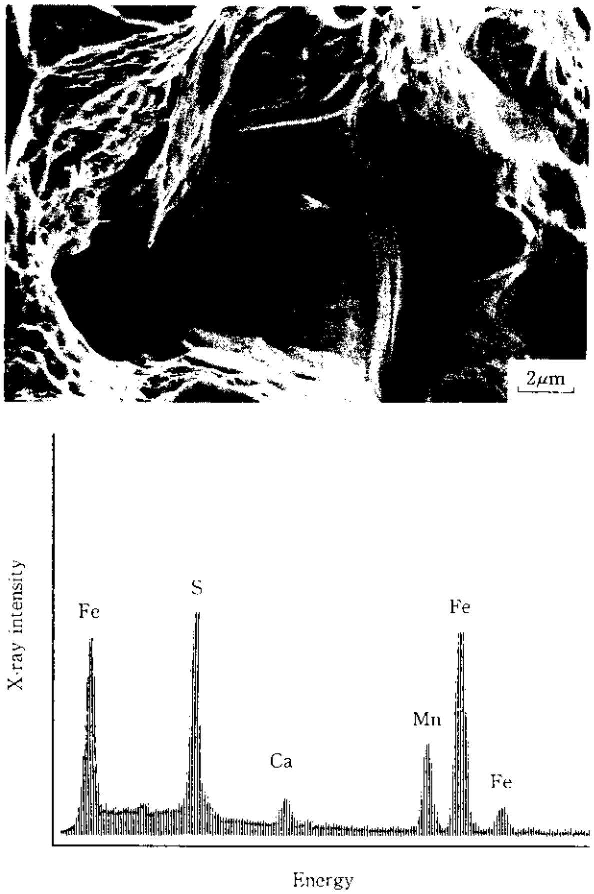

Photo 1 Scanning electron micrograph of fractured specimen in double-pressed, double-sintered and heat-treated 2%Ni-1%Mo compact, and impurity contents in inclusion revealed by dispersive energy analysis

The results described above make it clear that sintered materials with ultra high strength of 1 920 MPa can be obtained with 2%Ni-1%Mo steel powder by reducing impurity elements and controlling the austenite content of the sintered and heat-treated compact.

3 Factory Trial-Production Experiments

3.1 Experimental Method

3.1.1 Specimen materials

As made clear in the previous chapter, high purity (0.005%P, 0.003%S) 2%Ni-1%Mo steel powder KIP SIGMALOY 2010 is a composite alloy steel powder with an appropriate composition for use in heat-treated high-strength sintered parts. Characteristics of sintered and heat-treated compacts of this powder and 4%Ni-1.5%Cu-0.5%Mo steel powder (KIP SIGMALOY 415S) of comparative material produced in factory was investigated. The results are shown in **Table 2**. Sintered compacts were produced from these powders under the following sets of conditions.

(1) 1P1S-CQT Material (Single-pressed, Single-sintered, Carburized Compacts): Specimens were prepared by adding 1% zinc stearate as a lubricant, compacting at 690 MPa, and sintering for 60 min at 1 523 K in a 75%H_2-25%N_2 atmosphere. Carburizing was performed for 150 min at 1 193 K at a 0.9% carbon potential, after which the specimens were quenched in oil and tempered for 60 min at 453 K.

(2) 2P2S-BQT Material (Double-pressed, Double-sintered, Bright-treated Compacts): Specimens were prepared by adding 1% zinc stearate as a lubricant and 0.6% graphite precompacting at 690 MPa, and presintering for 30 min at 1 123 K in a 75%H_2-25%N_2 atmosphere, after which they were repressed at 690 MPa and resintered in a 75%H_2-25%N_2 atmosphere for 60 min at temperatures of 1 423, 1 523, and 1 573 K. After heating the specimens for 60 min at 1 143 K, bright quenching and tempering were performed. Finally, the specimens were tempered for 60 min at 453 K.

3.1.2 Machinability of sintered compacts, mechanical properties of sintered and heat-treated compacts

Lathe tests of the sintered compacts were conducted using carbide (K 10, P 20) and cermet tools. As cutting conditions, the depth of cut was 1 mm, the feed per revolution was 0.05 mm/rev, and the cutting speed was 50-200 m/min. The strength of the sintered and heat-treated compacts was measured in terms of tensile strength using small round-bar test specimens 5 mm in diameter and 15 mm long in the parallel section; toughness was evaluated from absorbed energy using unnotched Charpy impact test pieces 10 mm high, 10 mm wide, and 55 mm long. Rockwell hardness values were also measured. Next, endurance limits of fatigue strength were examined in the Ono-type rotating bending fatigue test using small round-bar specimens 8 mm in diameter and 15.4 mm long in the parallel section, and in the 6-ball Mori-type contact fatigue test with pieces 60 mm in outer diameter, 20 mm in inner diameter, and 5 mm high. The Ohgoshi-type wear test was used to investigate the wear resistance of the sintered and heat-treated compacts. As the wear environment, an SUJ-2 wear plate was used at a wear speed of 4.21 m/s and load of 12.6 kg in atmospheric air, with one drop of oil applied per second.

3.1.3 Fracture surface analysis

Changes in the austenite content in the vicinity of the contact fracture surface were investigated using a microbeam X-ray with an acceleration voltage of 45 kV and a collimator diameter of 50 μm. A relative evaluation of changes in the austenite content was made in terms of the integrated intensity ratio of the austenite (200) plane to the ferrite (200) and (110) planes.

3.2 Experimental Results and Discussion

3.2.1 Machinability of sintered compacts

Figure 8 shows the machinability test results for sintered compacts produced from 2%Ni-1%Mo steel powder and 4%Ni-1.5%Cu-0.5%Mo steel powder which was used for comparison purposes. Both materials contained 0.6% graphite, and were processed by pressing at 690 MPa, presintering at 1 123 K, repressing at 690 MPa, and resintering at 1 523 K. Tool life was defined as the time until the flank wear reached a width of 0.2 mm. Sintered compacts of the 2%Ni-1%Mo steel powder showed outstanding machinability with all tools in the low-speed cutting range. This phenomenon was attributed to the fact that the hardness of the sintered compacts of 2%Ni-1%Mo steel powder was low (HRB 89) when compared with that of the 4%Ni-1.5%Cu-0.5%Mo steel

Table 2 Characteristics of powders studied

Powder	Particle size distribution (%)						Apparent density (Mg/m³)	Flow rate (s/50 g)
	≧150 μm	106~150 μm	75~106 μm	63~75 μm	45~63 μm	≦45 μm		
KIP SIGMALOY 2010 (2Ni-1Mo)	2.5	27.7	25.2	14.9	14.9	14.8	2.90	18.5
KIP SIGMALOY 415S (4Ni-1.5Cu-0.5Mo)	3.1	21.9	25.4	9.0	16.1	24.5	2.90	25.6

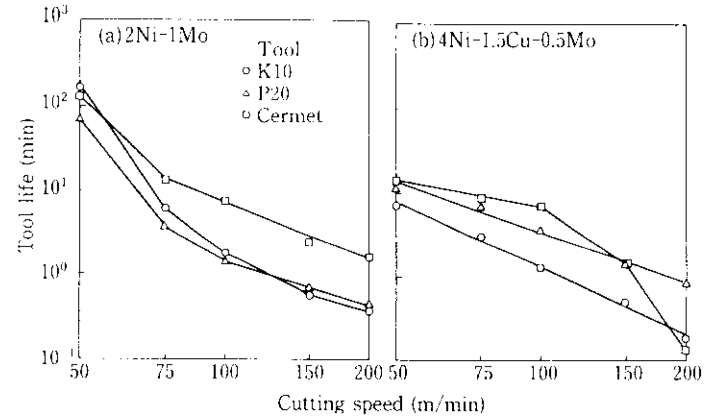

Fig. 8 Machinability test results of double-pressed, double-sintered compacts made from 2%Ni-1%Mo(a) and 4%Ni-1.5%Cu-0.5%Mo(b) composite-type alloyed steel powders with 0.6% graphite

powder compacts (HRB 102).

3.2.2 Mechanical properties of sintered and heat-treated compacts

Table 3 and **4** show the tensile strength, Charpy absorbed energy, and endurance limits of rotating bending fatigue strength and contact fatigue strength for 1P1S-CQT and 2P2S-BQT materials. In both the 1P1S-CQT and 2P2S-BQT materials the 2%Ni-1%Mo material was superior to the conventional material in all four strength parameters.

Table 3 Mechanical properties of sintered and heat-treated compacts made from a single-pressing, single-sintering, carburizing and tempering process (0%Gr., sintered at 1 523 K)

Power	Tensile strength (MPa)	Absorbed energy (J)	Endurance limit (MPa)	
			Rotating bending	Contact
KIP SIGMALOY 2010 (2Ni-1Mo)	1 500	21	460	2 560
KIP SIGMALOY 415S (4Ni-1.5Cu-0.5Mo)	1 380	20	410	2 430

Table 4 Mechanical properties of sintered and heat-treated compacts made from a double-pressing, double-sintering, bright-quenching and tempering process (0.6%Gr., sintered at 1 523 K)

Power	Tensile strength (MPa)	Absorbed energy (J)	Endurance limit (MPa)	
			Rotating bending	Contact
KIP SIGMALOY 2010 (2Ni-1Mo)	1 920	53	390	2 710
KIP SIGMALOY 415S (4Ni-1.5Cu-0.5Mo)	1 720	39	350	2 330

The effect of the sintering temperature on the tensile strength and Charpy absorbed energy of 2P2S-BQT materials is shown in **Figs. 9** and **10** respectively. At a

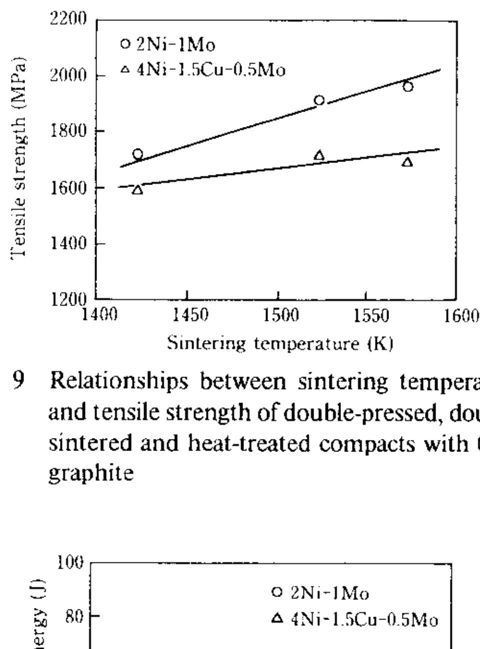

Fig. 9 Relationships between sintering temperature and tensile strength of double-pressed, double-sintered and heat-treated compacts with 0.6% graphite

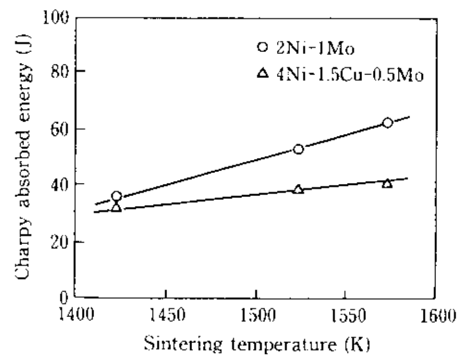

Fig. 10 Relationships between sintering temperature and Charpy absorbed energy of double-pressed, double-sintered and heat-treated compacts with 0.6% graphite

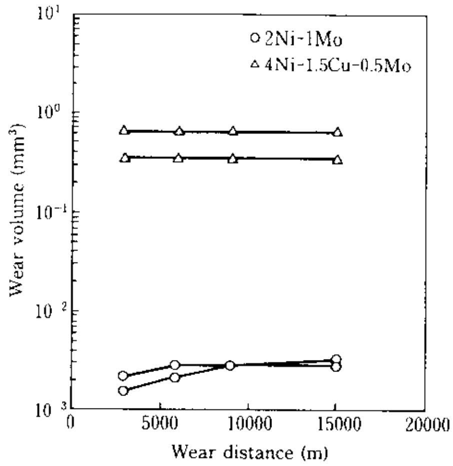

Fig. 11 Wear resistance test results of single-pressed, single-sintered and heat-treated compacts made from 2%Ni-1%Mo and 4%Ni-1.5%Cu-0.5%Mo composite-type alloyed steel powders

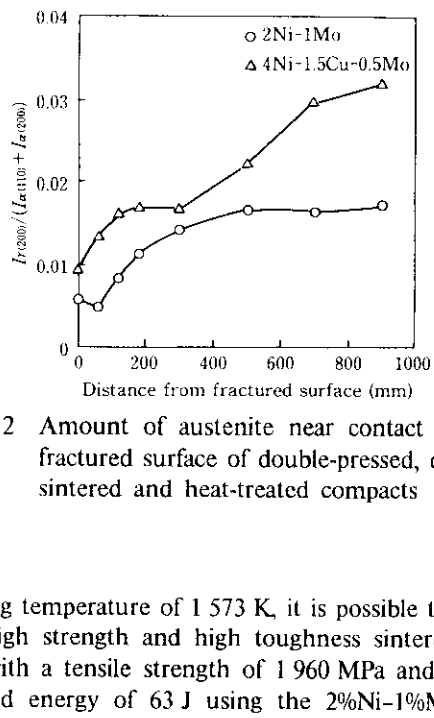

Fig. 12 Amount of austenite near contact fatigue fractured surface of double-pressed, double-sintered and heat-treated compacts

sintering temperature of 1 573 K, it is possible to obtain ultra high strength and high toughness sintered compacts with a tensile strength of 1 960 MPa and Charpy absorbed energy of 63 J using the 2%Ni-1%Mo steel powder.

The results of a wear resistance test of 1P1S-CQT materials are shown in **Fig. 11**. The wear resistance of the 2%Ni-1%Mo compacts was superior to that of the 4%Ni-1.5%Cu-0.5%Mo compacts. The surface hardnesses of the two materials were HRC 39 and HRC 34 respectively, but it is not possible to explain the excellent wear resistance of the 2%Ni-1%Mo compacts only on the basis of hardness.

The major reason for the superiority of 2%Ni-1%Mo compacts to 4%Ni-1.5%Cu-0.5%Mo compacts in the full range of characteristics including tensile strength, fatigue strength, and wear resistance is considered to be the strain-induced martensitic transformation of the austenite phase. **Figure 12** shows changes in the austenite content near the contact fracture surface of 2P2S-BQT materials, as obtained by microbeam X-ray measurement. In sintered and heat-treated compacts of both materials, martensitic transformation occurred immediately below the fracture surface, but a smaller amount of untransformed austenite remained in the 2%Ni-1%Mo compact. This fact is in agreement with the results shown in Fig. 5. Accordingly, in phenomena involving plastic deformation, such as tensile deformation, fatigue deformation, and wear, the superior features of sintered and heat-treated compacts of 2%Ni-1%Mo steel powder can be explained by the strain-induced matensitic transformation of the austenite phase.

4 Conclusions

KIP SIGMALOY 2010 with a new 2%Ni-1%Mo composition was developed as an alloyed steel powder for use in sintered parts which must be low in hardness prior to heat treatment, and thus offer good machinability and sizing characteristics, but must also provide high strength and high toughness after heat treatment. The results obtained in the development work are summarized below.

(1) The content of the alloying elements Ni and Mo can be increased to 2% and 1% respectively with no negative effect on the increased density secured by repressing. Accordingly, it is possible to obtain high-density sintered compacts by repressing, using a 2%Ni-1%Mo composite-type alloyed steel powder.
(2) Sintered compacts of 2%Ni-1%Mo steel powder are low in hardness and therefore offer better machinability in the low range of cutting speeds than sintered compacts produced from the conventional 4%Ni-1.5%Cu-0.5%Mo material.
(3) Higher tensile strength is obtained when the austenite content of the sintered and heat-treated compact is great and the amount of the strain-induced martensitic transformation is large. However, tensile strength decreases when the amount of stable austenite to strain increases.
(4) With 2%Ni-1%Mo steel powder, it is possible to obtain sintered compacts with a tensile strength of 1 500 MPa and an unnotched Charpy absorbed impact energy value of 21 J by single-pressing, single-sintering, carburizing and tempering. Extremely high strength (tensile strength, 1 920 MPa) and high toughness (unnotched Charpy absorbed impact energy, 53 J) can be obtained by double-pressing, double-sintering, bright-quenching and tempering (sintering temperature, 1 523 K). These features are superior to those of compacts produced from the conventional 4%Ni-1.5%Cu-0.5%Mo steel powder.

(5) Using 2%Ni-1%Mo steel powder, it is possible to obtain an endurance limit of rotating bending fatigue strength of 460 MPa and an endurance limit of contact fatigue strength of 2 560 MPa by single-pressing, single-sintering, carburizing and tempering. The high strength features obtained by applying double-pressing, double-sintering, bright-quenching and tempering include an endurance limit of rotating bending fatigue strength of 390 MPa and contact fatigue strength of 2 710 MPa. These values are higher than those with the conventional 4%Ni-1.5%Cu-0.5%Mo material. In addition, the wear resistance of sintered and heat-treated compacts of 2%Ni-1%Mo steel powder is superior to that of sintered and heat-treated compacts of the conventional steel powder.

(6) The reasons for the superior tensile strength, fatigue strength, and wear resistance of 2%Ni-1%Mo compacts is that optimization of the composition made it possible to increase the density of the sintered compact and reduce the amount of austenite which is stable to strain.

Based on the experimental results described above, KIP SIGMALOY 2010 was developed as a composite alloyed steel powder with a new composition, for use in heat-treated, high-strength sintered products. In line with the trend toward higher performance and reduced size in automotive engines and drive trains, this newly developed material is expected to find wide application in synchro hubs and other transmission parts. KIP SIGMALOY 2010 will also make it possible to produce mechanical parts which require higher levels of strength and toughness than those achieved with conventional powder metallurgy.

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