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Steel Powder and Die-lubricated Warm Compaction for Automotive Sintered Parts with High Density and Excellent Fatigue Property†

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Abstract:

JFE Steel has developed a new powder metallurgical process named die-lubricated warm compaction method for producing high-density sintered parts using premixed steel powder containing only small amount of internal lubricant. In this method, a newly developed spraying equipment precisely controls the amount of lubricant applied over the die wall. The sintered and bright-quenched compacts were prepared by this method using partially alloyed steel powder, KIP SIGMALOY 415S, containing 4 mass% Ni, 1.5 mass% Cu, and 0.5 mass% Mo. They gave a density as high as 7.5 Mg/m^3 and rotating bending fatigue strengths of 450 MPa and 470 MPa with carbon contents of 0.52 mass% and 0.92 mass% respectively. The density and fatigue strength were superior to those obtained by the conventional double-pressing and double-sintering method. Presumably, the fatigue strength was enhanced by increasing the carbon content because the amount of retained austenite was increased.

1. Introduction

Higher strength and better fatigue property are increasingly required to automotive parts in line with the necessity for making automobiles lighter in weight as well as for dealing with higher-performance engines.

In powder metallurgy, increasing sintered density is the most effective way for improving mechanical properties such as strength and fatigue property. Various approaches have been proposed to increase density. In the warm compaction method (W/C method), material powder is preheated and compacted in a die heated to about 130°C and then sintered.[1–3] In the double-pressing and double-sintering method, the process of cold compaction and sintering is repeated twice. Another approach is powder forging where a sintered compact prepared by the single-pressing and single-sintering method is subjected to hot forging. In these methods, however, production cost increases with attainable density. Hence, a new economic method for realizing high density is required.

JFE Steel has developed a new high-density compaction method named die-lubricated warm compaction method (W/D method)[4,5]. In this method, the premixed steel powder that allows high-density compaction while containing only a small amount of internal lubricant is used in the W/C method employing a die to which lubricant is applied by electrostatic spraying[6,7]. This method can realize a higher density at a cost nearly equivalent to that of the W/C method. Its application to the mass production of high-density automotive sintered parts is now being studied.

This paper first describes the features of the W/D method. Next, it presents the mechanical properties of the sintered compacts (density: 7.5 Mg/m^3) prepared by the W/D method using partially alloyed steel powder suitable for high-density compaction. Lastly, it describes the rotating bending fatigue property after bright quenching of the sintered compacts.

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2. Features of Die-Lubricated Warm Compaction (W/D) Method

As described above, the W/D method can achieve high-density compaction using premixed steel powder that contains only a small amount of internal lubricant.

With regard to the premixed steel powder that is composed of steel powder and lubricant, the relation between internal lubricant content and theoretically attainable green density is shown in **Fig. 1**. The theoretically attainable green density was calculated as the density of a green compact that was fully compacted to a state where void was completely eliminated using steel powder and lubricant having the real specific gravity of 7.87 and 1.00 respectively. The theoretical green density increases as the internal lubricant content decreases.

In the W/D method, the friction between the green compact and the die is alleviated by the powder lubricant applied on the die. In this way, it was made possible to compact the premixed steel powder that contains only a small amount of internal lubricant without causing problems such as cracking and chipping. In the conventional method, an internal lubricant content of about 0.8 mass% is required. In contrast, an internal lubricant content of less than 0.2 mass% is sufficient for performing the W/D method.

In this method, the premixed steel powder containing approximately 0.1 to 0.2 mass% of KW-wax[3)], a lubricant developed for use in the W/C method, is used. Theoretically, the decrease in internal lubricant content has the effect of increasing the green density approximately by 0.3 Mg/m^3.

The schematic diagram of the W/D method is shown in **Fig. 2**. It is composed of two processes. The first is a process of coating the die with powder external lubricant. The second is a process of heating the premixed steel powder that contains the required minimum amount of internal lubricant, filling the preheated die with it, and then compacting it. The external lubricant for coating the die needs to be excellent in electrostatic property in the charge gun, adhesive property on the heated die surface, and lubricating property in compacting the steel powder. Therefore, a new external lubricant was specifically developed in-house for use in this compaction method. Electrostatic charging could be performed by the corona charging method, which however tends to cause powder to excessively adhere to the convex areas when a die with a prominent convex-concave shape is used. Hence, the contact electrostatic charging method[8)] was adopted for giving a smaller amount of electric charge.

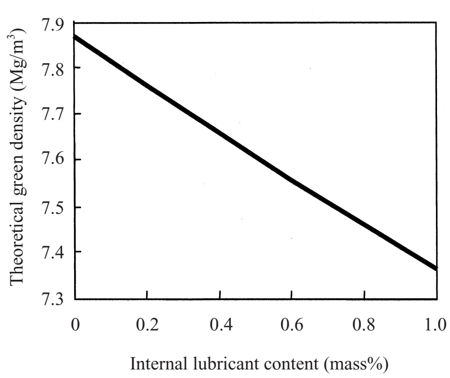

Fig. 1 Theoretical green density calculated using the mixing ratio of internal lubricant

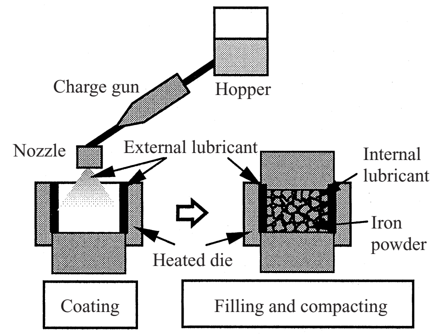

Fig. 2 Schematic diagram of die-lubricated warm compaction method

When the amount of sprayed lubricant is insufficient, excessive friction is caused on the contact surface between the compact and the die wall to generate wear scars on the sliding surface when the compact is ejected out of the die. The excessive friction not only damages the external appearance of the compact, but also increases the loads on the die and deteriorates it. Conversely, the areas where excessive amounts are sprayed tend to become the starting points of cracking and chipping, causing shape defects. These problems hitherto limited the productivity in the continuous compaction process using this method and hindered it from being applied in mass production.

Therefore, a new lubricant applicator that can suppress the variation in the amount of sprayed lubricant was developed in cooperation with Nordson KK.[9)] The schematic diagram is shown in **Fig. 3**. The equipment is composed of (1) a lubricant hopper equipped with a table feeder at the exit port that can discharge a constant amount highly accurately, (2) a charge gun, (3) a nozzle, (4) a bypass block, and (5) a dust collector. In this equipment, lubricant powder is continuously discharged from the table feeder and circulated in the equipment by dry air even when the lubricant is not being sprayed over the die. The lubricant is discharged from the nozzle and sprayed over the die when the air supply to the bypass block is stopped. The amount of lubricant that remains in the equipment is decreased by continuously circulating the lubricant. The unused lubricant collected in the dust collector is reused.

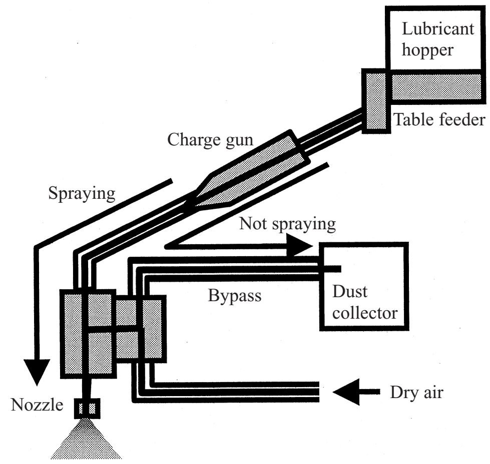

Fig. 3 Schematic diagram of the developed lubricant applicator

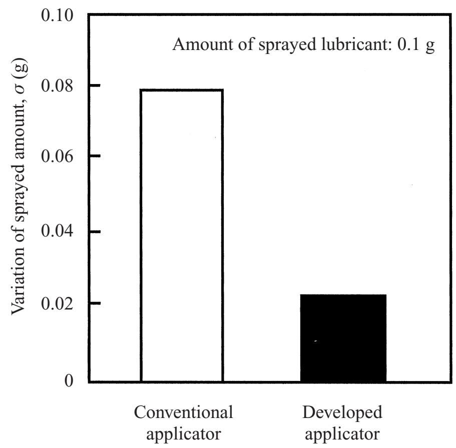

Fig. 4 Variation of amount of sprayed lubricant depending on ability of applicator

3. Accuracy in Sprayed Amount by Lubricant Applicator

3.1 Experimental Method

The variation in the amount of lubricant sprayed by the conventional and developed applicators was evaluated using a new external lubricant (WD2) specifically developed in-house for use in the W/D method. The sprayed amount was measured under the condition of spraying 0.1 g of lubricant at a time. Spraying ten times using each applicator, the standard deviation of the sprayed amount was calculated as the index of the variation.

3.2 Experimental Result

In **Fig. 4**, the standard deviation in the amount of lubricant sprayed by the developed applicator is compared with that of the conventional applicator. The width of the variation in the amount of sprayed lubricant was reduced to 1/4 using the developed applicator. This is due to the fact that the table feeder can discharge a constant amount of lubricant highly accurately, and the amount of lubricant that adheres in the equipment is decreased by continuous circulation.

4. Features of High-Density Sintered Compacts Prepared by the W/D Method

4.1 Experimental Method

4.1.1 Material

The experiment was conducted using the premixed steel powder composed of KIP SIGMALOY 415S (partially alloyed steel powder containing 4 mass% Ni, 1.5 mass% Cu, and 0.5 mass% Mo), graphite powder, and lubricant. The chemical compositions of the powders are shown in **Table 1**. The graphite powder was J-CPB made by Nippon Graphite Industries, Co., Ltd. Two types of internal lubricant were used: ZNS-730, zinc stearate produced by Asahi Denka, Co., Ltd., and KW-wax, a lubricant developed in-house for use in the W/C method. The external lubricant used was WD2, a powder lubricant specifically developed in-house for use in the W/D method.

4.1.2 Conditions for preparing test pieces

Using the equipment shown in Fig. 3, the lubricant WD2 was applied over the surface of the die heated to 130°C. Next, the die was filled with the aforemen-

Table 1 Chemical compositions and compacting conditions of the studied powders for investigating the effects of compaction methods

	Graphite	Internal lubricant	Compacting temperature	Compacting pressure	External lubricant
C/C method	0.6 mass%	Zinc stearate 0.75 mass%	Room temperature	686 MPa	–
W/C method		KW-wax* 0.6 mass%	130°C		
W/D method		KW-wax* 0.2 mass%	130°C		WD2**

* Lubricant for warm compaction, ** Lubricant for heated die

tioned premixed powder preheated to 130°C and compaction was performed. The compacting pressure was 490 to 686 MPa. The size of the compact was 55 mm long, 10 mm wide, and 10 mm high. For comparison purposes, compacts were also prepared by the cold compaction method (C/C method), which compacts room temperature powder using an room temperature die, as well as by the W/C method. These compacts were sintered in the N_2-10vol%H_2 atmosphere at 1 250°C for 60 min. Some of the sintered compacts were machined into round-bar tensile test pieces, each having a 5 mmϕ and 15 mm long parallel portion.

4.1.3 Evaluating method

The force required for ejecting a 55 mm long, 10 mm wide, and 10 mm high compact test piece was measured. The green density was also measured using the same test piece. The tensile strength was measured using the aforementioned round-bar tensile test piece at a speed of 5 mm/min. The impact value was measured using a 10 × 10 × 55 mm unnotched Charpy impact test piece.

4.2 Experimental Result

4.2.1 Ejection forces and green densities

The ejection forces and green densities of the compacts made by various compaction methods are shown in **Fig. 5**. The ejection force in the W/D method was lower than that in the W/C method and nearly equivalent to that in the C/C method. The green density obtained by the W/D method is as high as 7.4 Mg/m^3, which is an improvement of about 0.2 Mg/m^3 over the C/C method and of about 0.1 Mg/m^3 over the W/C method. This improvement in the density is due to the reduced amount of the internal lubricant. Generally, the friction force between die and compact increases with the green density. However, using the W/D method, a high-density compact is obtained by applying a comparatively low ejection force.

4.2.2 Mechanical properties of sintered compacts

The tensile strength and Charpy impact value of the sintered compact are shown in **Fig. 6**. Both tensile strength and Charpy impact value increase with sintered density. Under the same compacting pressure, the W/D method attained a sintered density about 0.2 Mg/m^3 higher than the C/C method. When compacted under the pressure of 686 MPa, a sintered density exceeding 7.5 Mg/m^3 was attained. The resultant tensile strength and impact value were as high as 1 000 MPa and 40 J/cm^2, respectively.

The pore distribution over cross section of the sintered compact prepared by the W/D method is shown in **Photo 1**. There was a concern that the lubricant applied over the die might penetrate into the surface layer of the compact and generate coarse pores after sintering. However, no such coarse pores are observed in the surface layer; instead, fine pores are uniformly distributed.

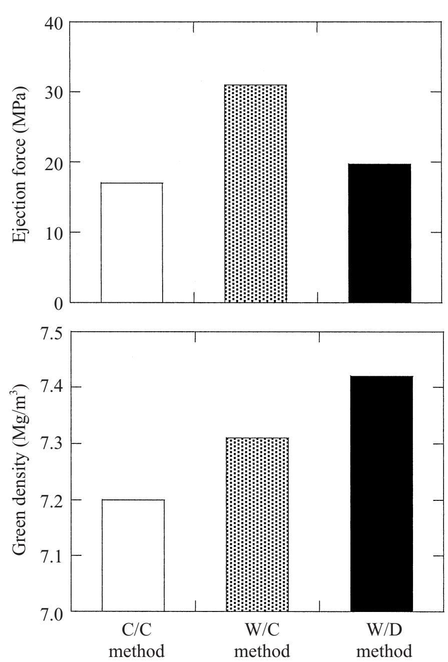

Fig. 5 Ejection forces and green densities of the compacts made by various compaction methods

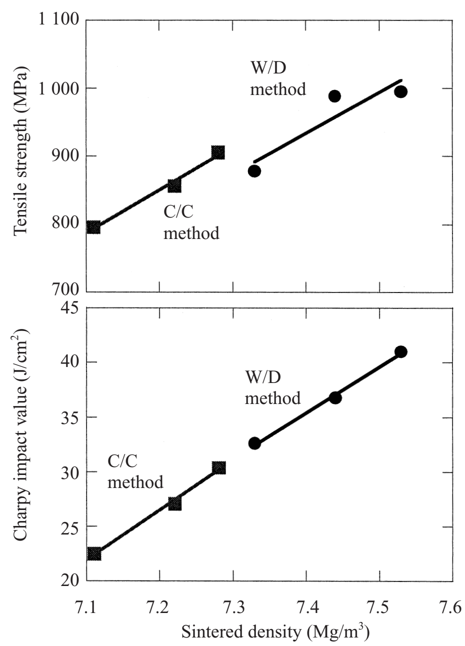

Fig. 6 Tensile strength and Charpy impact value of the sintered compact made by different compaction methods

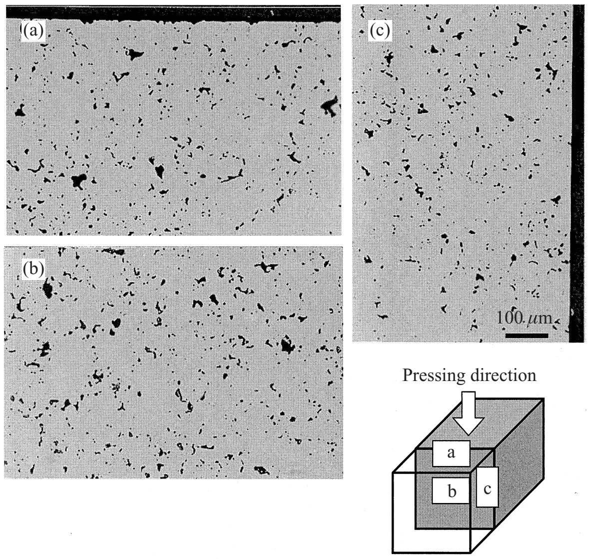

Photo 1 Pore structure of sintered compacts made by W/D method

5. Rotating Bending Fatigue Property of High-Density Sintered and Bright-Quenched Compact Prepared by W/D Method

5.1 Experimental Method

5.1.1 Material

The experiment was conducted using the premixed steel powder composed of KIP SIGMALOY 415S, graphite powder, and lubricant. The chemical compositions of the powders are shown in **Table 2**. The same graphite powder, internal lubricant, and external lubricant as in section 4.1.1 were used.

5.1.2 Conditions for preparing test pieces

Employing the same method as in section 4.1.2, test pieces with two different sizes were prepared by compaction and sintering treatments. One was 55 mm long, 10 mm wide, and 10 mm high; the another was 80 mm long, 15 mm wide, and 15 mm high. Some of the sintered compacts were machined into round-bar test pieces, each having a 5 mmϕ and 15 mm long parallel portion, for the tensile tests, and smooth round-bar test pieces, each having an 8 mmϕ and 15.4 mm long parallel portion, for the rotating bending fatigue test. After machining, these test pieces were heated in the Ar atmosphere at 900°C for 30 min., quenched in to oil at 50°C, and tempered at 200°C for 60 min.

5.1.3 Evaluating method

The tensile strength and impact value were measured employing the same methods as in section 4.1.3. Using the aforementioned smooth round-bar test pieces, the rotating bending fatigue test was performed in the Ono-type rotating bending fatigue tester under the conditions of a revolution speed of 3 000 rpm and the stress ratio $R = -1$. The endurance limit was obtained as the maximum stress level that did not cause failure at the number of cycles of 10^7. The amount of retained austenite (γ) was obtained by X-ray diffraction analysis using MoKα ray, where the integrated intensity on the (110), (200), and (211) planes for α and that on the (111), (200), (220), and (311) planes for γ were measured and the γ amount was calculated from Eq. (1) employing the direct comparison method.[10]

$$V_\gamma = 100 / (1 + I_\alpha R_\gamma / I_\gamma R_\alpha) \cdots\cdots (1)$$

V_γ : Volume fraction of austenite (vol%)
I_α : Integrated intensity of martensite
I_γ : Integrated intensity of austenite
R_α : Theoretical integrated intensity of martensite
R_γ : Theoretical integrated intensity of austenite

5.2 Experimental Result and Discussion

5.2.1 Mechanical properties of sintered and bright-quenched compacts

Densities and mechanical properties of the sintered and bright-quenched compacts made by the W/D method are shown in **Table 3**. The green density of the low carbon compact (graphite content: 0.6 mass%) is 0.07 Mg/m^3 higher than that of the high carbon compact (graphite content: 1.0 mass%). This is because the specific gravity of graphite (2.27) is lower than that of steel powder (7.87). Therefore, the theoretically attainable density decreases as the graphite content increases. However, the relative green densities (the ratios against theoretical densities) are the same 96% at both graphite contents. The sintered densities are also nearly equiv-

Table 2 Chemical composition and compacting condition of the studied powders for investigating the effect of carbon content

	Graphite	Internal lubricant	Compacting temperature	Compacting pressure	External lubricant
Low carbon compact	0.6 mass%	KW-wax 0.2 mass%	130°C	686 MPa	WD2**
High carbon compact	1.0 mass%				

* Lubricant for warm compaction, ** Lubricant for heated die

Table 3 Densities and mechanical properties of sintered and bright-quenched compacts made by W/D method

	Carbon content (mass%)	Green density (Mg/m^3)	Sintered density (Mg/m^3)	Tensile strength (MPa)	Charpy impact value (J/cm^2)
Low carbon compact	0.52	7.43	7.53	1 800	44
High carbon compact	0.92	7.36	7.51	1 250	25

alent at both graphite contents, exhibiting high values that exceed 7.5 Mg/m^3. The carbon contents after sintering decreased from the initial graphite contents by 0.08 mass% to 0.52 and 0.92 mass% respectively. The low carbon compact has a tensile strength of 1 800 MPa and impact value of 44 J/cm^2, both being higher than the values for the high carbon compact.

The results of the rotating bending fatigue test are shown in **Fig. 7**. The rotating bending fatigue strength of the low carbon compact is 450 MPa (endurance ratio: 0.25). Compared with the material prepared by the conventional double-pressing and double-sintering method[11] (graphite content 0.6 mass%, density 7.34 Mg/m^3, rotating bending fatigue strength 350 MPa), the fatigue strength was improved by approximately 30%. Presumably, the fatigue strength of the material prepared by the W/D method was improved because it had a higher density of 7.53 Mg/m^3. The partially alloyed steel powder with the composition as used in the present experiment tends to harden after sintering. Therefore, it is difficult to increase the density by the second pressing after sintering in the double-pressing and double-sintering method. In contrast, the W/D method is a single-pressing and single-sintering process with no second pressing after sintering. Therefore, this method has an advantage in that it enables the increase of the sintered density even when powder with a composition that tends to harden after sintering is used. The rotating bending fatigue strength of the high carbon compact is 470 MPa (endurance ratio: 0.38). This value is higher than that of the low carbon compact, reversing the trend exhibited in the tensile strength.

The microstructures of the sintered and bright-quenched compacts are shown in **Photo 2**. Both high carbon and low carbon compacts exhibit martensitic structures at the surface as well as at the center. However, they have distinctive microstructures corresponding to carbon contents: the low carbon compact has lath-like patterns while the high carbon compact has lens-like ones.

The hardness distribution along the distance from the surface is shown in **Fig. 8**. The hardness slightly fluctuates, but the values are not much different at the surface and at the center. The hardness of a martensitic structure is generally dependent on carbon content. In the present case, however, the difference by carbon content is not distinctive. Presumably, this is because the size of the tip

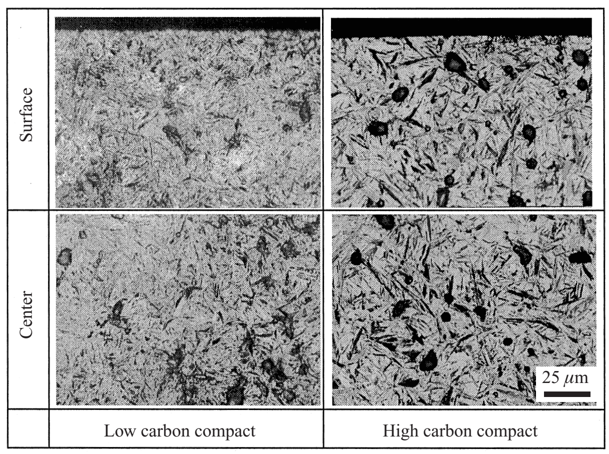

Photo 2 Microstructures of sintered and bright-quenched compacts

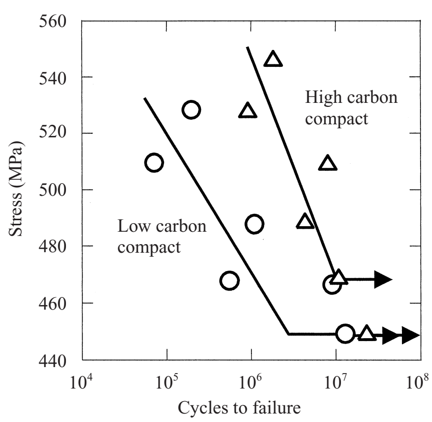

Fig. 7 *S*-*N* curves of rotating bending fatigue test of sintered and bright-quenched compacts

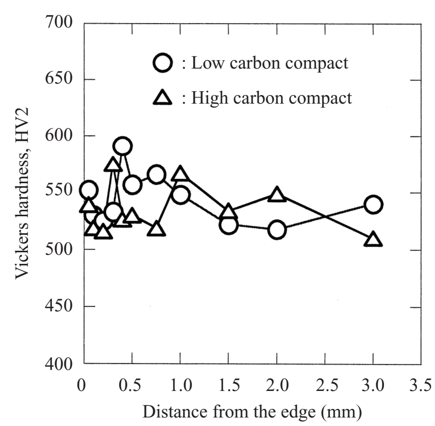

Fig. 8 Hardness distribution of sintered and bright-quenched compacts

of the Vickers hardness tester is sufficiently larger than those of martensite and austenite phases that are finely distributed, and therefore the hardness measurements are averaged.

5.2.2 Mechanism of realizing high fatigue strength

The distribution of Ni content in the sintered compacts was analyzed by EPMA. The result is shown in **Fig. 9**. In the present experiment, the steel powder partially alloyed by Ni powder diffusion was used. However, Ni does not diffuse uniformly after sintering and rather has an uneven distribution. In the high as well as low carbon compacts, the area where Ni of 2 to 6 mass% is present occupies the largest fraction. In the high carbon compact, the area where Ni of more than 6 mass% is present and the area where Ni of less than 2 mass% is present are smaller than in the low carbon compact. Thus, the Ni content has a comparatively more uniform distribution in the high carbon compact.

As shown in Eq. (2)[12], the starting temperature of martensitic transformation (M_S point) lowers as the Ni and C contents increase. As shown in Eq. (3)[13], the amount of γ increases as the M_S point lowers. The amounts of retained austenite were calculated by applying Eqs. (2) and (3) to the Ni content distribution as shown in Fig. 9. In **Fig. 10**, the calculated amounts of retained austenite are compared with those actually measured by the X-ray diffraction method. The amounts of retained austenite calculated from the Ni content distribution are in fairly good agreement with the measured ones. Both calculation and actual measurement indicate that the high carbon compact has a larger amount of retained austenite.

$$M_S = 550 - 350[C] - 40[Mn] - 30[V] - 20[Cr] - 17[Ni] - 10[Cu] - 10[Mo] - 5[W] + 15[Co] + 30[Al] \quad \cdots (2)$$

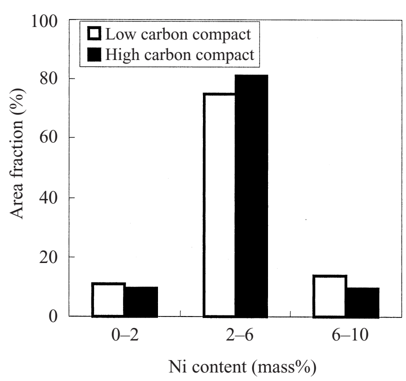

Fig. 9 Ni content distribution in low and high carbon compacts analyzed by EPMA area analysis

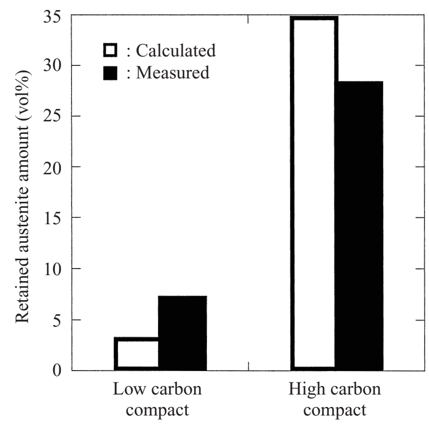

Fig. 10 Amounts of retained austenite calculated and actually measured by X-ray diffraction method

M_S: Starting temperature of martensitic transformation (M_S point) (°C)

[Alloying element]: Alloying element content (mass%)

$$V_\gamma = 6.95 \times 10^{-15}\{455 - (M_S - T_q)\}^{5.32} \quad \cdots (3)$$

V_γ: Volume fraction of austenite (vol%)

T_q: Transformation temperature (In the present experiment, quenching oil temperature is 50°C.)

Some of the research reports on the conventional steel claim that the retained austenite has a favorable effect on fatigue strength[14,15] while others claim it has an unfavorable effect[16]. Apparently, evaluation is dependent on the particular material and test condition employed in each experiment. Proponents for a favorable effect make various explanations on why the retained austenite increases the fatigue strength. One explanation is that compressive residual stress is generated when the retained austenite undergoes deformation-induced transformation. Another explanation is that the propagation of crack is suppressed by the retained austenite. Those who claim an unfavorable effect consider that since the retained austenite is inherently a soft structure, it easily becomes a starting point of fatigue fracture and thus decreases the fatigue strength.

The results of the present experiment indicate that, since the difference in carbon content does not cause much difference in sintered density as well as in hardness distribution, the increase in the amount of residual austenite resulting from the increased carbon content has a favorable effect on the fatigue property and contributes to the improvement of fatigue strength of high carbon compacts.

6. Conclusions

The die-lubricated warm compaction method was developed for producing high-density sintered parts using premixed steel powder containing only a small amount of internal lubricant. The high-density sintered compacts were prepared by this method using partially alloyed steel powder, KIP SIGMALOY 415S, containing 4 mass% Ni, 1.5 mass% Cu, and 0.5 mass% Mo, and their mechanical properties were investigated. The following results were obtained:

(1) Using a newly developed lubricant applicator, the width of the variation in the amount of sprayed lubricant was reduced to 1/4 that of the conventional equipment.

(2) Due to the effect of reducing the amount of internal lubricant, a green density as high as 7.4 Mg/m^3 was obtained by the die-lubricated warm compaction method. This is an improvement of about 0.2 Mg/m^3 over the cold compaction method and of about 0.1 Mg/m^3 over the conventional warm compaction method.

(3) Using the powder with a graphite content of 0.6 mass%, a sintered compact with a density exceeding 7.5 Mg/m^3 was made by the die-lubricated warm compaction method. This density is about 0.2 Mg/m^3 higher than that obtainable by the cold compaction method. The resultant tensile strength and impact value were as high as 1 000 MPa and 40 J/cm^2, respectively.

(4) A sintered and bright-quenched compact prepared using powder with the graphite content of 0.6 mass% exhibited a tensile strength of 1 800 MPa, and impact value of 44 J/cm^2. With a rotating bending fatigue strength of 450 MPa, the fatigue property exceeded that of the material prepared by the conventional double-pressing and double-sintering method.

(5) A sintered and bright-quenched compact prepared using the powder with the graphite content of 1.0 mass% exhibited a rotating bending fatigue strength of 470 MPa (endurance ratio: 0.38). This value is higher than that obtainable from the powder with the graphite content of 0.6 mass%. This improvement of the fatigue strength is presumably attributed to the increase in the amount of residual austenite resulting from the increased carbon content.

In future, the newly developed premixed steel powder and compaction method that can attain high density are expected to be widely applied to the production of powder-metallurgical automotive parts that are required to have high fatigue strength.

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