

# Development of High-strength and High-wear Resistance Nitrocarburizing Steel by Precipitation Strengthening of Mo-V Carbonitride

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## ABSTRACT

The project discussed in this paper developed a nitrocarburizing steel that balances strength, wear resistance, and workability. The hardness of the steel is controlled as low as possible for machining after forging, and the heat applied in the nitrocarburizing process is then used to achieve high hardness in both the surface layer and the base material. As a result, the developed steel achieves at least 40% higher bending fatigue strength than a conventional nitrocarburized steel. The balance between high fatigue strength and workability in the developed steel is realized through the exploitation of Mo-V carbonitride precipitate strengthening. When the material is machined, the Mo and V are in a state of solid solution, but during the nitrocarburization process following machining, fine complex Mo and V carbonitrides are precipitated in lattices coherent with the base material, resulting in high hardness. The fact that the compound layer that formed during nitrocarburization was brittle and lacked ductility represented a disadvantage in terms of abrasive wear, and it was necessary to increase the toughness of the compound layer in order to increase wear resistance. The crystal grains in the compound layer were therefore refined by promoting the precipitation of Mo and V carbonitrides in the compound layer also, resulting in the realization of greater toughness and increased wear resistance.

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## 1. Introduction

Today, against a background of increasingly stringent environmental regulations targeting factors such as CO<sub>2</sub> emissions, we are witnessing rising demands for the reduction of the environmental burden represented by automobiles, for example in the demand for increased automotive fuel efficiency in order to reduce CO<sub>2</sub> emissions. In the area of reciprocating engines, the realization of weight savings and increased compactness through reduction of size is demanded. In line with this, it is necessary to make engine parts more compact, which tends to increase the load on each part, necessitating the realization of high levels of strength and wear resistance. In the case of most engine, drivetrain and other powerplant parts, strength and wear resistance are increased by applying heat treatments such as carburizing, induction hardening or nitrocarburizing when the part has been machined following formation by means of forging or casting<sup>(1), (2)</sup>. Because machining processes cover a wide range of procedures involving lathe machining, drilling and grinding, good workability is essential, and

the balancing of the achievement of high strength with workability is therefore strongly demanded<sup>(3), (4)</sup>.

Given the fact that they impart minimal strain and tend not to result in dimensional change, nitrocarburizing treatments are applied to a variety of parts<sup>(5), (6)</sup>. However, because the realization of increased strength necessitates the use of high-hardness materials, a decline in workability is unavoidable, and limits therefore exist to potential strength increases<sup>(7), (8)</sup>.

In addition, the compounds that form on the surface layer when ordinary carbon steels are nitrocarburized are brittle and display poor ductility<sup>(9)</sup>, and this represents a disadvantage in wear environments in which abrasive wear is prominent.

The research discussed in this paper exploited the precipitation strengthening of molybdenum (Mo) and vanadium (V) during nitrocarburization in order to address the issues discussed above. The paper will analyze the mechanism of precipitation strengthening, and discuss the design of an optimal alloy composition and a microstructure that maximizes precipitation strengthening ability.

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## 2. Development Goals

The research discussed in this paper set out to develop a nitrocarburizing steel that would contribute to the realization of weight savings and increased compactness in engines and transmissions, would be suitable for use in sliding parts exposed to high-friction environments in diesel engines, and would display the following characteristics:

- (1) High strength (40% or higher increase in bending fatigue against conventional nitrocarburizing steels); and
- (2) High friction resistance.

## 3. Concept of Material Composition Design

Figure 1 shows the positions of the porous layer, compound layer, nitrided layer, and base material in a nitrocarburized steel. The porous layer is part of the compound layer, and is composed of the numerous pores that form on the outermost layer. The compound layer is a dense layer formed by the iron nitride compounds under the porous layer. The nitrided layer indicates the depth to which nitrogen diffused from directly below the compound layer during the nitrocarburizing process, and the base material indicates areas to which nitrogen did not diffuse.

### 3.1. Material Concept

Figure 2 shows hardness profiles for a conventional nitrocarburized steel (termed “conventional steel” below) after forging and after nitrocarburization. The increase in the hardness of the conventional steel after nitrocarburization (solid line) against its hardness after forging (broken line) results from the strengthening only of the nitrided layer due to the diffusion of nitrogen in the nitrocarburization process. However, when using the conventional method, because it is necessary to increase hardness following forging when seeking to increase the hardness profile in order to increase strength, a decline in workability is unavoidable. The present research therefore focused on precipitation strengthening using the heat from a nitrocarburizing treatment applied following machining. Figure 3 shows hardness profiles for the developed steel after forging and

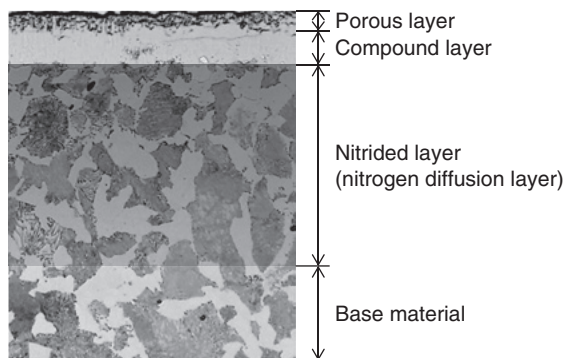


Fig. 1 Layers of nitrocarburizing steel

after nitrocarburization. In the developed steel, because the surface layer and the base material are both increased in hardness by the precipitation of carbonitrides in the nitrided layer of the surface layer and carbides in the base material during nitrocarburization (precipitation strengthening), the steel's hardness after forging (broken line) is lower than its hardness after nitrocarburization (solid line). The material therefore balances high strength with workability, realizing a high level of strength while sacrificing workability as little as possible. In addition, the compound layer that forms as the outermost layer during nitrocarburization is strengthened by precipitation strengthening at the same time.

### 3.2. Concept of Alloy Design for Precipitation Strengthening

Mo, V, Nb, and Ti are among the elements that display high precipitation strengthening ability<sup>(10)</sup>. Figure 4 shows precipitation strengthening ability when each of these elements is individually added to S20C carbon steel. The horizontal axis shows hardness prior to heat treatment at 600 °C for two hours, and the vertical axis shows hardness following heat treatment. The hardness differential,  $\Delta H_V$ , given by Equation (1) indicates precipitation strengthening ability.

$$\Delta H_V = (\text{Hardness after heat treatment}) - (\text{Hardness before heat treatment}) \quad (1)$$

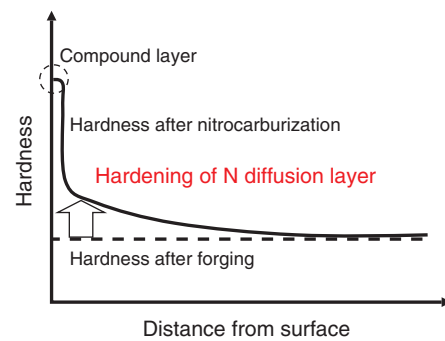


Fig. 2 Hardness distribution of conventional steel

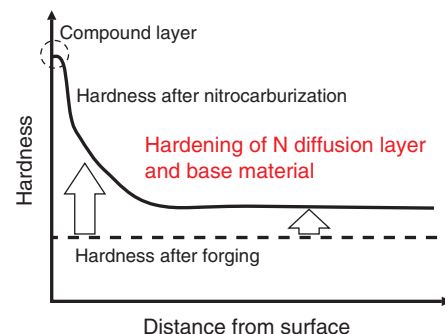


Fig. 3 Hardness distribution of developed steel

The results of this study showed that Mo and V displayed the greatest precipitation strengthening ability, and it was therefore decided to attempt to make effective use of these elements in the new material.

3.3. Mechanism of Precipitation Strengthening of Mo, V-added Steel (Nitrided Layer)

The precipitates in the nitrided layer following nitrocarburization at 600 °C for two hours were analyzed in conventional steel and in the Mo, V-added steel (termed Mo-V steel). Figure 5 shows the results of observation of the nitrided layer (the surface layer at a depth of 0.05 mm) of a conventional steel using transmission electron microscopy (TEM) and Fig. 6 shows the results of precipitate identification using energy-dispersive X-ray spectroscopy (EDS). Figure 7 shows an image of the lattice structure of the precipitates. As Fig. 5 shows, the precipitates in the conventional steel display an NaCl-type crystal structure. The crystal orientation in the plane of observation is the base material, Fe, and [001] $\alpha$ /[ $\bar{1}$ 01]

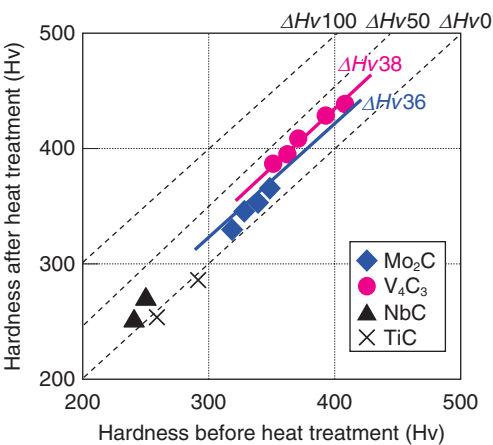


Fig. 4 Precipitate strengthening ability:  $\Delta H_v$  of alloy

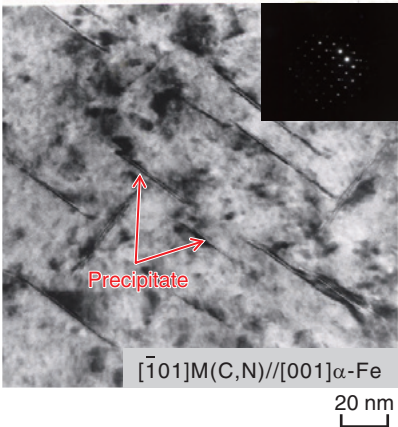


Fig. 5 Transmission electron micrograph taken from diffusion layer (0.05 mm depth) of conventional steel after nitrocarburization

Table 1 Precipitate length and number at 0.05 mm depth after nitrocarburization

|                    | Precipitate length | Precipitate number (/μm <sup>2</sup> ) |
|--------------------|--------------------|----------------------------------------|
| Mo,V-added steel   | 8.2 nm             | $3.8 \times 10^4$                      |
| Conventional steel | 34.4 nm            | $1.5 \times 10^3$                      |

precipitates. That is, precipitation occurs in a Baker-Nutting orientation relationship. The results of analysis of the precipitates using EDS (Fig. 6) show high amounts of Mn and Cr, and they can be judged to be Mn-containing Cr nitrides. The results of the analysis shown in Fig. 5 indicate a precipitate distribution of  $1.5 \times 10^3 / \mu\text{m}^2$  at an average precipitate size of 34.4 nm (Table 1). In addition, the image of the precipitate lattice (Fig. 7) indicates that the lattice of the Fe base material collapses at the boundary where the precipitates are macroaggregated; that is, the boundary has become incoherent.

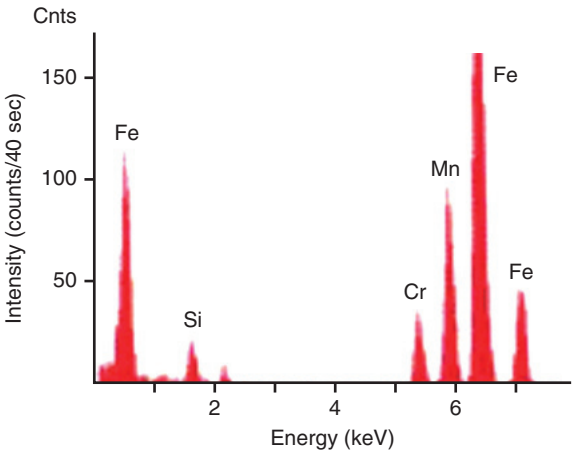


Fig. 6 Energy dispersive X-ray spectrometry analysis of conventional steel precipitate

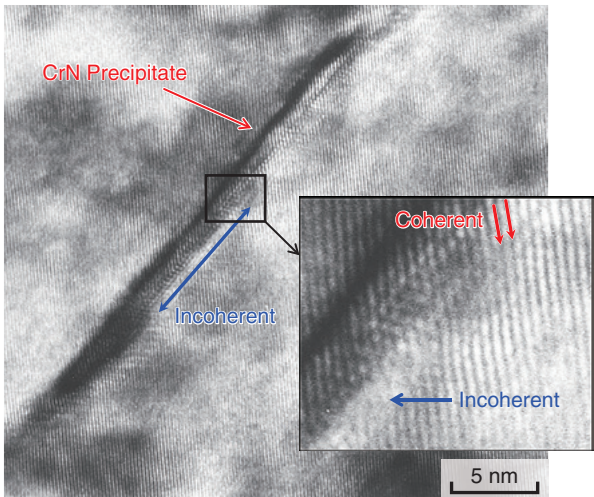


Fig. 7 Lattice image of CrN precipitate

Figure 8 shows the results of observation of the nitrided layer (the surface layer at a depth of 0.05 mm) of the Mo-V steel using TEM and Fig. 9 shows the results of precipitate identification using EDS. Figure 10 shows an image of the precipitate lattice structure. As Fig. 8 shows, the precipitates observed in the Mo-V steel also display an NaCl-type crystal structure. In addition, the crystal orientation in the plane of observation is the base material Fe and  $[001]_{\alpha}/[0\bar{1}1]$ . The Mo-V steel therefore also resembles the conventional steel in the fact that precipitation occurs in a Baker-Nutting orientation relationship. The results of EDS analysis of the precipitates (Fig. 9) show them to contain large amounts of Mo and V, followed by Cr and Mn, and they can be judged to be Cr- and Mn-containing Mo-V carbonitrides. The results of the analysis shown in Fig. 8 indicate a precipitate distribution of  $3.8 \times 10^4/\mu\text{m}^2$  at an average precipitate size of 8.2 nm, with the precipitates both finer and existing in greater quantities than in the case of the conventional steel (Table 1). The image of the precipitate lattice (Fig. 10) shows that strain occurs in the

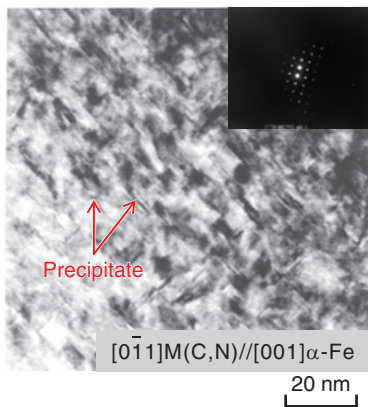


Fig. 8 Transmission electron micrograph taken from diffusion layer (0.05 mm depth) of Mo, V-added steel after nitrocarburization

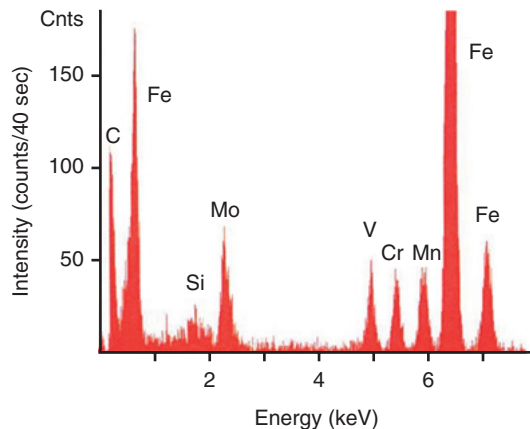


Fig. 9 Energy dispersive X-ray spectrometry analysis of Mo, V-added steel precipitate

lattice (termed “lattice strain”) at the boundary between the Fe base material lattice and the Mo-V carbonitrides. No collapse in the Fe lattice is observed at the boundary, and the precipitate displays extremely good coherence with the Fe base material lattice. It can therefore be indicated that a high level of lattice strain occurs in the Mo-V steel.

Based on the results discussed above, it is considered that because the Mo-V steel, by contrast with the conventional steel, precipitates a large number of fine Mo-V carbonitrides in the nitride layer and the precipitate maintains good coherence with the lattice of the Fe base material, the steel possesses high precipitate strengthening ability.

### 3.4. Mechanism of Precipitate Strengthening of Mo-V Steel (Base Material)

Figure 11 shows the results of TEM observation of the base material of the Mo-V steel, where nitrogen does

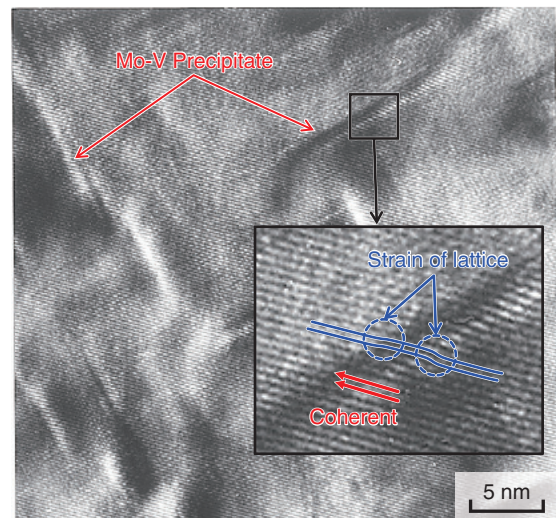


Fig. 10 Lattice image of Mo-V precipitate

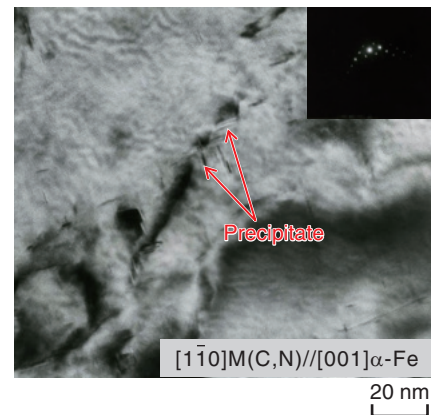


Fig. 11 Transmission electron micrograph taken from non-N diffusion layer of Mo, V-added steel after nitrocarburization

not diffuse, and Fig. 12 shows the results of identification of the precipitates in the base material using EDS. Figure 13 shows an image of the precipitate lattice. As Fig. 11 shows, as in the nitrided layer, the precipitates in the base material display an NaCl-type crystal structure, and the crystal orientation relationship is a Baker-Nutting relationship. Based on the results of the EDS precipitate analysis (Fig. 12), the precipitates contain Mo and V, and can be judged to be Cr, Mn-containing Mo-V carbides. It is believed that carbides are the main precipitate because no nitrogen diffusion occurs in the base material. The results of the analysis shown in Fig. 11 indicate a precipitate distribution of  $5.1 \times 10^3 / \mu\text{m}^2$  at an average precipitate size of 6.8 nm, finer than the surface layer. The image of the precipitate lattice (Fig. 13) shows that strain occurs in the lattice (lattice strain) at the boundary between the Fe base material lattice and the Mo-V carbonitrides, as in the surface layer. No collapse in the Fe base material lattice was observed at the boundary, and the precipitate displays extremely good coherence with the Fe base material.

As this indicates, it is possible to precipitation-strengthen both the nitrided layer and the base material of

the Mo-V steel by means of Mo-V carbonitrides (carbides). This high precipitation hardening ability is believed to increase yield strength, and to produce high fatigue strength.

### 3.5. Mechanism of Increase in Wear Resistance of Mo-V Steel Compound Layer

Figures 14 and 15 respectively show the results of TEM observation of cross-sections of the compound layers of conventional steel and the Mo-V steel. Figure 16 shows the results of EDS identification of the precipitates in the Mo-V steel. As Figs. 14 and 15 show, the Mo-V steel has finer crystal grains than the conventional steel. As Figs. 15 and 16 indicate, Mo-V carbonitrides of a grain size of around 10 nm are also precipitated in the compound layer of the Mo-V steel. These precipitates have a pinning effect, controlling grain growth and refining the compound layer. It is believed that this produces a high-toughness compound layer, resulting in a high level of wear resistance.

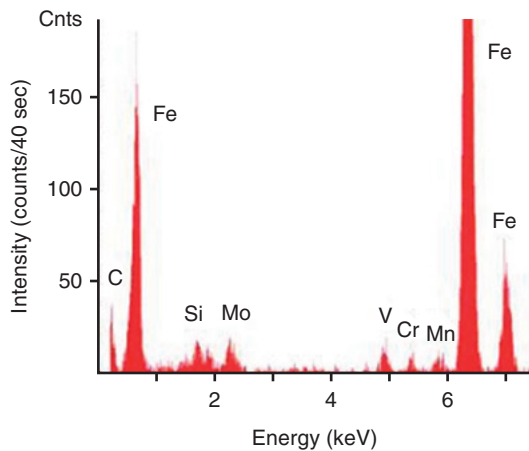


Fig. 12 Energy dispersive X-ray spectrometry analysis of inside precipitate in Mo, V-added steel

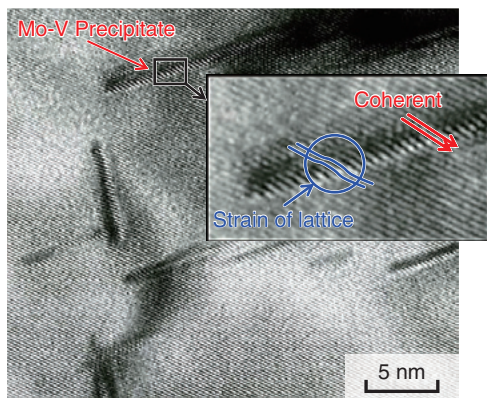


Fig. 13 Lattice image of Mo-V precipitate

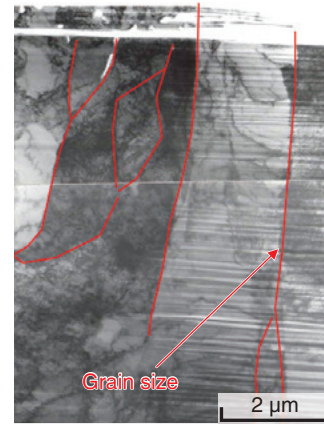


Fig. 14 Transmission electron micrograph taken from diffusion layer of conventional steel compound layer

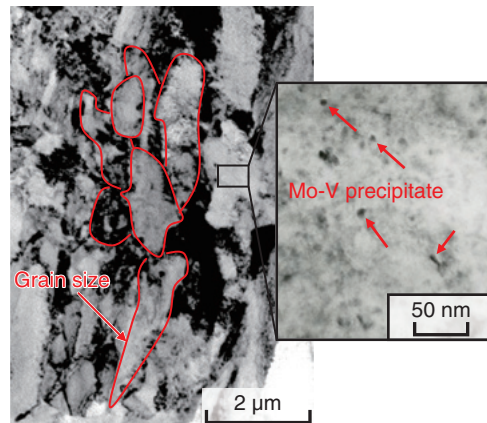


Fig. 15 Transmission electron micrograph taken from diffusion layer of developed steel compound layer

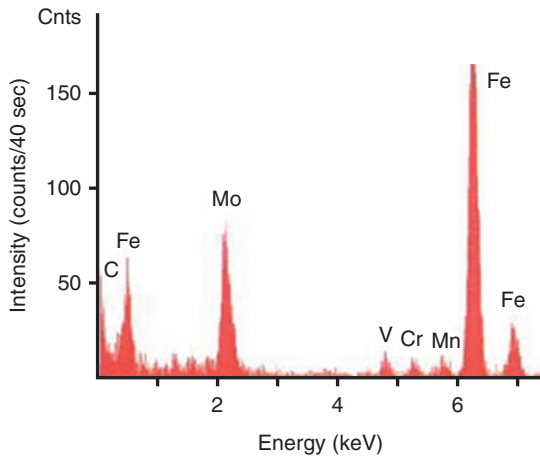


Fig. 16 Energy dispersive X-ray spectrometry analysis of precipitate in developed steel compound layer

## 4. Material Composition Design

### 4.1. Target Hardness and Microstructure

Prior to the design of the material composition, the target hardness profile following nitriding was set. Figure 17 shows the hardness profile (solid line) determined from the stress gradient necessary for the achievement of the target fatigue strength. It was necessary for the hardness profile of the developed material to exceed the hardness profile determined from the stress gradient<sup>(11), (12)</sup>. For the developed material, the depth of nitrogen diffusion during nitrocarburization was set at 0.3 mm or more, and the hardness at a depth of 0.3 mm was set at 295 Hv or higher to help ensure that the fracture point would occur at a depth of 0.3 mm or more. The hardness at a depth of 0.05 mm in the surface layer was set at 450 Hv or higher.

With regard to the microstructure, in order to maximize the Mo-V precipitation strengthening ability necessary to help ensure fatigue strength and the wear resistance of the compound layer, it was necessary to maintain the Mo and

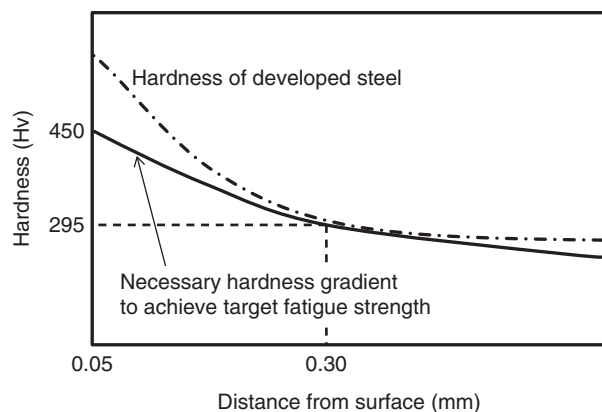


Fig. 17 Hardness transition curve design after nitrocarburization

V in a state of solid solution following forging and cooling. Ferrite forms in the steel during cooling following forging. Figure 18 shows a TEM image of the Mo-V carbides that form in the ferrite microstructure. When ferrite forms during cooling following forging, Mo and V do not maintain a state of solid solution, but rather, as Fig. 18 shows, Mo-V carbides are precipitated as large agglomerations. These Mo-V carbides precipitated in ferrite are more agglomerated and spherical than the Mo-V carbides precipitated in bainite shown in Figs. 11 and 13. This is identical to the state of precipitation of ferrite in general vanadium microalloyed steels. Because the precipitates became larger with the application of heat in the following nitrocarburization process, the expected precipitation strengthening ability could not be achieved. Figure 19 shows the results of a study of the precipitation strengthening ability of ferrite microstructures and bainite microstructures. The horizontal axis shows nitriding time, and the vertical axis shows hardness. The results indicate that the hardness differential before and after heat treatment,  $\Delta H_v$ , is lower for the ferrite microstructure than the bainite microstructure. A bainite microstructure was therefore selected for the material under development in order to boost the effectiveness of increasing strength with nitrocarburization.

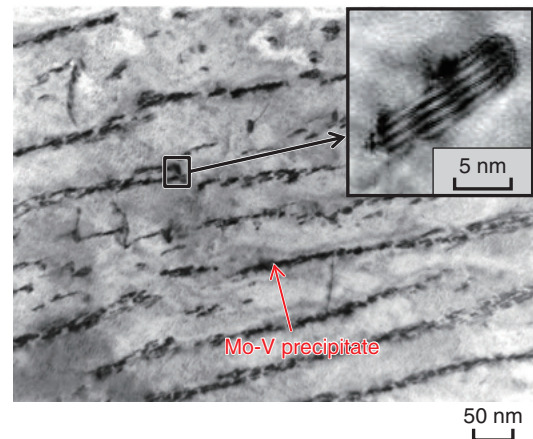


Fig. 18 Mo-V precipitate in ferrite after forging

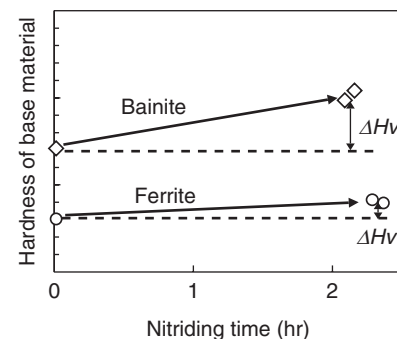


Fig. 19 Relationship between Mo-V precipitate strengthening ability and microstructure

#### 4.2. Setting of Quantities of Mo and V for Addition

The amounts of Mo and V to be added were set on the basis of the amount of V, which displays a high level of precipitation strengthening ability. Figure 20 shows the relationship between the amount of V added and precipitation strengthening ability. At 0.25 mass% or more V, precipitation strengthening ability tends to become saturated, and in general the greater the quantity of V added, the higher the hardness of the base material after forging, resulting in a decline in workability. Given this, the upper limit for the amount of V to be added was set at 0.25 mass%.

Figure 21 shows the relationship between the amount of added Mo and V and hardness at a depth of 0.3 mm following nitrocarburization at 600 °C for two hours. Target values for the amount of added Mo and V to achieve a hardness of 295 Hv or higher were set at 0.20 mass% for Mo and 0.20 mass% for V.

The amounts of Mo and V to be added were set as described above with consideration of the realization of precipitation strengthening ability and the necessary level of hardness following nitrocarburization. In addition, the amounts of C, Si, Mn and Cr added were adjusted to produce the desired bainite microstructure.

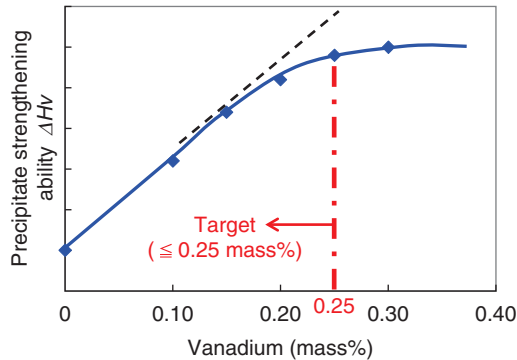


Fig. 20 Relationship between vanadium content and precipitate strengthening ability:  $\Delta H_v$

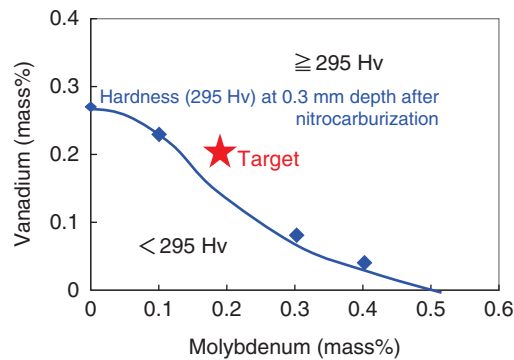


Fig. 21 Relationship between Molybdenum, Vanadium content and hardness at 0.3 mm depth after nitrocarburization

Table 2 Chemical composition of developed and conventional steel

|                    | (mass%) |      |      |      |      |      |
|--------------------|---------|------|------|------|------|------|
|                    | C       | Si   | Mn   | Cr   | Mo   | V    |
| Developed steel    | 0.20    | 0.50 | 1.85 | 0.20 | 0.20 | 0.20 |
| Conventional steel | 0.48    | 0.30 | 1.45 | 0.10 | –    | –    |

Table 2 shows the material composition set on the basis of the results discussed above. The table also shows the composition of conventional steel.

### 5. Material Characteristics

#### 5.1. Fatigue Characteristic

Rotating-bending fatigue tests were conducted using the test specimen specifications shown in Fig. 22. Test specimens of 44 mm × 44 mm were produced by machining after microalloyed forging, following which they were subjected to nitrocarburizing treatment at 600 °C for two hours. Figure 23 shows hardness following forging (broken lines) and hardness profiles following nitrocarburization (solid lines). While the hardness of the developed material following forging is about 270 Hv (the blue broken line in the graph), following nitrocarburization the material's internal hardness increases by 30 Hv or more, reaching the target hardness (the black solid line in the graph), including in the nitrified layer. In addition, as Fig. 24 shows,

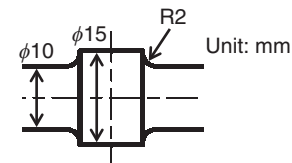


Fig. 22 Geometry of rotating-bending fatigue test specimen

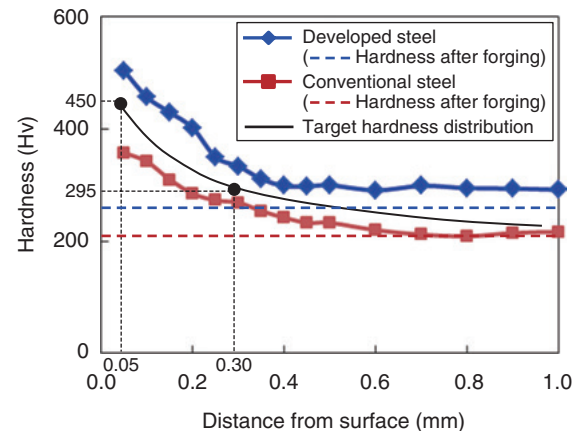


Fig. 23 Hardness distribution of test specimen after nitrocarburization

while the conventional steel displays a ferrite-pearlite microstructure, the developed steel realizes a monolayer bainite microstructure. The bending fatigue strength of the developed material is at least 40% higher than that of the conventional steel, as Fig. 25 shows.

## 5.2. Wear Resistance of Metal Sliding Shaft Parts

Toughness against abrasive wear due to hard particles such as carbon particles in deteriorated oil is demanded for engine parts. Using  $20 \times 20 \times 10$  mm (L  $\times$  W  $\times$  H) test specimens, scratch tests were therefore conducted on the conventional and the developed steel under a constant load of 80 N, and the characteristics of the compound layer formed on the outermost surface were evaluated. Scratch tests are suitable to evaluate the characteristics of compound layers in relation to abrasive wear due to hard particles.

The steels employed in the samples for the study of nitriding quality, as shown in Fig. 24, were used to produce the test specimens. The results of the tests indicated that the developed steel formed a compound layer approximately 80 Hv harder than the conventional steel. Figure 26 shows the results of an analysis of the crystal grains in cross-sections of the compound layers of the developed and conventional steels using electron backscattering patterns (EBSP). The results demonstrate that the developed steel forms a fine compound layer, with the crystal grain diameter of  $1.04 \mu\text{m}$  around  $1/3$  of the figure for the conventional material of  $3.12 \mu\text{m}$ . In order to verify the effect of this refinement of the crystal size in the compound layer on wear resistance, samples of the conventional and developed steels were produced in which the porous layer that forms

on the outermost surface of the compound layer had been removed by abrasion prior to scratch tests. Following the scratch tests, the scratch marks were observed using an optical microscope in order to evaluate the detachment resistance of the compound layer (Fig. 27). The results show that while detachment occurred across the entire surface of the conventional steel, detachment did not occur in the developed steel. The results discussed above indicate that the developed steel forms a high-hardness and high-toughness compound layer.

Unit tests reproducing an actual engine environment were also conducted in order to evaluate wear resistance in an actual engine. Figure 28 shows a diagram of the test apparatus. This test uses oil with a high carbon content to reproduce deteriorated oil, and is suitable to quantitatively evaluate shaft wear in actual diesel engines, a severe abrasive wear environment, in a short period<sup>(13)</sup>. The results are shown in Fig. 29, with time on the horizontal axis and amount of wear on the vertical axis. As the figure shows, the amount of wear in the developed steel following 200 hours of testing was approximately 50% of that in the conventional steel, verifying that the developed steel forms a high-abrasion-resistance compound layer.

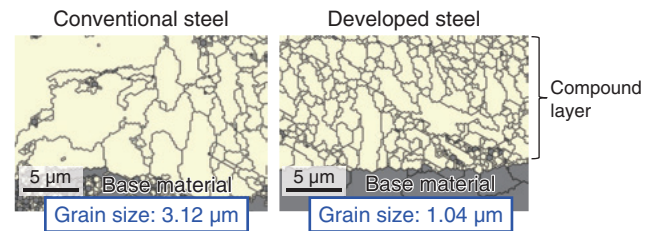


Fig. 26 EBSP analysis of compound layer

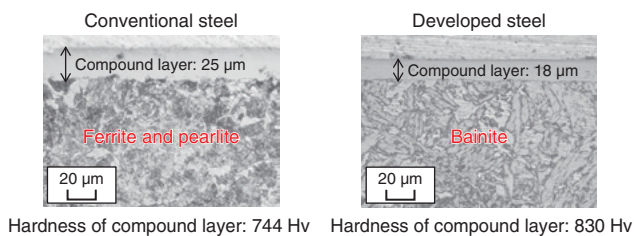


Fig. 24 Microstructure of test specimen after nitrocarburization

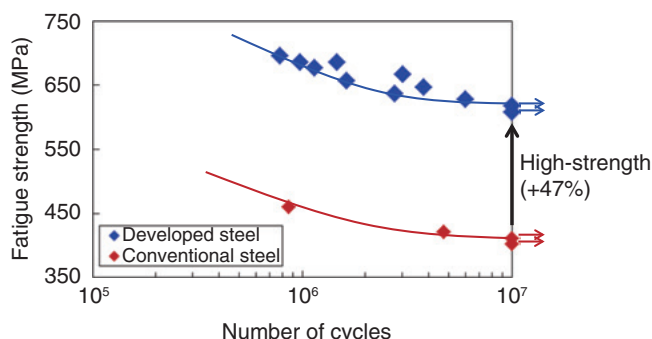


Fig. 25 Result of rotating-bending fatigue strength

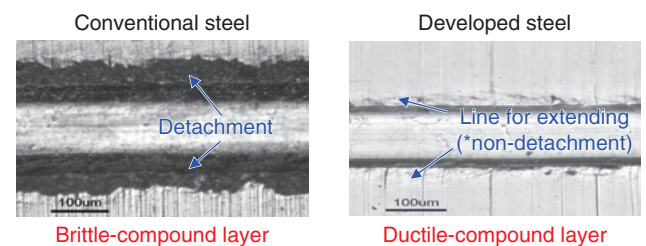


Fig. 27 Compound layer of developed steel and conventional steel after scratching test

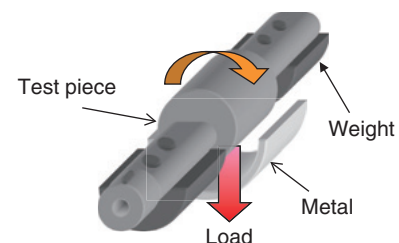


Fig. 28 Abrasion test of shaft

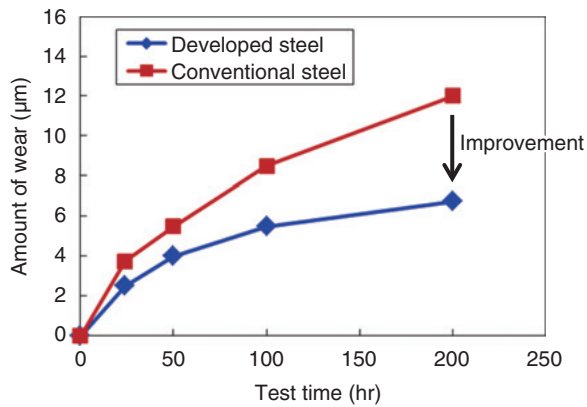


Fig. 29 Relation between test time and wear amount

## 6. Conclusion

A high-strength and high-wear-resistance nitrocarburizing steel has been developed using Mo-V carbonitride precipitation strengthening. The development produced the following findings:

- (1) Using Mo-V carbonitride precipitation strengthening, it was possible to control the hardness of the base material to 270 Hv during machining and to achieve high hardness levels of 300 Hv or higher following nitriding, thus realizing high strength without sacrificing workability.
- (2) The use of a bainite microstructure made it possible to maintain Mo and V in a state of solid solution following forging and cooling, helping to enable the realization of the maximum Mo and V age-hardening ability.
- (3) The high strength achieved through the addition of Mo and V resulted from the high lattice strain produced by the precipitation of fine (about 8 nm) Mo-V precipitates coherent with the Fe lattice in the nitrided layer that forms during nitrocarburizing and the base material.
- (4) The precipitation of Mo-V carbonitrides in the compound layer refined and increased the strength of the layer, helping to enable the realization of a compound layer displaying good wear resistance.

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