

Development of Steel for High-strength Crankshaft by Precipitation Strengthening of Mn-nitride

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ABSTRACT

A high-strength gas soft-nitrided crankshaft steel that does not rely on the addition of rare metals has been developed. High strength is realized in Honda's conventional high-strength crankshaft steel through the addition of the rare metal molybdenum (Mo). By contrast, a high level of strength was achieved in the developed steel by making effective use of manganese (Mn), a universal component of steel, in the production process. High strength was realized by strengthening the ferrite phase using Mn content of 0.9 wt% or higher. It was determined that the strengthening mechanism was precipitation strengthening by means of Mn nitrides (η -Mn₃N₂) formed in the surface layer ferrite phase as a result of gas soft nitriding. Straightenability, a characteristic that is antagonistic to high strength, is also demanded from crankshaft steels following gas soft nitriding. This demand was responded to in the developed steel by increasing toughness through the reduction of carbon (C) content in order to increase the area ratio of the ferrite phase. The innovations described above have made it possible to reduce material costs by doing away with Mo while realizing an equivalent level of strength to the conventional high-strength steel. Strength has also been increased by 16% against crankshafts produced using the conventional standard steel, which contains no rare metals.

1. Introduction

Against the background of increasing demands from society for the realization of higher automotive fuel efficiency, automakers are pursuing the achievement of size and weight reductions in the crankshaft and other drivetrain parts in order to realize weight savings and reduce friction. A high level of fatigue strength is necessary in the crankshaft because it is constantly subject to repeated stress. A variety of mechanisms can be used to increase the strength of steel materials, including work hardening (dislocation strengthening), grain refinement strengthening, solid solution strengthening, and precipitation strengthening. Secondary strengthening methods used to elicit these strengthening mechanisms include induction hardening, roll processing, and gas soft nitriding (GSN)⁽¹⁾⁻⁽⁴⁾. GSN is a method that increases hardness close to the surface of the material by diffusing nitrogen (N) from the surface to the inside. This method also makes it possible to increase the wear resistance of the sliding parts by means of the hard nitrogen compound layer (ϵ phase: Fe₂₋₃N; γ' phase: Fe₄N) that forms the surface. In many cases, rare metals such as Mo and vanadium (V) are added when GSN is used as

a strengthening method^{(5), (6)}. However, due to significant increases in the cost of rare metals and the instability of the procurement situation, manufacturers are today focusing attention on reducing rare metal content or eliminating the use of the metals altogether. For example, a method has been developed that makes it possible to achieve a high strength without adding rare metals by optimizing the copper (Cu) and chrome (Cr) content of the scrap using an electric furnace, promoting precipitation strengthening by means of the Cr and ferrite solid solution strengthening by means of the Cu⁽⁷⁾. The research discussed in this paper studied a method of achieving a high level of strength in steel produced in a blast furnace process without adding rare metals.

2. Development Goals

Following forging and machining, GSN is applied to Honda crankshafts for gasoline engines. Non-normalized steels, which eliminate the normalizing process following forging, are used as the crankshaft materials. Depending on the load levels of bending and twisting stress to which they will be subjected, standard steels and high-strength steels

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used properly. Mo is added in order to realize a high level of strength in the high-strength material, and reducing the amount of this rare metal used therefore represents an issue. The present research studied a method of compensating for the decline in strength when Mo is not added by effectively using the basic elements composing the steel. At the same time, the crankshafts are prone to bending due to the heat stress produced during GSN, and bend reform is therefore performed in order to help ensure that the level of bending remains within specified limits. However, when the hardness of the surface increases following the application of GSN to increase strength, straightenability tends to decline. The present research therefore focused on the realization of a gas soft-nitrided steel for crankshaft use that could reconcile the antagonistic characteristics of high strength and straightenability without using rare metals by means of material design with the basic elements carbon (C) and Mn as a base.

In the development of the steel, material specifications were studied based on the following development aims:

- (1) Equivalent level of fatigue strength to conventional high-strength steel
- (2) Equivalent level of straightenability to conventional high-strength steel
- (3) Equivalent cost to conventional standard steel

3. Concept of Material Composition Design

3.1. Concept of Strengthening of Microstructure

Non-normalized steels are employed in crankshafts to make it possible to rationalize the heat treatments used in the production process. The pinning effect of fine nitrides formed by the addition of nitrogen helps to control the coarsening of the microstructure during hot forging. The material displays a two-phase microstructure, with a hard pearlite microstructure and a soft ferrite phase. Figure 1 shows a standard microstructure and hardness profile for a non-normalized steel following GSN. GSN forms a hard compound layer at the surface of the material, and hardness of the nitrogen diffusion area is increased against the matrix.

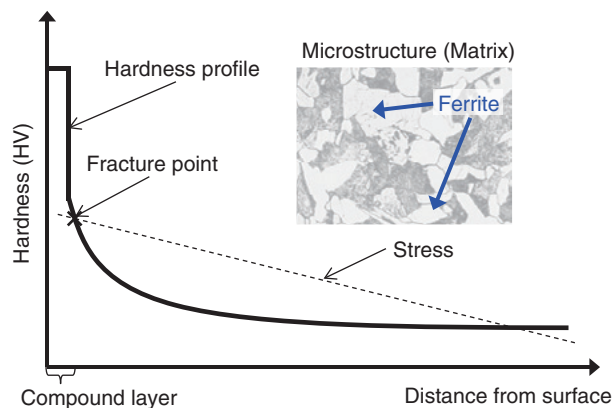


Fig. 1 Schematic of hardness profile after GSN

Controlling cracks is an important consideration in crankshafts, given the potential for fatigue failure resulting from repeated stress. Because cracks originate and develop in the ferrite phase directly below the compound layer when stress exceeds material strength, it is important to strengthen the ferrite phase in order to realize high strength.

The present research measured hardness at a depth of 50 μm from the surface layer using Vickers hardness tests in order to evaluate the effect of strengthening of the ferrite phase directly below the compound layer. This was defined as 50 μm depth hardness.

3.2. Study of Elements for the Realization of High Strength

The present development attempted to realize high strength through the effective use of basic elements in the steel as a substitute for the addition of rare metals. The basic elements present in steel are C, silicon (Si), Mn, sulfur (S), and phosphorous (P). Of these, Mn is known to be highly effective in strengthening the ferrite phase by means of solid solution strengthening. This strengthening mechanism occurs due to the fact that Mn atoms are a different size to Fe atoms, and when Mn exists in solid solution in Fe, it distorts the lattice arrangement of the Fe atoms, increasing deformation resistance. It was considered that the effective introduction of nitrogen by means of nitriding to a material with strain fields of this type would increase strength by further increasing deformation resistance.

It has also been reported that the fatigue strength of plasma-nitrided C-Mn alloys increases with an increasing additive amount of Mn, indicating the possibility that the mechanism of strengthening is precipitation strengthening due to nitrides⁽⁸⁾.

In place of the conventional method of realizing high strength through the addition of Mo, the present research therefore sought to increase strength through nitrides, by means of increasing the additive amount of Mn, and studied the mechanism producing the effect.

3.3. Effect of Mn

The effect of the additive amount of Mn on fatigue strength was investigated as a preliminary study towards chemical composition design. Test specimens were produced by varying the additive amount of Mn between 0.5 wt% and 1.5 wt% in a 0.30-0.35 wt% carbon steel and applying GSN. Figure 2 shows the results of rotary bending tests. Fatigue strength increases with the addition of increasing amounts of Mn, and becomes equivalent to that of conventional high-strength steel at 0.9 wt% and above. This was therefore set as the necessary additive amount of Mn for the realization of high strength.

3.4. Concept of Realizing Straightenability

A method of increasing straightenability by controlling 50 μm depth hardness and increasing the area ratio of the ferrite phase has been reported⁽⁷⁾. Figure 3 shows the area

ratio of the ferrite phase, α , in a model alloy when the amount of C added is varied. The less C added the greater the increase in the ferrite phase. Chemical composition design in the present research sought to increase the area ratio of the ferrite by controlling the additive amount of C, a basic element, in order to help realize the target straightenability.

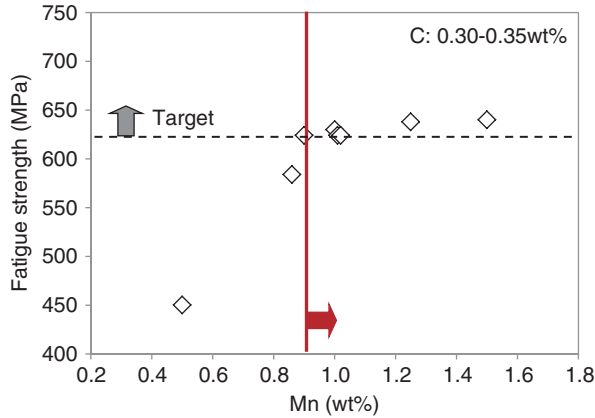
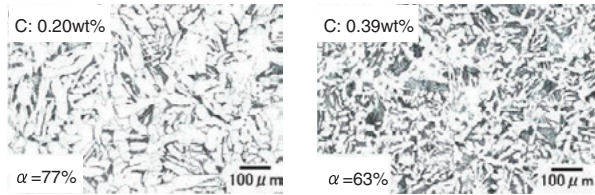


Fig. 2 Relationship between Mn and fatigue strength



(a) 0.20wt%C-0.03wt%Si-0.02wt%Mn (b) 0.39wt%C-0.02wt%Si-0.02wt%Mn

Fig. 3 Relationship between ferrite area ratio and C content

4. Chemical Composition Design

4.1. Setting Target Hardness

The range of 50 μm depth hardness following GSN to reconcile bending fatigue strength and straightenability was studied. Figure 4 shows the relationship between 50 μm depth hardness and bending fatigue strength. Fatigue strength standards were satisfied at hardness levels of 300 HV and above. Figure 5 shows the relationship between 50 μm depth hardness and straightenability. Straightenability standards were satisfied at 50 μm depth hardness of 360HV and below. The range of 50 μm depth hardness necessary to realize parts functions was therefore set at 300-360 HV.

4.2. Setting Components for Material under Development

In designing the chemical composition for the material under development, it was important to set a range for the components that would satisfy the 50 μm depth hardness set in Section 4.1. In doing so, it was necessary to establish an indicator, in consideration of the fact that the effect of

each of the elements making up the steel differed. The equivalent amount of Mn (M_{neq}) was set as an indicator of the 50 μm depth hardness for the chemical composition of the material under development. M_{neq} is defined by Eq. (1). M_{neq} is obtained by converting the amount of the elements that have an effect on the 50 μm depth hardness following GSN into an amount of Mn, and is set using Mn, C, and Cr, which have a strong effect on hardness. Figure 6 shows the

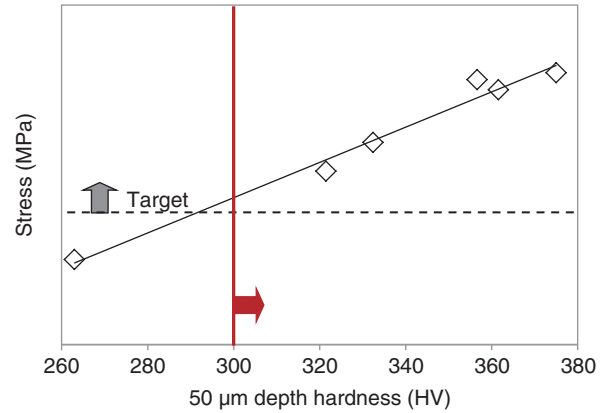


Fig. 4 Relationship between 50 μm depth hardness and bending fatigue strength

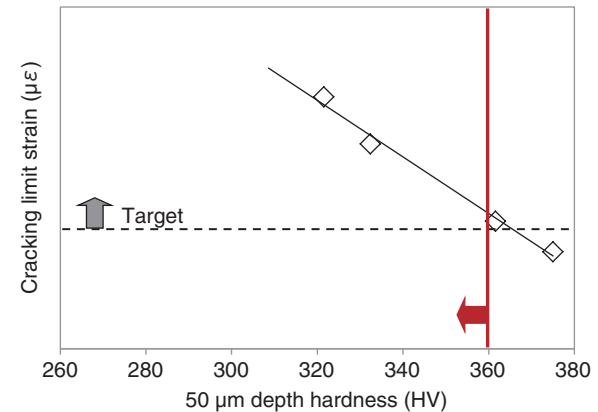


Fig. 5 Relationship between 50 μm depth hardness and cracking limit strain

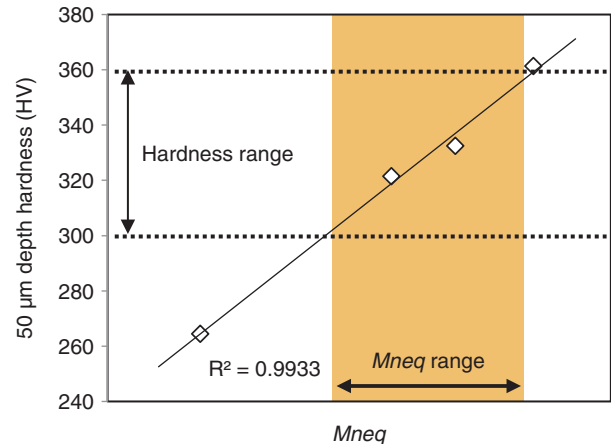


Fig. 6 Relationship between M_{neq} and 50 μm depth hardness

Table 1 Chemical composition of developed and conventional steel (wt%)

	C	Si	Mn	P	S	Cr	Mo
Developed high-strength steel	0.30	0.20	1.00	0.015	0.05	0.05	-
Conventional high-strength steel	0.37	0.20	0.82	0.015	0.07	0.05	0.16
Conventional standard steel	0.40	0.20	0.60	0.015	0.05	0.05	-

relationship between $Mneq$ and the 50 μm depth hardness. As the figure shows, the 50 μm depth hardness increases proportionally with increase in $Mneq$, indicating the validity of the setting of $Mneq$. Using this relationship, a range for $Mneq$ that would help to enable the realization of the target 50 μm depth hardness was set.

$$Mneq = Mn + a1 \cdot C + a2 \cdot Cr \quad (1)$$

Mn, C, Cr: Amount of each element (wt%)
 $a1, a2$: Constants

With the additive amount of Mn set in Section 3.3 (0.9 wt% and above) as a base, the chemical composition for the developed high-strength steel that would satisfy Eq. (1) was set. Table 1 shows representative chemical compositions.

The composition of the developed high-strength steel uses the composition of the conventional standard steel as a base, and eliminates the Mo content, increases the Mn content, and reduces the C content against the conventional high-strength steel. Because excessive reduction of the C content might result in a decline in wear resistance, C content was set at 0.3wt% for the developed high-strength steel.

5. Characteristics of Developed High-strength Steel

5.1. Material Characteristics

Figure 7 shows a cross-section of the microstructure close to the surface, Fig. 8 shows the nitrogen concentration profile, and Fig. 9 shows the hardness profile of the developed high-strength steel following gas soft nitriding. Figure 8 shows that the nitrogen concentration close to the surface of the developed high-strength steel is equivalent to that of the conventional high-strength steel. Figure 9 shows that the level of hardness of the inside of the developed high-strength steel is equivalent to that of the

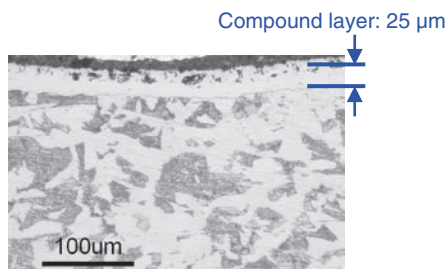


Fig. 7 Microstructure of developed steel after GSN

conventional standard steel, while gas soft nitriding has increased the surface hardness of the material to a level equivalent to that of the conventional high-strength steel. In other words, while the developed high-strength steel uses the same constituent elements as the conventional standard steel, it achieves a higher nitrogen concentration and a higher surface hardness. This is believed to be an effect of precipitation strengthening due to Mn nitrides formed as a result of the addition of an increased volume of Mn.

5.2. Characteristics of Crankshaft

A crankshaft for an inline 4-cylinder gasoline engine was produced using the developed high-strength steel, and the characteristics of the crankshaft in isolation were evaluated. Figure 10 compares the bending fatigue and straightenability of the developed steel with those of the conventional steels. The levels of these characteristics are shown as percentages, with the conventional high-strength steel as 100.

The fatigue strength of the developed high-strength steel is similar to that of the conventional high-strength steel, and 16% higher than that of the conventional standard steel. In terms of straightenability, while the developed high-strength steel falls behind the conventional standard steel, which has low surface hardness, its level is similar to that of the conventional high-strength steel.

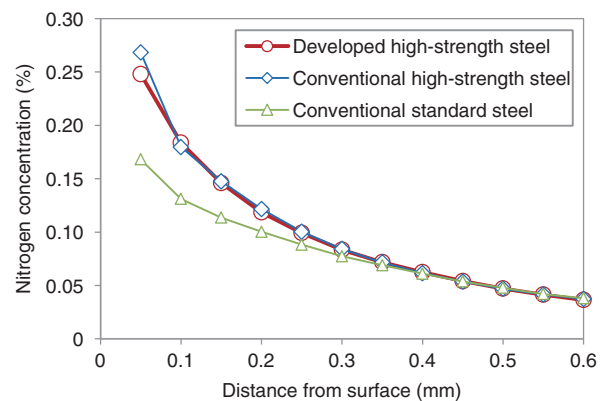


Fig. 8 Nitrogen concentration profile after GSN

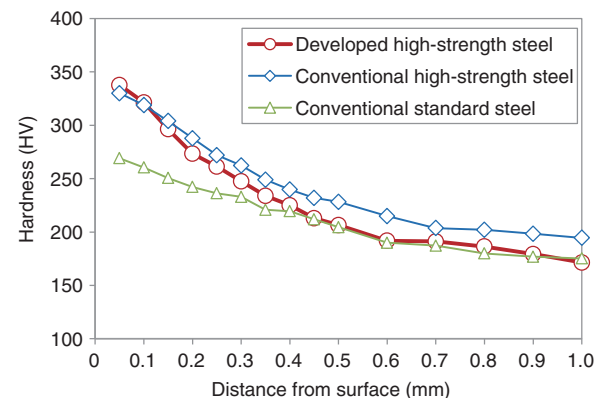


Fig. 9 Hardness profile after GSN

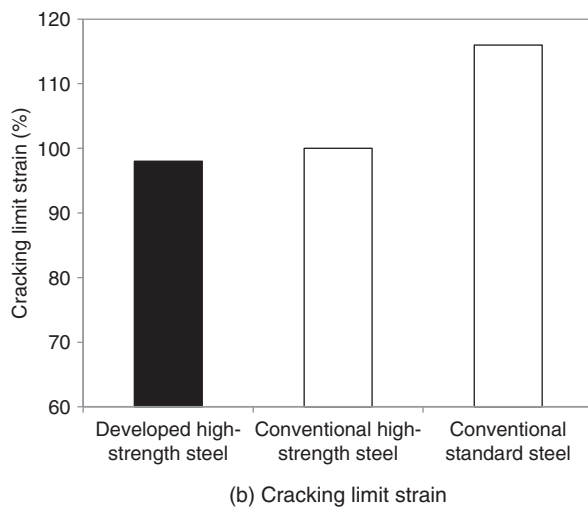
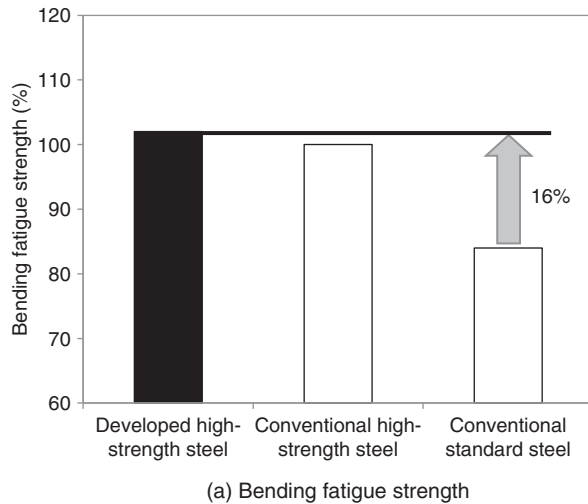


Fig. 10 Crankshaft characteristic

Table 2 Chemical composition (wt%)

	C	Si	Mn	P	S
0.3C-0.5Mn	0.30	0.01	0.48	0.002	0.001
0.3C-1.0Mn	0.29	0.01	0.97	0.002	0.001
0.3C-1.5Mn	0.30	0.01	1.43	0.002	0.001
0.3C-2.0Mn	0.29	0.01	1.92	0.002	0.001

ratio of the pearlite microstructure (the black regions in the photographs). The pearlite microstructure increases in proportion with increasing Mn content, and it is thought that hardness increases as a result, but in order to realize high strength it is necessary to increase the strength of the soft ferrite phase.

6.2.2. Effect of Mn on N concentration

Figure 13 shows the Mn content and nitrogen concentration of the test materials following GSN. The nitrogen concentration near the surface increases with increasing Mn content. It is believed that Mn effectively contributes to the diffusion of nitrogen in the inside of the material.

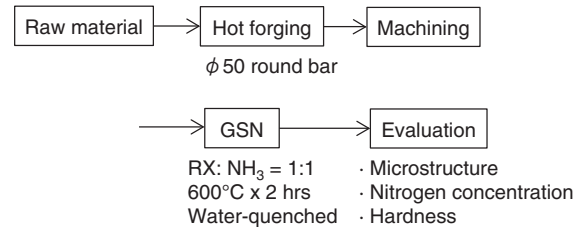


Fig. 11 Method of specimen preparation and property evaluation

6. Analysis of High-Strengthening Mechanism

6.1. Test Specimen and Test Method

Model alloys of 0.3C-(0.5, 1.0, 1.5, 2.0)Mn were produced in order to clarify the high-strengthening mechanism via Mn (Fig. 11). Table 2 shows the chemical compositions of the model alloys used in the tests. The test materials were forged at 1250°C to form round bars of $\phi 50$ mm, which were machined for use in rotary bending tests. Following GSN (using RX gas and NH_3 gas) at 600°C for two hours, the test specimens were water-quenched. The microstructures of these test specimens were observed, and measurements of nitrogen concentration and hardness were conducted.

6.2. Effect of Mn on Material Characteristics

6.2.1. Effect of Mn on microstructure

Figure 12 shows photographs of cross-sectional microstructures of the model alloys close to the surface. Increasing the amount of Mn added increases the area

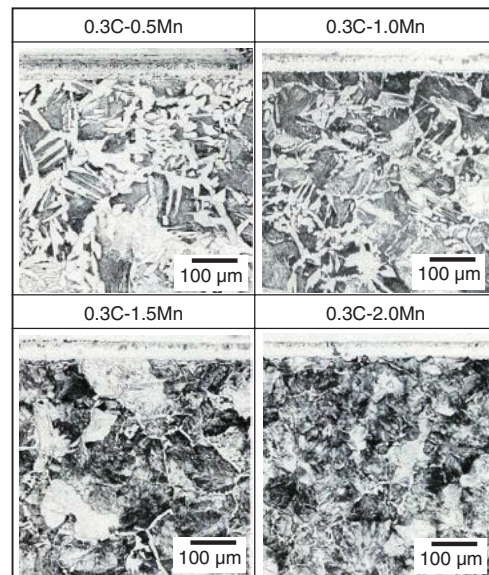


Fig. 12 Microstructure after GSN

6.2.3. Effect of Mn on hardness

Figure 14 shows measurement results for the 50 μm depth hardness following GSN. Hardness was measured by means of Vickers hardness tests using a 300 g indentation load. Vickers hardness increases with increasing Mn content. It is considered possible that this result originates in the hard pearlite microstructure, which increases as the Mn content increases. In order to study the effect of Mn on increasing the hardness of the soft ferrite phase, the hardness of the ferrite phase was measured by reducing the indentation load to 50 g and localizing the measurement area. The hardness of the ferrite phase increased with increasing Mn content. It is conjectured that the increase in fatigue strength with increased Mn content shown in Fig. 2 originates in an increase in the hardness of the ferrite phase.

6.3. Identification of Precipitates in Ferrite

The fine structure of the ferrite phase was observed using field emission-type transmission electron microscopy (FE-TEM) in order to study the strengthening mechanism by Mn. The 0.3C-1.0Mn material, having the same amount of C and Mn as was set in Section 4.2, was selected as the chemical composition for FE-TEM observation to clarify the strengthening mechanism. The test specimen

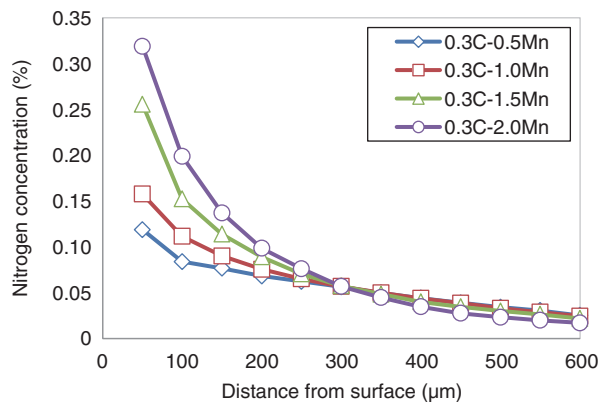


Fig. 13 Nitrogen concentration after GSN

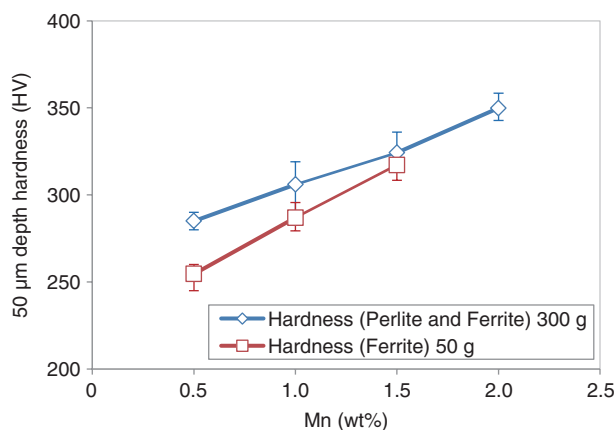


Fig. 14 50 μm depth hardness after GSN

was cut from the surface to the 50 μm depth hardness to allow observation of the ferrite phase directly below the compound layer. Figure 15(a) shows a bright-field image of the ferrite phase following GSN, while Fig. 15(b) shows the results of an element mapping analysis using scanning TEM energy-dispersive X-ray spectroscopy (STEM-EDS). Figure 15(b) shows that Mn is concentrated at the points at which nano-order acicular precipitates were observed in Fig. 15(a), indicating the formation of Mn precipitates. This demonstrates that precipitates are formed by the basic element Mn.

6.4. Strengthening Mechanism by Precipitates

6.4.1. Precipitate structure

The structure of the precipitates shown in Fig. 15 was subjected to a detailed analysis. Figure 16 shows a TEM image and an electron diffraction pattern for the 0.3C-1.0Mn steel. In Fig. 16(a), precipitates ranging in length from 10 nm to 50 nm in a mutually orthogonal relationship can be seen in the field of observation. The diffraction pattern in Fig. 16(b) shows spot groups of iron, and groups of streak-shaped spots extending from the precipitates. Because the lattice formed by the precipitate spots displays a good correlation with the $\eta\text{-Mn}_3\text{N}_2$ crystal zone axis $[1\bar{1}0]_\eta$, the precipitates were identified as $\eta\text{-Mn}_3\text{N}_2$.

Figure 17 shows schematics of the mother phase Fe ($\alpha\text{-Fe}$) and $\eta\text{-Mn}_3\text{N}_2$ unit cells. $\alpha\text{-Fe}$ is a cubic crystal with lattice constants of $a=b=c=2.87\text{\AA}$. $\eta\text{-Mn}_3\text{N}_2$ is a tetragonal crystal with lattice constants of $a=b=4.20\text{\AA}$ and $c=12.03\text{\AA}$.

6.4.2. Precipitate morphology

The crystal zone axis shown in Fig. 16(b) is $[001]_\alpha // [1\bar{1}0]_\eta$, and the relationship $[020]_\alpha // [220]_\eta$ can be obtained

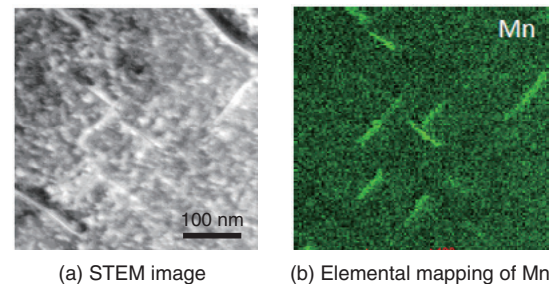


Fig. 15 STEM-EDS micrograph

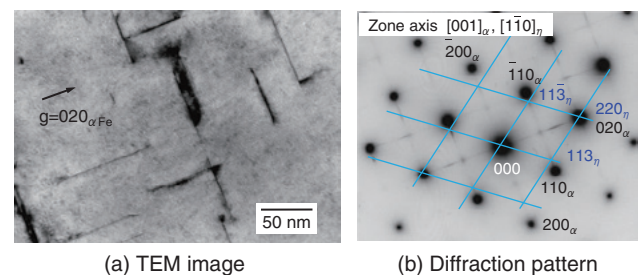


Fig. 16 Structure analysis of precipitation

from the diffraction pattern. In other words, Fe and η - Mn_3N_2 have a Baker-Nutting orientation relationship (B-N relationship). Figure 18 shows a schematic of the precipitate interfaces in the B-N orientation relationship. Figure 18(a) shows α -Fe on (001), while Fig. 18(b) shows η - Mn_3N_2 on (001). The precipitation morphology shown in Fig. 18(c) is a 45° rotation of the η - Mn_3N_2 on (001) with $[001]_\alpha$ and $[001]_\eta$ as the axes to produce $[001]_\alpha // [1\bar{1}0]_\eta$ and $[020]_\alpha // [220]_\eta$, against 2x2 α -Fe unit cells on (001). There is good overlap between Fe and Mn atoms. The misfit between the interatomic distances of the α -Fe atoms and the Mn atoms forming Mn_3N_2 precipitates is 3%, and consistency can therefore be considered good.

With the super-saturation of solid solution nitrogen

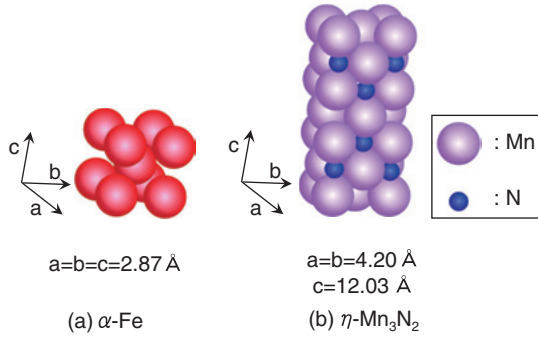


Fig. 17 Schematic of unit cell

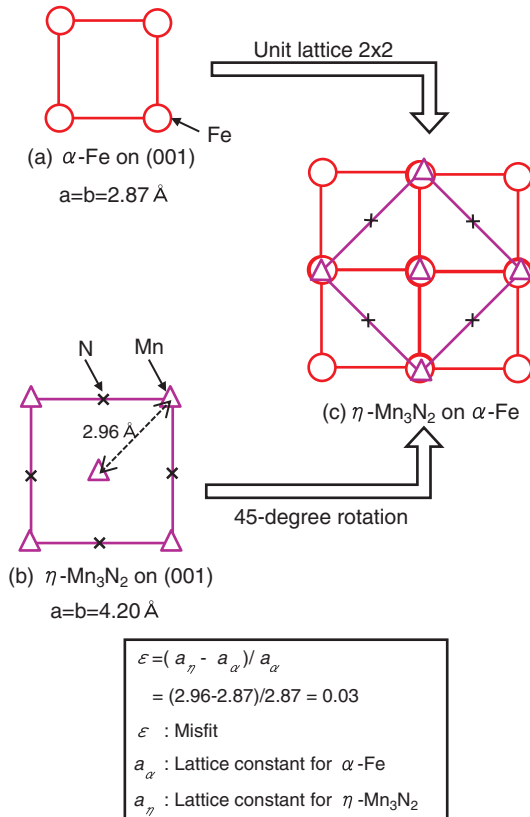


Fig. 18 Baker-Nutting orientation relationship between Fe and Mn_3N_2

as the driving force, the precipitates grow in such a way as to control the interface energy to a low level. Because of this, the growth of the precipitates increases the area of the $(001)_\eta$ plane (plane with low interface energy, perpendicular to the direction of the c axis), on which the Mn_3N_2 displays good consistency with the Fe atoms. In the case of growth in the direction of the C axis, it is believed that the precipitates double in thickness (to the equivalent of two unit cells) and form steps, at least in sections. It is conjectured that for plane other than $(001)_\eta$, because consistency with the Fe atoms is low (interface energy is high), the precipitates tend not to form steps, and precipitate growth in the direction of the c axis tends not to occur. It is therefore considered that because the Mn_3N_2 precipitates grow in this way in the directions of the a and b axes with one unit cell as the basic element, the precipitates are formed disk-shape in three dimensions.

Figure 19 shows a high-resolution TEM image of η - Mn_3N_2 precipitates. The precipitates marked by the arrows in Fig. 19(a) precipitated onto $(100)_\alpha$ and $(010)_\alpha$ in

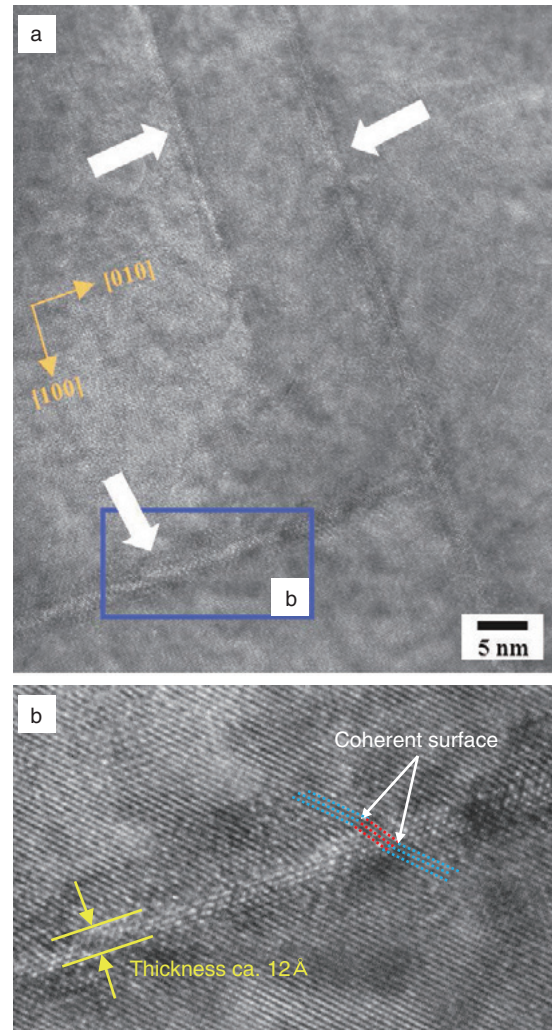


Fig. 19 High-resolution TEM image of Mn_3N_2

the ferrite phase. Figure 19(b) shows a magnified image of $\eta\text{-Mn}_3\text{N}_2$ that has precipitated onto a $(100)_\alpha$ (this is the area surrounded by the square in Fig. 19(a)). The thickness of this precipitate is $12\text{\AA}$, which corresponds well with the length of the $\eta\text{-Mn}_3\text{N}_2$ in the direction of the c axis ($12.03\text{\AA}$). This demonstrates that the observed precipitates grow anisotropically to specific planes, and the precipitate shown in Fig. 19(b) grows continuously (coherently) with the atomic arrangement of the mother phase. The strengthening mechanism of the developed steel by means of GSN is therefore conjectured to be strengthening through the formation of precipitates with a high degree of coherency in a B-N relationship (Fig. 18), which results in the formation of strain fields, increasing deformation resistance.

6.4.3. Precipitation behavior of precipitates

Figure 20 shows a schematic of the precipitation behavior of Mn_3N_2 .

In Stage I, Mn exists as a solid solution in the ferrite phase (prior to GSN).

In Stage II, the application of GSN results in the diffusion of nitrogen in the ferrite phase, which forms a solid solution.

In Stage III, time of exposure to GSN has increased, resulting in increased nitrogen diffusion. In this state the precipitates display coherence with the mother phase, and this is believed to increase strength.

Stage IV represents the state of the precipitates in the ferrite phase when heat treatment for long periods or at excessively high temperatures has resulted in over-aging. In this case, the Mn_3N_2 precipitates become coarse. It is believed that some of the coarse precipitates become non-coherent, and their ability to strengthen the material therefore declines.

The results discussed above demonstrate that by contrast

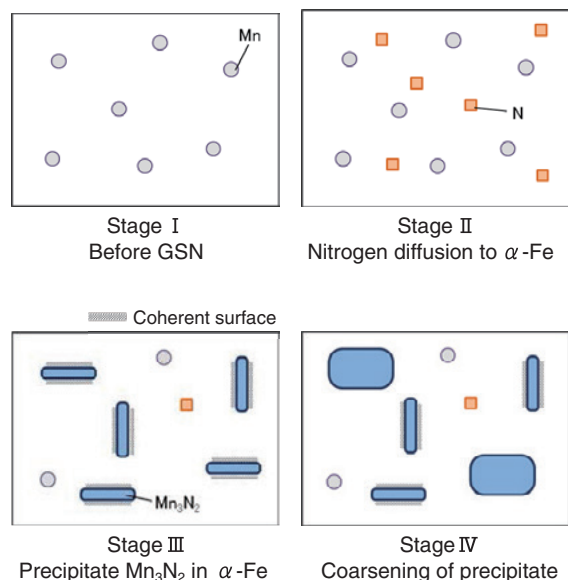


Fig. 20 Schematic of precipitate behavior

with the action of Mo in strengthening the conventional high-strength steel by clustering or concentrating the nitrogen surrounding it, the strengthening mechanism in the developed high-strength steel is the microscopic precipitation of $\eta\text{-Mn}_3\text{N}_2$ in the ferrite phase. In addition, it is conjectured that the precipitates formed in the developed high-strength steel are effective in strengthening the material because they display coherence with the mother phase, as shown in Stage III in Fig. 20.

7. Conclusion

A high-strength blast furnace steel for crankshafts that eliminates the use of rare metals has been developed by optimizing the amount of Mn, a basic element composing steel. The developed high-strength steel is strengthened by the precipitation of $\eta\text{-Mn}_3\text{N}_2$, and by this means realizes equivalent strength to the conventional high-strength steel. Eliminating the addition of rare metals to the steel has reduced procurement risk and material costs. The development process produced the following results:

- (1) It was possible to realize an equivalent level of fatigue strength to the conventional high-strength steel containing the rare metal Mo by increasing Mn content to 0.9 wt% in 0.30-0.35 wt%C carbon steels.
- (2) The range of $50\text{ }\mu\text{m}$ depth hardness that made it possible to reconcile fatigue strength and straightenability was set. Taking into consideration the effect of each constituent element on $50\text{ }\mu\text{m}$ depth hardness, the equivalent amount of Mn (M_{neq}), based on the elements with the greatest effect on hardness (Mn, C, Cr), was introduced as a new indicator, and a range was set for this parameter.
- (3) The formation of Mn nitrides was observed in a 1.0wt% Mn steel that had been gas soft nitrided. These were identified as $\eta\text{-Mn}_3\text{N}_2$ from their electron diffraction pattern.
- (4) The strengthening mechanism with increased Mn is considered to be the precipitation strengthening of $\eta\text{-Mn}_3\text{N}_2$ with a B-N relationship in the ferrite phase.

The developed high-strength steel was first employed in the crankshaft for the 2013 model year CR-Z. The substitution of this crankshaft for a conventional standard steel crankshaft realized a 4 kW increase in power. Crankshafts produced using the developed steel will be employed successively in new gasoline engines.

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