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Method of Prior Austenite Grain Refining Using Induction Hardening†

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Abstract:

A technique for obtaining ultra-fine prior austenite grain in the high frequency induction quenched portion for automotive parts was developed for increasing strength. The prior austenite grain size greatly depended on the maximum heating temperature of the induction quenching and the addition of Mo. Choosing maximum heating temperature of above Ac3 and adding up to 0.4 mass% of Mo make the prior austenite grain remarkably fine. The prior austenite grain size obtained was about 3 μm. The tensile properties of the induction quenched portion showed the tensile strength of more than 2 000 MPa and the elongation of more than 10%. The strength/ductility balance of the high frequency induction quenched portion is superior than that of conventional quenched and tempered steel.

1. Introduction

Global warming is now recognized as a serious social problem. The causes of this phenomenon are not limited to emissions from factories; CO_2 emissions from automobiles are also one major factor. In responding to this issue, improvement of automobile fuel consumption by weight reduction is extremely important, and unit sales of high fuel economy vehicles are increasing[1)].

Reduction of the weight of automobile drive system parts, which is termed "unsprung weight," is considered an efficient approach to improving fuel economy and thereby reducing CO_2 emissions by automobiles. "Unsprung weight" is the total of the weights of the constant velocity joint, drive shaft, hub, propeller shaft, differential gear, and related parts. The essential mechanical properties required in these parts are static torsional strength, torsional fatigue strength, rotating bending fatigue strength, and thrust rolling contact fatigue strength.

This paper reports on the development[2)] of a technique for obtaining an ultra-fine prior austenite grain size (hereinafter, prior γ grain size) in the induction quenched portion using a technique which is applicable to industrial operations, with the aim of securing high strength and high fatigue strength in automobile drive train parts.

2. Necessity of Ultra-fine Grain Microstructure

Techniques for achieving high strength in induction quenched portions of automobile parts include increasing the carbon content or alloy content of the material. However, these methods have the demerit of reducing elongation, reduction of area, and the impact value. Furthermore, in many cases high strength does not contribute to high fatigue strength. **Figure 1** shows the effect of relative hardness on the torsional fatigue strength of carbon steel. Up to a relative hardness of around Hv650, torsional fatigue strength increases accompanying increases in hardness. However, when the relative hardness exceeds Hv700, no increase in fatigue strength is observed. At this hardness level, the fracture mode shifts from intragranular ductile fracture to intergranular fracture of prior austenite. This shift to intergranular fracture is considered to be the main reason why fatigue strength does not increase in spite of higher strength.

Intergranular fracture occurs in cases where the bond strength at the grain boundaries is weaker than the intragranular fracture strength. Takagi compiled calculated

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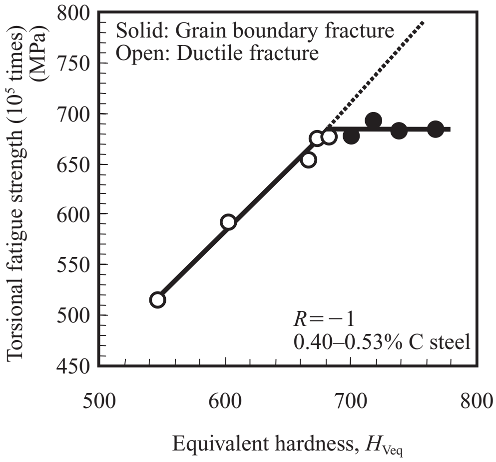

$H_{Veq}=\frac{1}{a^3}\int_0^a H(r)r^2\mathrm{d}r$ a: Radius of parallel portion, $H(r)$: Hardness, r: Distance from center[3)]

Fig. 1 Relation between torsional fatigue strength and equivalent hardness of 0.40–0.53% C steels

results showing that reduction of stress concentration per unit of area and reduction of the concentration of impurities at grain boundaries by grain refinement increase grain boundary (intergranular) strength[4)]. In other words, intergranular fracture can be suppressed by grain refinement.

In automobile suspension and drive train parts, the portions which are subjected to the highest loads are strengthened by performing quenching and loads tempering (hereinafter, QT) treatment. Much research on refinement of the ferrite grains[5)] of the base material has been done in the past with the aim of achieving high strength and high toughness. However, when using QT materials, refinement of the prior γ grains in the quenched portion, and not refinement of ferrite grains in the base material, is critical for application to suspension and drive train parts.

3. Microstructure Refinement Techniques

3.1 Study of Elements

In order to identify elements which are effective for refining prior γ grains, steel ingots for research purpose were prepared by adding various elements to JIS S48C (JIS: Japanese Industrial Standards) steel as the base steel as test materials for induction quenching experiments. Addition was set at the amount of each element which bonds with a constant amount of carbon. To eliminate the effect of the prior microstructure, specimens were heated to 1 000°C and quenched in the first stage, followed by induction quenching in the second stage at specified heating temperatures. Prior γ grains in the quenched portion were measured by an intercept method. In all cases, the prior γ grains were revealed using a dedicated etchant (Gamma R etchant) [6,7)] which

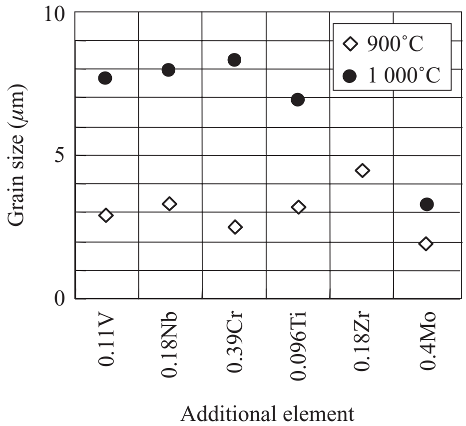

Fig. 2 Effect of additional element on prior austenite grain size

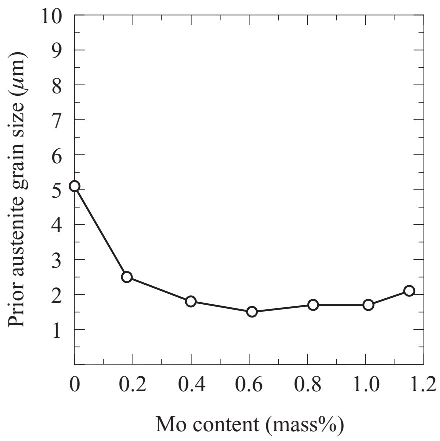

Fig. 3 Effect of molybdenum content on prior austenite grain size

was developed by the authors for use in revealing ultrafine γ grain boundaries. As shown in **Fig. 2**, Mo was found to have the largest prior γ grain refining effect among the elements studied. Next, induction quenched specimens were prepared in the same manner, using ingots with various Mo contents up to 1.2%. Induction quenching was performed at 1 000°C in the first pass and 900°C in the second pass, and the prior γ grain size of the quenched portion was measured. The results are shown in **Fig. 3**. The grain refinement effect was substantially saturated at Mo addition of 0.4 mass% or more.

3.2 Study of Induction Heating Conditions

The effect of induction quenching conditions was studied using 0.4 mass% Mo steel. As induction quenching conditions, experiments were performed at heating rates from 40 to 1 000°C/s, maximum heating temperatures from 850 to 1 000°C, and time from heating to quenching in water varied between 0 and 1.5 s.

As shown in **Fig. 4**, within the range of conditions in these experiments, the effects of the heating rate and the time from heating to quenching on the prior γ grain size were small, and the results were basically determined by

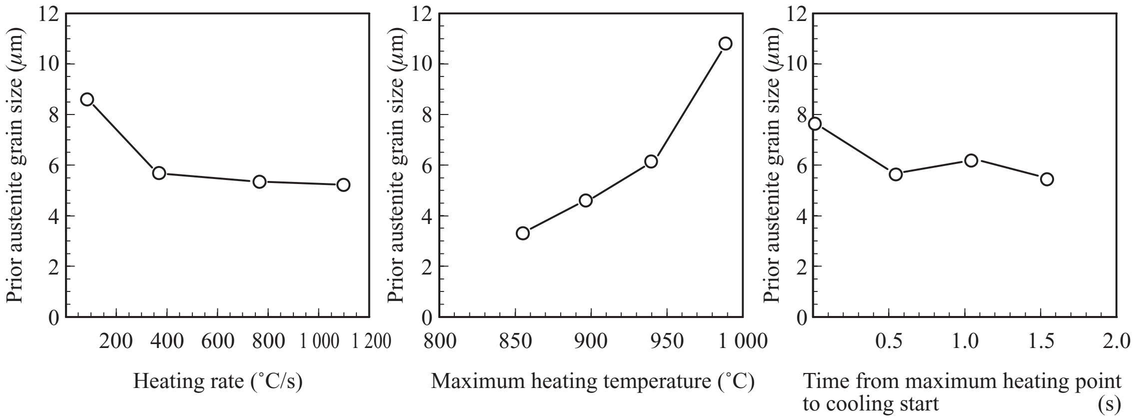

Fig. 4 Effect of heating rate, maximum heating temperature, and time from maximum heating point to cooling start on prior austenite grain size

the maximum heating temperature.

4. Pursuit of Grain Refinement

In the following, "developed steel" refers to steel with refined prior γ grain microstructure obtained by a combination of Mo addition and low temperature induction heating. The chemical composition of the developed steel is shown in **Table 1**. For comparison purposes, a JIS S53C equivalent steel (hereinafter, S53C) was used. The developed steel was produced using ingots for research purpose by hot forging to 60 mm in diameter after holding at 1 200°C × 1h, followed by normalizing in air at 850°C × 1 h. The comparison steel, S53C, was a standard mass-production material produced by commercial steelmaking and rolling processes. Micro tensile test pieces were taken from the axial direction (L direction) of the obtained bars at the 1/4 position in the diameter (hereinafter, *D*/4 position), and induction quenching was performed using a maximum heating temperature of 1 050°C in the first pass and various maximum heating temperatures from 850 to 1 150°C in the second pass, followed by tempering at 170°C × 30 min. Microstructures were obtained by cutting the parallel part and etching with Gamma R.

As representative examples of the microstructures, the developed steel with a carbon content of 0.53% and the comparison steel, S53C, are shown in **Photo 1**. When the maximum heating temperature in the second pass is set at 950°C, the prior γ grain size of the developed steel is extremely fine, at 2.9 μm, in comparison with the grain size of 12.4 μm of the S53C steel. Furthermore, as mentioned previously, it can be understood that the prior γ grain size becomes smaller with decreases in the maximum heating temperature in the second pass of induction quenching.

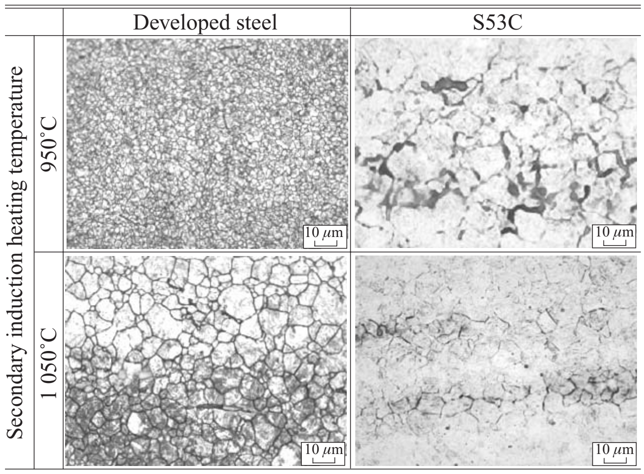

Photo 1 Prior austenite microstructure of induction heated portion(Gamma R echant; First induction heating temperature is 1 050˚C)

Next, the following experiment was performed to determine the ultimate limit of refinement of prior γ grains. After heating the material to 1 100°C, plate rolling was performed to a thickness of 35 mm. Next, the material was cold-rolled to a thickness of 16.5 mm. Specimens for use in induction quenching were taken from this material, and induction quenching was performed at 1 100°C in the first pass and 870°C in the second pass, followed by tempering at 170°C × 30 min. The specimens were cut, and the microstructure was revealed using the Gamma R etchant. **Photo 2** shows

Table 1 Chemical composition of steels

(mass%; B, N, O: ppm)

Steel	C	Si	Mn	P	S	Al	Mo	Ti	B	N	O
Developed steel	0.48	0.74	0.61	0.013	0.015	0.027	0.40	0.025	24	42	10
S53C	0.53	0.21	0.87	0.015	0.006	0.027	Cr 0.15		—	52	10

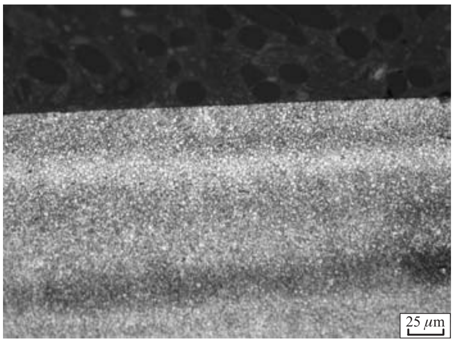

Photo 2 Prior austenite microstructure of the steel after cold rolling, induction heating, tempering

the homogeneous prior γ grain structure with an average prior γ grain diameter of 1.5 μm in quenching the induction quenched portion obtained in this experiment.

5. Basic Properties of Developed Steel

5.1 Tensile Properties

Figure 5 shows the nominal stress-nominal strain curve (hereinafter, SS curve) of the developed steel, together with a scanning electron microscope (SEM) image of the fracture surface. The SS curve is a round type, and shows tensile strength exceeding 2 000 MPa and total elongation of more than 10%. A ductile fracture surface was observed. The relationships between the uniform elongation and yield stress (YS) of the developed steel and conventional quench-tempered steels (QT steels) are shown in **Fig. 6**. With the conventional QT steels, uniform elongation decreases to around 5% when YS exceeds 600 MPa. In contrast to this, the developed steel shows uniform elongation of nearly 5% even when YS exceeds 1 700 MPa.

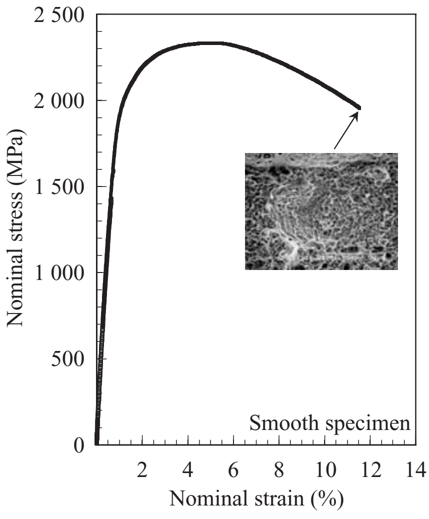

Fig. 5 SS curve and fracture surface of developed steel

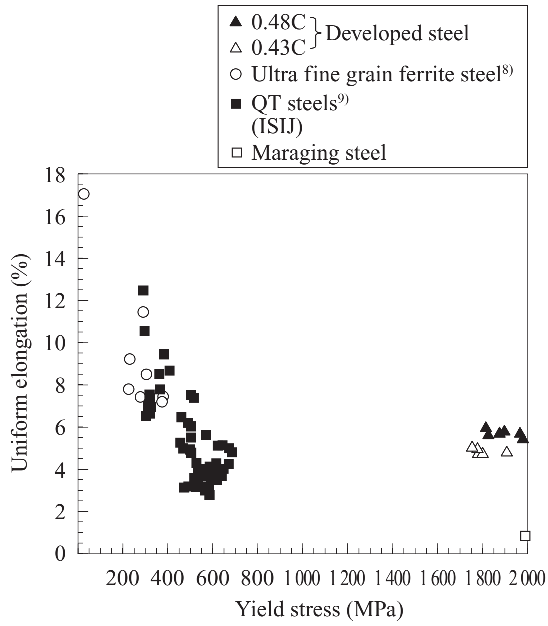

Fig. 6 Relation between uniform elongation and yield stress

5.2 Rotating Bending Fatigue Properties

Rotating bending fatigue tests were performed with the developed steel and S53C steel, respectively, using specimens with a parallel part diameter of 4 mm taken from bars in the L direction at the D/4 position. One pass induction quenching at 1 020°C was performed with both the developed steel and the S53C steel. Complete quenching to the core of the material was confirmed with both the developed steel and the S53C steel. Following this, tempering was performed in an oil bath at 170°C × 30 min. Test specimens were prepared by mechanically polishing the parallel part of the obtained samples. Tests were performed using an Ono rotating bending test machine.

The results are shown in **Fig. 7**. On the high stress side ($<1.0 \times 10^5$ cycles), the rotating bending fatigue strength of the developed steel exceeded that of the S53C steel by more than 20%. However, near the fatigue limit, the fatigue strength of the two steels became virtually identical. The residual stress and cross section hardness distribution of the specimens before the test are shown in **Table 2** and **Fig. 8**, respectively. There was no significant difference in either the residual stress or the hardness of the developed steel and the S53C steel. However, the prior γ grain sizes of the two steels were 2.1 μm and 15.4 μm, respectively. **Photo 3** shows images of the fracture start point in rotating bending fatigue specimens of the developed steel and S53C steel under applied stresses of approximately 800 MPa and approximately 1300 MPa. Under the applied stress of approximately 800 MPa, the fracture start point was an inclusion-type origin (hereinafter, “fish eye”) in both steels, whereas, at approximately 1 300 MPa, both steels

Symbol	Steel	Grain size (ím)	Induction heating temp. (°C)
○	Developed steel	2.1	1020–867
△	S53C	15.4	1 020

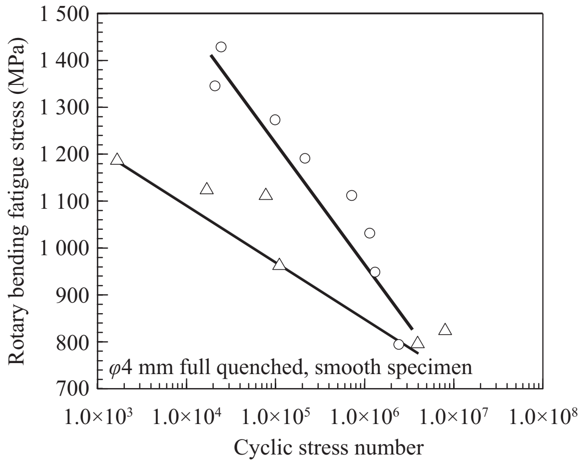

Fig. 7 Comparison of fatigue strength between developed steel and S53C

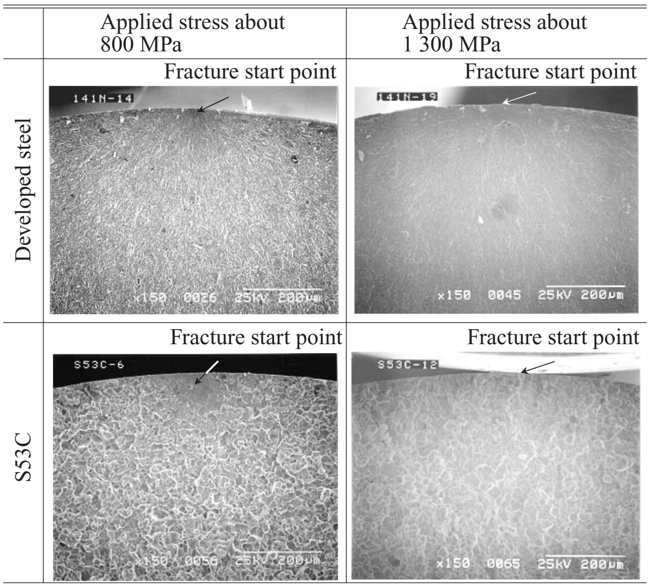

Photo 3 Fracture start point of rotary bending fatigue specimen

Table 2 Residual stress of rotary bending fatigue specimen

Steel	Residual stress (MPa)	
	L direction	C direction
Developed steel	–439	–123
S53C	–482	–89

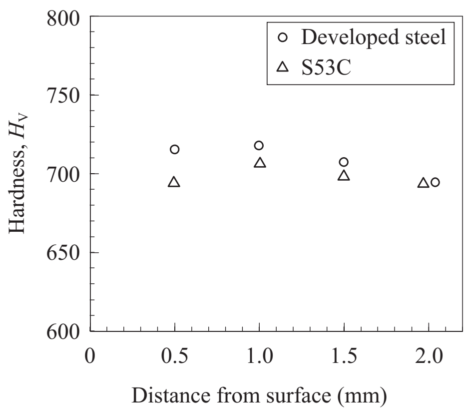

Fig. 8 Hardness distribution of rotary bending fatigue specimen

displayed a surface fracture origin. Moreover, in the S53C steel, intergranular fracture occurred in parts other than the fish eye.

Based on the above, it was understood that the prior γ grain size of the developed steel is fine in comparison with that of the S53C steel, and the developed steel is resistant to intergranular fracture. From this, it is considered that the developed steel exhibits higher fatigue strength than the S53C steel on the high stress side, and refinement of the prior γ grains contributes to improvement of fatigue strength. However, the fatigue limits of the two steels were virtually identical. Where this is concerned, based on the fact that the fracture origins were fish eyes, it can be inferred that the effect of inclusions on the materials was rather stronger than that of intergranular fracture. Accordingly, as a next stage, improvement of the cleanliness of the steel is considered essential for achieving further increases in fatigue strength.

6. Mechanism of Microstructure Refinement

It can be understood that, even when the effect of the prior microstructure is eliminated, the achieved prior γ grain sizes of the developed steel and S53C steel are different, and it is difficult to obtain an ultra-fine grain size without addition of Mo. Therefore, the following will consider the effect of Mo. The conceivable effects of the prior γ grain boundary which suppress grain growth are the pinning effect of the prior γ grain boundary by precipitates[10], and the drag effect due to fixation of elements on the prior γ grain boundaries by solid-solution solute elements[11]. **Figure 9** shows an example of transmission electron microscope (TEM) observation of the induction quenched portion of the developed steel. Although composite precipitates of Mo and Ti can be observed, preferential precipitation to the grain boundary was not detected. An evaluation of the precipitation ratio by analysis of the residue of electrolytic extraction using high brightness synchrotron radiation showed that the amount of Mo precipitation was 30% or less, and the changes in the amount of precipitation before and after induction quenching were slight. It is estimated that much of the Mo exists in a solid solution state. However, because segregation to the grain boundary cannot be detected, as shown in **Fig. 10**, no data positively support the drag effect. Based on the above, the authors propose a mechanism of grain grown suppression through

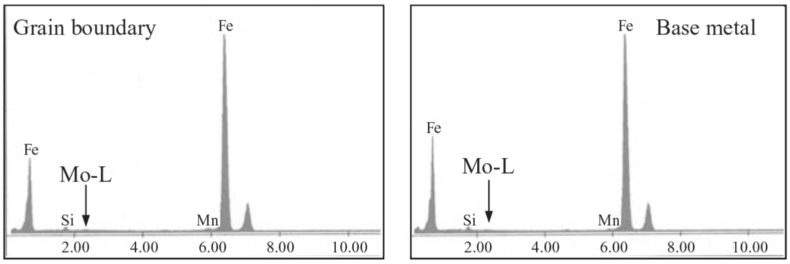

Fig. 10 Result of point analysis using energy dispersion X-ray spectroscopy

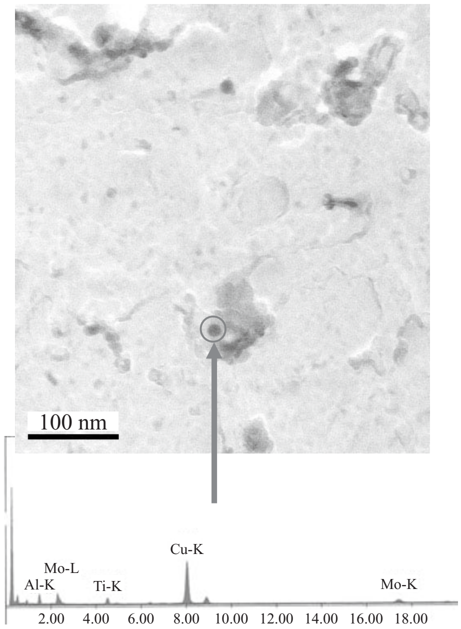

Fig. 9 Transmission electron microscopic photo of molybdenum precipitation and energy dispersion X-ray spectrum from precipitation indicated by arrow

interaction with carbon as the effect of the solid solution Mo. Although Mo interacts with C in austenite, its bonding force is smaller than that of strong carbide forming elements such as Ti, Nb, etc. Because this Mo addition makes the largest contribution to grain refinement, it is considered that grain grown during short-time heating, like that in induction quenching, contributes through suppression of the diffusion of C.

7. Conclusion

In order to reduce emissions of global warming gases and conserve resources, a grain refinement technique which makes it possible to obtain high strength in automobile suspension and drive train parts was developed. Steel produced using this technology is protected under the registered trademark "Fine *γ*."[12] "Fine *γ*" is a steel in which an ultra-fine prior γ grain size is secured in the quenched portion by optimization of the induction quenching conditions and Mo addition, and can be manufactured in industrial operations. "Fine *γ*" displays tensile strength exceeding 2000 MPa and total elongation of more than 10%, and shows an excellent strength-ductility balance in comparison with conventional quenched and tempered steels.

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