

# Development of Application Technology for Vacuum Carburizing

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## Abstract

*Contrary to benefits of vacuum carburization, such as less carbon dioxide emission due to higher processing temperature and efficiency, none of the control system for gas carburizing was adapted to vacuum carburizing. To control carbon concentration profile, it was suitable for calculating the carbon flux from the gradient of chemical potential. Excess carburizing which occurs at the edge of machine parts decrease the fatigue property due to increase of the residual austenite and formation of coarse carbides. For the remedy of this disadvantage, new vacuum carburizing process was developed. We confirmed the fine carbide dispersed in the case the carburizing followed by gas cooling, reheating and oil quenching is carried out at high temperature. Edge shape parts carburized by such process performed high fatigue property.*

## 1. Introduction

Transmission parts such as gears and CVT pulleys require high performance regarding the wear resistance and strength for contact fatigue and bending fatigue, and therefore generally undergo carburizing and quenching today. The surface treatment method that previously prevailed was pack carburizing, a treatment that involves heating steel immersed in charcoal powder to a high temperature. It forced workers to work in a hazardous environment, and was not even productive. After the 1950s, gas carburization in which steel is carburized in an accurately controlled CO atmosphere spread, and it still prevails. From the recent perspective of environmentally-conscious manufacturing, however, the carburization treatment is also being advocated to reduce the CO<sub>2</sub> emission, with vacuum carburizing attracting attention for its advantageous efficiency.

Vacuum carburizing is a treatment that consists of at least one cycle each of the carburization period during which steel parts are heated under vacuum and a hydrocarbon gas is injected into the vacuum carburizing furnace at low pressure, and the diffusion period in which the parts are held with vacuum to diffuse hydrocarbon only inside the steel.<sup>1)</sup> Vacuum carburizing allows for high temperature carburization at 1000°C or more under which steel can be held in the furnace with a higher diffusion coefficient than that when gas carburizing with the upper limit of approx. 950°C is used. Furthermore, since hydrocarbon gases are thermally unstable, treatments need to be performed at low pressure in order to prevent soot and tar from forming, which assists the efficient entry of carbon into steel.

The carbon concentration near the surface often exceeds the solid solubility limit (hereinafter referred to as the “A<sub>cm</sub> line”) and cementite (hereinafter referred to as “ $\theta$ ”) precipitates depending on the steel composition and temperature. This causes the carbon concentration gradient in austenite (hereinafter referred to as “ $\gamma$ ”) to grow very large, increasing the diffusion flux in turn, allowing for deep carburization for a short time.<sup>2)</sup> Moreover, since no oxide content is contained in the atmosphere, treated products have no incompletely-quenched layer in the surface layer, meaning that higher strength than that obtained through gas carburizing can be achieved using vacuum carburizing.

There is, however, a problem in vacuum carburizing associated with the excessive carburization at the edges of the treated products. The excessive carburization causes coarse  $\theta$  to be formed at the grain boundaries, which reduces the strength. A strength reduction also occurs due to an increase of residual  $\gamma$  with excessive carburization, even without cementite precipitation.<sup>3)</sup> In other words, even if incompletely-quenched layers are eliminated, poor edge strength makes the durability inferior to that required of an automobile part. Given this, we started to consider a method to render the excessive carburization at edges harmless rather than preventing it. This paper describes a method to eliminate the adverse effects of the excessive carburization by having fine  $\theta$  precipitate in the excessively-carburized portion.

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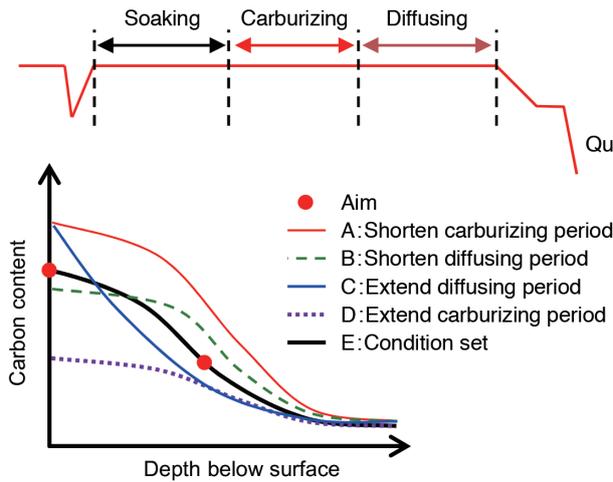
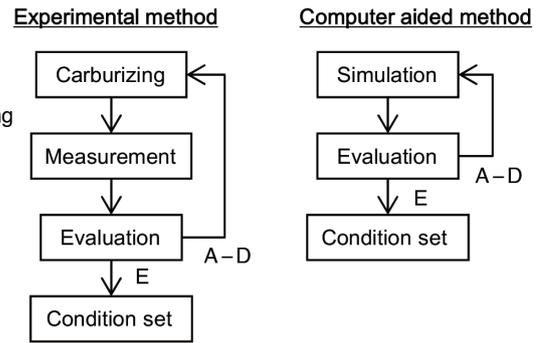


Fig. 1 Setting method for vacuum carburizing condition



## 2. Carbon Concentration Control Method

Since fatigue strength and wear resistance against bending and torsion are expected for most carburized automobile parts, it is necessary to harden steel to a specified depth and to have an appropriate surface carbon concentration. For this reason, a method to optimize the carburization conditions for each part type and to accurately control the carbon concentration is required.

According to Morita, et al.,<sup>4)</sup> the carbon concentration is increased in the carburization period of vacuum carburizing until the surface enters an equilibrium state with the graphite. During a subsequent diffusion period in which the carbon supply from above the surface is stopped and carbon diffuses in the depth direction, the carbon concentration at the surface is declined. Thus, the carbon concentration is controlled by adjusting the durations of the carburization period and diffusion period, while each part type has its own requisite carburization depth.

Figure 1 shows schematic views illustrating the concept of carburization condition adjustment. First, the target carbon concentration is determined for the surface and desired depth. Then, the carbon concentration is examined using the heat patterns of a certain carburization period and diffusion period, and the durations of the carburization period and diffusion period are adjusted based on the difference from the target. If the carbon concentration is high at the surface and desired depth, the carburization period is shortened; if it is low, the carburization period is prolonged. If the differences between the carbon concentration level and target are not the same at the surface and desired depth, the differences are adjusted by changing the diffusion period duration. When the diffusion period is prolonged, the carbon concentration at the surface is decreased. When it is shortened, the carbon concentration at the surface is increased. Since the change in the carbon concentration at the desired depth varies depending on how deep the desired depth is, it is necessary to change the two process parameters in a repetitive manner and evaluate them each time, in order to set optimum carburization conditions. If trials of optimum carburization conditions are conducted by repeatedly performing carburizing and analysis, enormous effort and time are required. In view of this, optimum carburization conditions need to be examined in simulations.

As described before, during vacuum carburizing,  $\theta$  precipitates in the surface layer and alloy elements are distributed to  $\theta$  and  $\gamma$ . The diffusion of carbon in the depth direction in  $\gamma$  and  $\theta$ , which

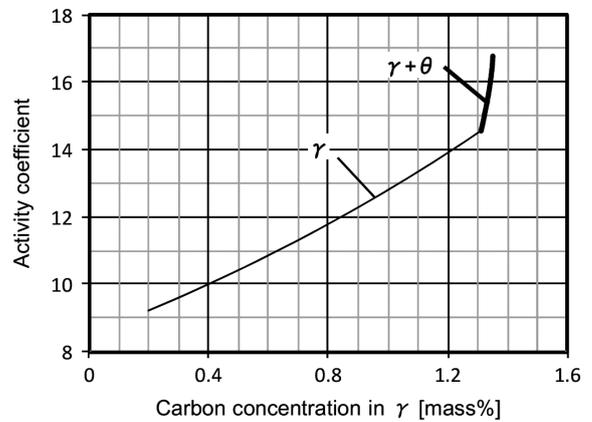


Fig. 2 Activity coefficient of carbon referred to graphite in carburized steel SCR420 at 1223 K

starts from  $\gamma$ , is strongly influenced by the alloy elements contained in  $\gamma$ . For example, Si is not dissolved in  $\theta$ , and Cr incrases in  $\theta$ .<sup>5)</sup> Since Si increases the carbon activity while Cr decreases the same, the amount of  $\theta$  precipitation is increased to distribute alloy elements in  $\theta$  and  $\gamma$ , and the carbon activity is increased. Figure 2 shows the carbon activity coefficient when SCR420 is carburized at 950°C. Thermodynamic calculation software Pandat 8.2 and the PanFe 2014 database were used.<sup>6,7)</sup> (Pandat was used for the thermodynamic calculation described hereinafter.) The two-phase region of  $\gamma$  and  $\theta$  has a larger activity coefficient gradient than the  $\gamma$  single-phase region. The diffusion flux is represented by the following equation (1),<sup>5)</sup> which includes a composition differential term of the activity coefficient,

$$J = -m \frac{\partial \mu}{\partial z} = -mRT \left\{ 1 + x \frac{\partial (\ln \gamma)}{\partial x} \right\} \frac{\partial x}{\partial z} \quad (1)$$

where  $m$  is the carbon mobility and  $x$  is carbon mole-concentration. For gas carburizing during which no precipitation due to carburization generally occurs or in a negligible amount, if any, the diffusion flux is calculated from the gradient of carbon concentration. In contrast, for vacuum carburizing with a large amount of  $\theta$  precipitation, the change of the activity coefficient is not negligible. From the above, for simulating vacuum carburizing, equation (1) is differentiated to be solved using the diffusion drive force as the gradient of

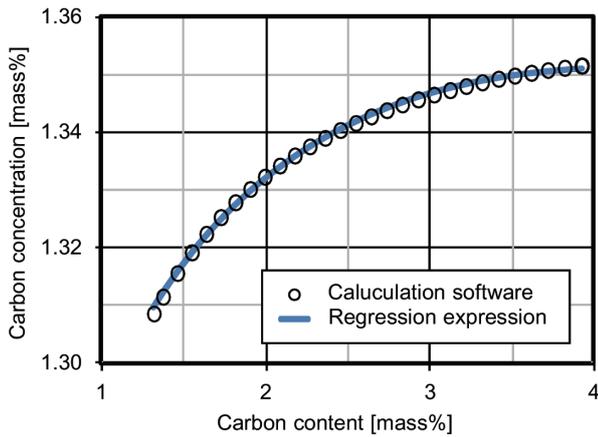


Fig. 3 Carbon concentration in  $\gamma$ -SCR420 at 1223 K

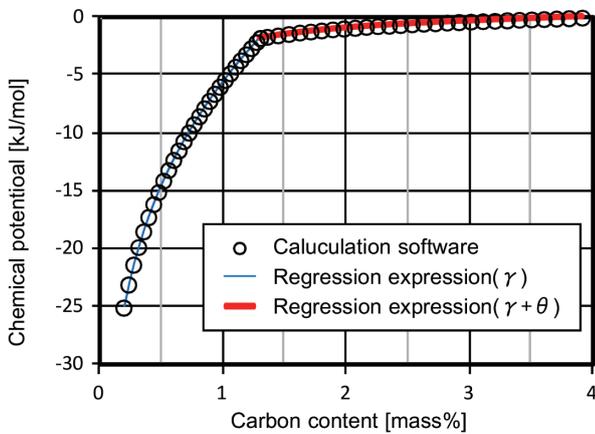


Fig. 4 Chemical potential of carbon referred to graphite in SCR420 at 1223 K

chemical potential  $\mu$ . Then, the carbon concentration distribution can be obtained under the condition that the law of mass conservation is satisfied.

As shown in Fig. 3 and Fig. 4, we obtained the equilibrium state for each diffusion calculation step using regression equations written with respect to the results obtained in advance by the thermodynamic calculation software. This eliminated the need for thermodynamic equilibrium calculation for calculating the diffusion, reducing the calculation workload.

Figure 5 shows the carbon concentration distribution in an SCR420 round bar 26 mm in diameter that underwent vacuum carburizing consisted of a one-hour carburization period, together with the simulation results. Figure 6 shows the carbon concentration distribution in the same material that underwent vacuum carburizing consisted of a one-hour carburization period and a one-hour diffusion period, also together with the simulation results. The carbon concentration in a chip that was obtained by machining 0.05 mm into the surface layer for each vacuum carburized bar was analyzed. In the case of the vacuum carburizing with a one-hour carburization period, the carbon concentration at 0.05 mm deep in the surface layer reached approx. 2.0 mass% with a two-phase ( $\gamma$ - $\theta$ ) structure formed; in the case of the vacuum carburizing with a one-hour carburization period and a one-hour diffusion period, the carbon concentration reached approx. 1.0 mass% with a single phase ( $\gamma$ ) struc-

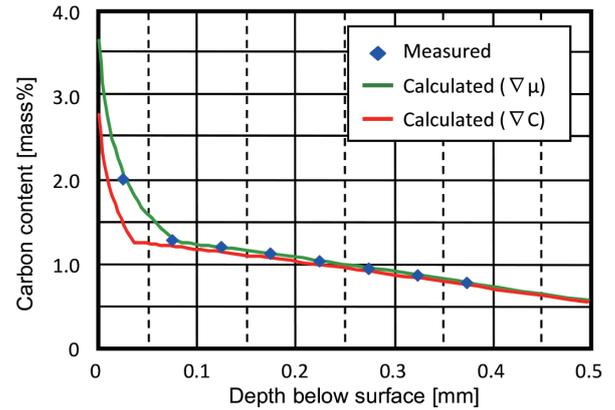


Fig. 5 Carbon profile of SCR420 carburized for 1 h at 1223 K

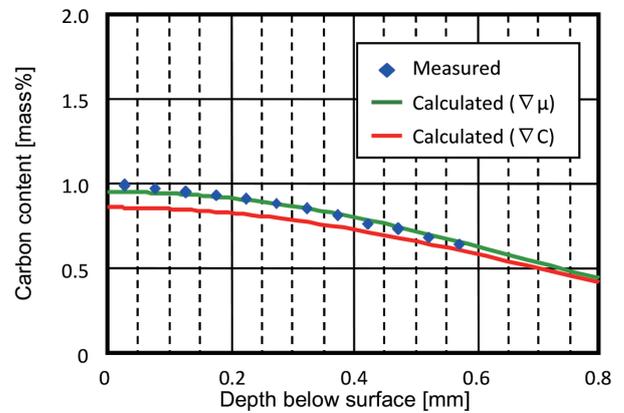


Fig. 6 Carbon profile of SCR420 diffused 1 h after 1 h carburizing at 1223 K

ture formed. In both carburization conditions, the results of calculation that derived the diffusion flux from the chemical potential gradient were closer to the measurement values. Since a difference was seen only on the extreme surface layer with the vacuum carburizing with a one-hour carburization period, the amount of  $\theta$  precipitation in the carburization period has a significant impact on the carbon concentration distribution after the diffusion. Accurate projection of the  $\theta$  precipitation quantity in the carburization period is considered important.

### 3. Elimination of the Adverse Effects of Excessive Carburization

#### 3.1 Control guidelines for excessively-carburized structures

Figure 7 shows the simulation results of a cross-section of 90° edge, using the method described above. The carburization conditions were: Temperature 950°C; a 28-minute carburization period; a 40-minute diffusion period; and cooling to 860°C at the rate of 0.05°C/s and quenching after 30-minute soaking. Whereas after the 28-minute carburization period, carbon uniformly penetrated from the surface, after the 40-minute diffusion period, and the carbon concentration was higher at the edge (upper left in the image) than on the flat portion surface, exceeding the  $A_{cm}$  line. Furthermore, while at the quenching temperature of 860°C, the carbon concentration at the flat portion was 0.8 mass%, it was approx. 1.8 mass% at the edge. The carbon concentration during vacuum carburizing increases to a point where it is in equilibrium with graphite in a carbu-

rization period, and then the carbon is diffused in the depth direction during a diffusion period, reducing the surface carbon concentration. However, at an edge, the diffusion fields collide with each other to hinder the carbon concentration from decreasing to a sufficient degree. This occurs when the surface carbon concentration in the carburization period was higher than the target carbon concentration. As long as processing is conducted at 800°C or more, excessive carburization at the edge is inevitable.

In view of this, we studied a method to eliminate the adverse effects of excessive carburization. It is considered that the carbon concentration at the edge exceeds the  $A_{cm}$  line after the diffusion, and coarse  $\theta$  precipitates, resulting in the deterioration of fatigue properties. Given this, we studied a method to suppress the deterioration of fatigue properties by distributing fine  $\theta$ . As a precipitate formed under high temperature tends to be coarse, it is desirable to form a  $\gamma$  single-phase structure under carburization temperatures.

Figure 8 shows a computational phase diagram of SCR420 using the carbon concentration as a variable. From the diagram, under high temperature, when the  $A_{cm}$  line is on the high carbon concentration side and the carbon concentration of an excessively-carburized portion exceeds 1.5 mass%, it is necessary to set the carburizing temperature to 1000°C or more to obtain a  $\gamma$  single-phase structure.

At the edge where excessive carburization occurred, a  $\gamma$  single-phase structure can be formed as well. However, if the material is hardened as-is, the high carbon concentration will cause a large amount of residual  $\gamma$  to be formed, rendering both hardness and strength low. For this reason, after the carburization and diffusion during which a  $\gamma$  single-phase structure is developed, the material is quickly cooled for suppressing the  $\theta$  precipitation to develop the pearlite transformation. Then, when the material is heated to the quenching temperature, in the excessively-carburized portion, ferrite ( $\alpha$ ) is transformed to  $\gamma$  while a part of  $\theta$  in pearlite is melted, and a two-phase ( $\gamma$ - $\theta$ ) structure is formed. After the material in this state is quenched, a hardened structure with diffused fine  $\theta$  can be obtained.

**3.2 Strength properties of a product that has undergone the measure for eliminating the excessive carburization adverse effects**

Next, we studied the fatigue strength of a material in which fine  $\theta$  was distributed at the excessive carburization portion using a batch-type vacuum carburizing furnace as well as acetylene as the carburization gas. Figure 9 shows the heat pattern. We conducted both general vacuum carburizing at 950°C and vacuum carburizing at 1040°C added with a process consisting of gas cooling, reheating to 860°C, and quenching for the purpose of eliminating the adverse

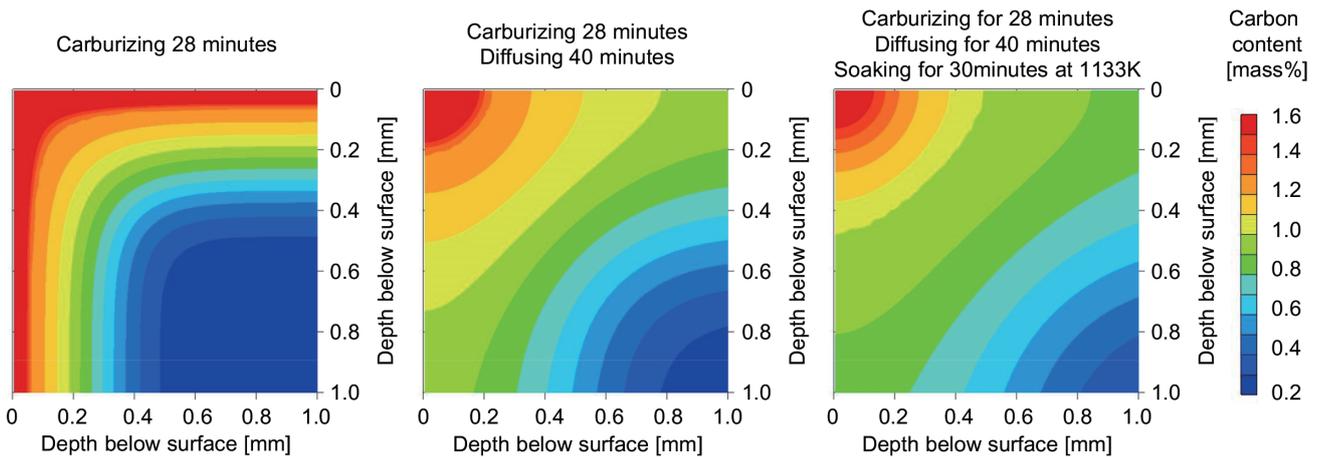


Fig. 7 Carbon distribution of carburized SCR420 at 1223 K and soaked at 1133 K

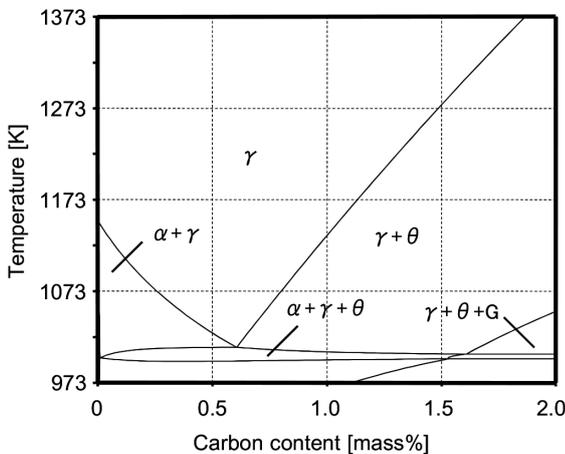


Fig. 8 Calculated phase diagram of SCR420

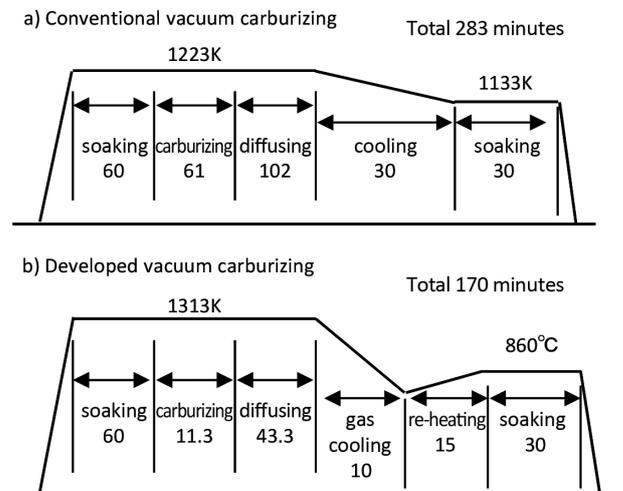


Fig. 9 Heat pattern of vacuum carburizing test (times in minute)

effects of excessive carburization. For the normal vacuum carburizing, a SCM420 sample was vacuum carburized at 950°C. For the vacuum carburizing with a process for eliminating the excessive carburization adverse effects, a sample of high chromium steel was vacuum carburized at 1040°C to form fine cementite. With the process for eliminating the excessive carburization adverse effects, which is conducted under high temperature, the total processing time is shortened by approx. 40%.

At 860°C, the samples were quenched in oil of 120°C at a pressure of 500 hPa, and then tempered at 170°C for two hours. For the strength evaluation, using a 13 mm × 13 mm prismatic column with a semicircular notch with a radius of 2 mm at its center as a specimen, the four-point bending fatigue test shown in Fig. 10 is conducted under the conditions of the controlled load, pulsating stress (stress ratio of 0.1), and test frequency of 10 Hz. Since the stress that is applied to the specimen in the direction along the 13-mm width is concentrated on the semicircular notch bottom of the specimen, the load is applied to both the flat portion and edge at the same time as with the case of the bottom of a real gear. Fracturing of the edge, which is not restrained in the width direction, is actually difficult compared with the flat portion. Therefore, as long as the strength of the edge is not notably decreased, the flat portion is considered to be the starting point of fatigue failure.

Figure 11 shows photographs of the edge microstructure as carburized by the above two methods, general carburizing and the new one with the process for eliminating the adverse effects of excessive carburization. In the image of a), which is the normally carburized sample, coarse  $\theta$  exceeding 10  $\mu\text{m}$  in length can be seen. In contrast, in b), which has undergone the new carburizing method, fine  $\theta$  can

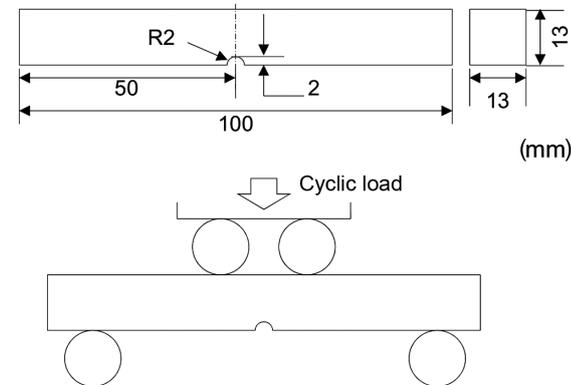


Fig. 10 Shape of notched specimen and schematic illustration of four point bending test

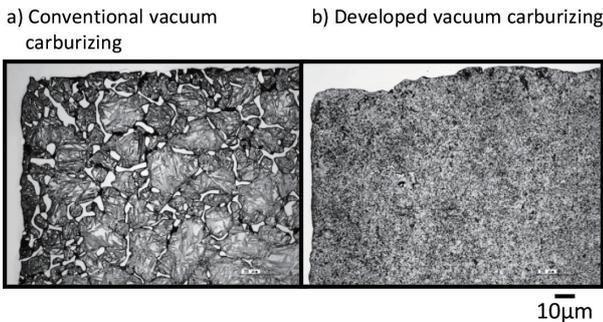


Fig. 11 Micrograph of super carburized edge

be found precipitating instead of coarse  $\theta$ .

Figure 12 shows the fatigue strength verified through four-point bending tests. Figure 13 shows representative example images of fatigued surfaces carburized by the above two methods. Figure 14 is a schematic view of the start of fatigue cracking. The strength of the sample that underwent the process for eliminating the excessive carburization adverse effects was higher than the normally vacuum carburized sample. Furthermore, whereas the normally vacuum carburized sample had fractured from the edge, the sample that underwent the process for eliminating the excessive carburization adverse effects had fractured from the flat portion. The development of fine  $\theta$  at the excessively-carburized portion leads to the fatigue strength improvement at the portion. For this reason, the fatigue cracking started from the flat portion.

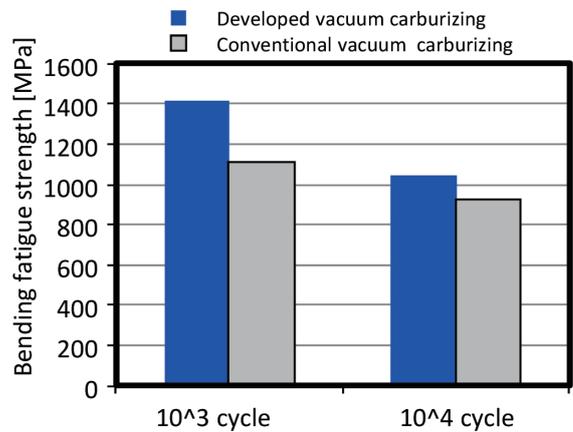


Fig. 12 Fatigue strength of vacuum carburized steel

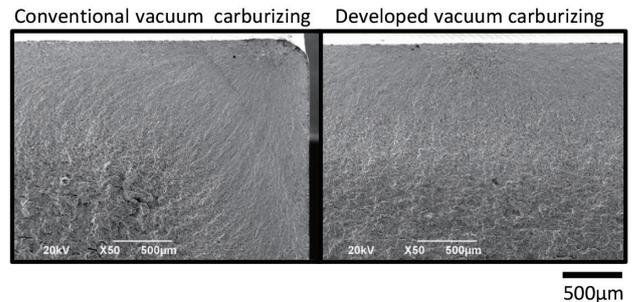


Fig. 13 Fatigue crack of vacuum carburized steel

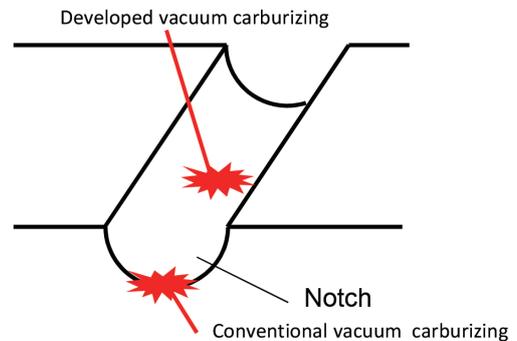


Fig. 14 Schematic illustration of crack starting area

#### 4. Conclusion

Vacuum carburizing, although capable of reducing the environmental load, has issues regarding the accurate prediction of carbon concentration distribution and the prevention of strength decrease due to excessive carburization. Against this backdrop, we studied a method to optimize the conventional treatment focusing attention on the steel properties.

##### (1) Control of the carbon concentration

For vacuum carburizing, there has not yet been a method to accurately predict the carbon concentration. To improve the simulation method, we took particular note of the fact that the amount of  $\theta$  that precipitates during the carburization period significantly influences the carbon concentration distribution after the diffusion period, and that whether  $\theta$  precipitates or not greatly changes the carbon activity. We handled the carbon diffusion drive force when  $\theta$  precipitates in a more rigorous manner, which led to the accuracy improvement of the obtained carbon concentration distribution. This allowed for determination of more accurate carburization conditions.

##### (2) Excessive carburization at the edge

Through simulations, we confirmed that the cause of the exces-

sive carburization that occurs at edges is the collision between diffusion fields. As long as processing is conducted at a temperature at which the  $A_{cm}$  line causes the surface carbon concentration to be higher than the target one, excessive carburization at the edge cannot be avoided. However, after carburization at high temperature, we found that a series of processes consisting of gas cooling, reheating, and quenching after carburization at high temperature effectively distributes fine  $\theta$ , which leads to the improvement of the bending fatigue strength.

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