Vacuum Carburizing of a Dual Phase Steel*

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Synopsis

A dual phase steel has been developed for which grain refining treatment subsequent to vacuum carburizing treatment at high temperature above 1 000 °C is not necessary. This 2 wt% Si steel, which contains 0.3 wt% V and 0.04 wt% Nb, has a fine structure in both the core and case, sufficient surface hardness and excellent mechanical properties equivalent to those of SCr 420 steel, even without grain refining treatment subsequent to vacuum carburizing at 1 050 °C.

I. Introduction

The power trains of vehicles and other machines for both industrial and agricultural use contain various structural machine parts such as gears, bearings and shafts. In general, carbon steels or alloy steels for machine structural use are employed as the material for such structural machine parts, and in many cases, machine parts fabricated by the applicable forming method are subjected to a surface hardening treatment such as gas carburizing.

Conventional gas carburizing treatment is carried out at temperatures below 950 °C, which consumes much time in accomplishing the required case depth. Therefore, surface hardening treatment presents an obstacle to enhancing efficiency in the production of these machine parts.

In this technological background, a vacuum carburizing method has been developed as one of the recent carburizing techniques which are expected to enable to accomplish sufficient carburizing in a fairly short time.¹⁻³⁾ In general, vacuum carburizing treatment is carried out at relatively high temperatures, usually above 950 °C.

However, the employment of higher temperatures in carburizing has presented new problems in the commercial production of machine parts. When structural machine parts which are formed of a conventional structural steel, *i.e.*, it is usually referred to as case hardening steel, are subjected to the high temperature vacuum carburizing treatment, this temperature is liable to cause coarsening of the grain size of the treated steel, resulting in a high potential for large thermal strain to the machine parts or significant lowering of their mechanical properties. As a countermeasure, it is normal to carry out the so-called grain refining treatment after the vacuum carburizing treatment is completed. The procedure is that the carburized machine parts are once cooled to below the transformation temperature and heated

up again to the austenitizing temperature and then quenched. However, the addition of the cooling and reheating processes to the vacuum carburizing treatment means a considerable increase in the length of time required to accomplish surface hardening. As a result, the total operation time required does not differ much from that of the conventional gas carburizing treatment. This is a major reason why the vacuum carburizing method has been slow to catch on in industry in spite of its advantages in carburizing efficiency. In order to solve these problems in the vacuum carburizing method, it became necessary to develop a steel whose grains do not easily become coarse during high temperature vacuum carburizing treatment.

Recently, Takayama *et al.*⁴⁾ have investigated the grain growth characteristics of dual phase steel, and have found that the grain growth rate of this steel is markedly slower than that of single phase steel. Kamata *et al.*⁵⁾ have proposed the application of dual phase steel to case hardening steel.

In the present study, in order to shorten the total operation time of vacuum carburizing treatment, an attempt was made to develop a dual phase steel, which would not require grain refining treatment subsequent to vacuum carburizing at a high temperature above 1 000 °C.

II. Experimental Procedure

Low carbon steel containing Si has an austenite (γ)-ferrite (α) two-phase structure at a high temperature above 1 000 °C, as shown in Fig. 1. Supposing the dual phase steel composed of α - and γ -phases at 1 000 °C, as shown by the solid circle in Fig. 1, were carburized at 1 000 °C, the core structure would be fine due to the (α + γ) dual phase structure. However, the structure of the carburized layer would become coarse, because it would have changed into γ -single phase from (α + γ) dual phase during carburizing.⁴⁾ As a countermeasure to grain coarsening in the carburized layer, the authors have examined carbide dispersion carburizing which was studied by Chiba *et al.*⁷⁾ by adding V to the steel. Figure 2 shows the isothermal section diagram of the Fe-V-C system at 1 000 °C.⁸⁾ When the steel, whose composition is shown by the solid circle in Fig. 2, is carburized at 1 000 °C, the surface composition of the steel changes into that indicated by the open

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circle during carburizing, and V_4C_3 precipitates in the carburized layer. The precipitation of V_4C_3 in the carburized layer should be effective in depressing the grain growth of γ -phase. On the basis of this concept, the authors studied the vacuum carburizing of dual phase steels containing Si and V, which were expected to be suitable for carburizing at a high temperature above 1 000 °C.

Table 1 shows the chemical composition of 4 kinds of dual phase steels and also of SCr420 for comparison purposes. The content of C and Si of dual phase steels was chosen through preliminary examination to obtain the most suitable volume fraction of α/γ phases at carburizing temperature above 1 000 °C. The content of Mn and Cr was chosen to obtain hardenability. The elements of V and Nb were chosen as the grain growth depressing ones.

Each of these steels was prepared by vacuum

Fig. 1. Vertical section diagram of Fe-Si-C system at 2 wt% Si; after Satō.⁶⁾

Fig. 2. Isothermal section diagram of Fe-C-V system at 1 000 °C; after Uhrenius.⁹⁾

melting and cast into ingots of 50 kg, and forged at 1 200 °C to cylindrical rods of 32 mm in diameter. The forged steel rods were normalized at 925 °C for 1 hr. Afterwards the steel rods were machined into cylindrical test pieces of 25 mm in diameter. Using these test pieces, the carburizing properties and mechanical properties of the steels were examined.

Figure 3 shows the vacuum carburizing heat pattern used in this experiment. In vacuum carburizing, the test pieces were quenched into oil directly without undergoing grain refinement treatment after being carburized. These test pieces were examined in respect to the grain size in both the case and core, the distribution of carbon as well as hardness of the case, and the amount of retained austenite phase in the surface. For comparison purposes, SCr420 was gas-carburized at 920 °C for a total of 9.5 hr, this heat pattern also being shown in Fig. 3.

The mechanical properties of the core were examined using the specimens which were heat-treated simulating vacuum carburizing.

III. Results and Discussion

1. Microstructure

Photograph 1 shows the comparison of a typical microstructure of the core of dual phase steel with that of SCr420 when quenched following vacuum carburizing at 1 050 °C. The dual phase steel had a ferrite phase and a bainite one which was γ -phase before quenching. These grains were very fine. The grain size numbers of α -phase and γ -phase were 9 and 8, respectively, according to JIS G0551. On the other hand, SCr420 had a very coarse structure. In this paper, the γ -phase means the austenite one

Fig. 3. Carburizing heat patterns.

Table 1. Chemical composition of specimens. (wt%)

Steel	C	Si	Mn	P	S	Cr	Al	N	Nb	V
No. 1	0.07	1.98	1.00	0.015	0.015	0.99	0.022	0.016	0.018	0.11
No. 2	0.07	2.00	1.01	0.015	0.015	1.00	0.021	0.013	0.020	0.19
No. 3	0.07	2.02	1.00	0.015	0.015	1.00	0.022	0.013	0.020	0.29
No. 4	0.07	2.00	0.99	0.015	0.015	1.00	0.020	0.013	0.040	0.30
SCr 420	0.22	0.26	0.82	0.014	0.022	1.14	0.024	0.0012	—	—

- (a) Steel No. 4
Grain size number α : 9.0, γ : 8.0
Mean grain diameter α : 18 μm , γ : 25 μm
- (b) SCr420
Grain size number: 5.2
Mean grain diameter: 64 μm

Photo. 1. Microstructure of the cores of dual phase steel No. 4 and SCr420 carburized at 1 050 °C. The γ -phase is the austenite one at 1 050 °C.

Fig. 4. Grain size of α - and γ -phases in the cores of the specimens carburized at 1 050 °C for 1.5 hr. The γ -phase is the austenite one at 1 050 °C.

at high temperature.

Figure 4 shows the relationship between the grain size of α - and γ -phases in the core and the volume fraction of γ -phase of the specimens carburized at 1 050 °C for 1.5 hr. The grain size of α -phase was under 20 μm in diameter on an average; the grain size number was over 8.5. The grain size of γ -phase became finer with a decrease in its volume fraction. Under carburizing at 1 050 °C, SCr420, which had 100 % γ -phase, had coarse grains of over 65 μm in diameter on the average. In contrast, the dual phase steels had finer and more regular grains of under 40 μm in diameter; the grain size number was over 6.

Photograph 2 shows the prior austenite grain boundaries in the typical microstructures of the carburized layer after carburizing. A half picture was superimposed on the tracing of the grain boundaries in order to make them clearer. The carburized layer of SCr420 had coarse grains with a diameter of 90 μm on the average, which corresponded to 4.3 in grain size number. On the other hand, the carburized layer of dual phase steel No. 4 had fine

- (a) Steel No. 4
Grain size number: 8.2
Mean grain diameter: 23 μm
- (b) SCr420
Grain size number: 4.3
Mean grain diameter: 90 μm

Photo. 2. Microstructure of carburized layers of dual phase steel No. 4 and SCr420 carburized at 1 050 °C for 1.5 hr.

Fig. 5. Austenite grain size in the carburized layers of the specimens carburized at 1 050 °C for 1.5 hr.

grains of 23 μm in diameter on the average, which corresponded to 8.2 in grain size number.

Figure 5 shows the relationship between the prior austenite grain size in the carburized layer and the amount of the grain growth depressing elements of V and Nb. The dual phase steel which contained 0.1 wt% V and 0.02 wt% Nb had finer austenite grain size than SCr420. The austenite grain in the carburized layer became finer with an increase in V content. The dual phase steel containing 0.3 wt% V and 0.04 wt% Nb had the finest structure of all the specimens.

In order to examine the precipitation of V_4C_3 in the carburized layer, the dual phase steel containing 0.1 wt% V was examined by electron micrography using the extracted replica technique. Photograph 3 shows the electron micrograph of extracted replica at a depth of 25 μm from the case surface of dual phase steel No. 1. The particle indicated with an arrow was confirmed by an analysis of its diffraction pattern to be a vanadium carbide. Although Nb_4C_3 could not be confirmed under this electron micrography using extracted replica technique, the precipitates are presumed to have precipitated in the carburized layer. These precipitates can be considered effective in depressing austenite grain growth in the

Photo. 3. (a) Electron micrograph of extracted replica at a depth of 25 μm inward from the surface of dual phase steel No. 1 carburized at 1 050 $^{\circ}\text{C}$ for 1.5 hr. (b) Electron diffraction pattern taken from the precipitate (indicated by an arrow).

(a) Dual phase steel treated by vacuum carburizing
(b) SCr420 treated by gas carburizing

Photo. 4. Microstructure of the carburized layer (after buffing).

carburized layer of dual phase steel during carburizing. No precipitation of V_4C_3 in the core was detected under electron micrography using extracted replica technique.

Photograph 4 shows the intergranular oxidation of vacuum-carburized dual phase steel and gas-carburized SCr420. There was no intergranular oxide layer observed in the vacuum-carburized dual phase steel. The same fact is reported in Ref. 9) as for the vacuum-carburized SCr420 steel. The intergranular oxide layer in the gas-carburized SCr420 was shown in Photo. 4 for comparison purposes.

2. Distributions of Carbon and Hardness

Figure 6 shows the distributions of carbon in the carburized layer. As a result of comparing the distributions of carbon in vacuum-carburized and gas-carburized SCr420, the carburizing rate in vacuum carburizing at high temperatures is confirmed to be most excellent. The carbon content in the carburized layer of the dual phase steels was lower than that of SCr420, although they were carburized under the same condition. The reason is that the initial carbon content of the dual phase steels was lower

V.C.: Vacuum carburizing, G.C.: Gas carburizing

Fig. 6. Distributions of carbon in the carburized layer.

V.C.: Vacuum carburizing, G.C. Gas carburizing

Fig. 7. Distributions of hardness in the carburized layer.

Table 2. Amount of retained austenite in the surface of the carburized layer.

Steel	Average (%)	
	$\{211\}_{\alpha}-\{311\}_{\gamma}$	$\{211\}_{\alpha}-\{220\}_{\gamma}$
No. 1		27.9
No. 2		30.7
No. 3		37.4
No. 4		33.4
SCr 420		46.7
SCr 420 (G.C.)		25.0

than that of SCr420, and the dual phase steels contain Si which is a depressing element in carburizing.¹⁰⁾ However, it can be considered that the Si content presents no significant problem in carburizing, because the carbon content was above 0.8 wt% at the surface of the carburized layer, and the effective carburizing depth (*i.e.*, the case depth at 0.4 wt% C) was above 0.9 mm under vacuum carburizing at 1 050 $^{\circ}\text{C}$ for 1.5 hr.

Figure 7 shows the distributions of hardness in the carburized layer. Table 2 shows the amount of

retained austenite phase in the surface, as determined by the X-ray diffraction method. Although these dual phase steels were not tempered at 160 °C, their case hardenability is considered to be sufficiently good. The hardness of the surfaces of the dual phase steels was higher than that of SCr420 treated by the gas-carburizing, in spite of the fact that dual phase steels had more retained austenite than SCr420. This would appear favorable in depressing pitting. On the other hand, SCr420 treated by vacuum carburizing had low surface hardness, because the carbon content at the surface was 1.1 wt%, resulting in retained austenite of 46 %.

3. Mechanical Properties

As mentioned above, the dual phase steels had sufficiently fine grains in both the case and the core, and sufficiently good distributions of carbon and hardness in the case. Among them, dual phase steel No. 4 had the finest structure in the case. Therefore, the mechanical properties of dual phase steel No. 4 were chosen for examination.

Tensile test pieces (JIS No. 4 reduced size) and Charpy impact test pieces (JIS No. 3) were cut off from heat-treated steel rods of 25 mm in diameter which had been subjected to the heat treatment process shown in Fig. 8. This heat pattern simulates vacuum carburizing. In dual phase steel, the ratio of the α/γ phases in the core depends on the holding temperature; therefore, the mechanical properties would change according to the quenching temperature. Accordingly, the quenching temperature was varied from 1 000 to 850 °C at intervals of 50 °C in order to determine which quenching temperature would provide the most excellent mechanical properties.

The tensile test and Charpy impact test were made

Fig. 8. Heat pattern for examining the mechanical properties of the core of dual phase steel No. 4.

Fig. 9. Mechanical properties of dual phase steel No. 4.

on these test pieces at room temperature.

Figure 9 shows the relationship between the ultimate tensile strength and the quenching temperature and also the relationship between the Charpy impact value and the quenching temperature. For comparison, the values of SCr420 are quoted from the Data Sheet published by the Iron and Steel Institute of Japan,¹¹⁾ in which SCr420 was subjected to heat treatment at 925 °C for 4 hr, then at 880 °C for 1 hr, followed by oil quenching, then reheating at 180 °C for 4 hr and finally quenching into water.

The ultimate tensile strength of dual phase steel No. 4 increased with an increase in quenching temperature. When quenched at 1 000 °C, it was 87 kgf/mm², which was at the same level as SCr420. On the other hand, the Charpy impact value was almost constant, that is 11 kgf·m/cm², in spite of the quenching temperature, which was almost the same as that of SCr420.

Figure 10 shows the volume fraction of γ -phase in dual phase steel No. 4 quenched at each temperature. The volume fraction of γ -phase increased linearly with an increase in the quenching temperature. The volume fraction of γ -phase was 50 % at 1 000 °C.

Figure 11 shows the relationship between the mechanical properties and the volume fraction of γ -phase. The ultimate tensile strength depended strongly on the volume fraction of γ -phase which would harden by quenching. On the other hand, the Charpy impact value was almost constant within the limits of this experiment in spite of the different volume fraction of γ -phase. This characteristic is considered to be due to its fine duplex structure.

Fig. 10. Volume fraction of γ -phase of dual phase steel No. 4. The γ -phase is the austenite one at high temperature.

Fig. 11. Mechanical properties of dual phase steel No. 4. The γ -phase is the austenite one at high temperature.

IV. Conclusions

The carburizing and mechanical properties in the vacuum carburizing at 1 050 °C were examined on 2 wt% Si steels containing V and Nb, and the following results were obtained.

(1) The core structure of the specimens during carburizing was in ($\alpha+\gamma$) dual phase, and the grain size numbers of the α - and γ -phases were 9.5~8.8 and 9.5~6.7, respectively.

(2) The prior austenite grain size of the case structure became finer with an increase in the content of V and Nb. The finest structure of the case was obtained in the steel containing 0.3 wt% V and 0.04 wt% Nb.

(3) The precipitation of V_4C_3 was observed in the case through electron micrography using extracted replica technique. These precipitates can be considered effective in depressing austenite grain growth in the case.

(4) The mechanical properties of core were excellent and almost the same as those of thermally refined SCr420.

(5) As mentioned above, the 2 wt% Si steel containing 0.3 wt% V and 0.04 wt% Nb had fine structure in both the core and case, sufficient surface hardness, and mechanical properties equivalent to those of SCr420 even when the steel was subjected to vacuum carburizing at 1 050 °C without refining

treatment. Therefore, this steel is expected to be suitable for vacuum carburizing at a high temperature above 1 000 °C, and to be able to shorten the total operation time in vacuum carburizing treatment.

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