

# Effect of carbon and nitrogen concentrations on the structure formation of compound layer in nitrocarburizing

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For the purpose of controlling the structure of the compound layer produced by the nitrocarburizing to the  $\gamma'$  phase, nitrocarburizing with various changes of added gas was carried out, and the phase structure of the compound layer, carbon and nitrogen concentration distribution in the compound layer were investigated. As a result, it was confirmed that the behavior of carbon concentration in the compound layer changed depending on the type of added gas in nitrocarburizing. It was shown that the decarburized compound layer favorable for the  $\gamma'$  phase formation was formed when the decarburizing atmosphere gas was used. Furthermore, if nitriding with controlled nitriding potential is carried out,  $\gamma'$  phase control of the compound layer can be achieved even in nitrocarburizing.

**Keywords:** nitrocarburizing, compound layer,  $\gamma'$  phase, nitriding potential

## 1. Introduction

Surface hardening is applied to parts used in transport equipment such as motorcycles and various industrial machines to improve fatigue strength. Carburizing is a type of surface hardening that is widely applied to structural components because of its excellent productivity, cost, and quality balance. On the other hand, martensite produced by carburizing is a factor in heat treatment deformation of structural parts. Large deformations reduce productivity and cost because of the need for corrective processing.

Nitrocarburizing is an effective candidate for solving these problems because heat treatment deformation is smaller than carburizing. However, since the compound layer formed on the surface is very brittle and porous, it is easy to crack, so that a decrease in fatigue strength is often a problem. Recent studies have shown that fatigue strength reduction can be suppressed by controlling the structure of the compound layer from the general  $\epsilon$  phase to the  $\gamma'$  phase<sup>1-3</sup>) in the case of nitriding. Therefore, various methods for controlling the structure of the compound layer by nitriding have been reported, and it has been shown that it is important to control the nitriding potential ( $K_N$ ) and to reduce the carbon concentration in the compound layer in order to make the compound layer a  $\gamma'$  phase<sup>4</sup>). Although a technique for controlling the compound layer to the  $\gamma'$  phase has been established in this way, nitriding treatment which is a long-time treatment is a prerequisite, and it is important to establish a technique for nitrocarburizing which can be treated in a shorter time in industry. However, there is no report on the structural control of compound layer by nitrocarburizing.

In order to control the structure of the compound layer by nitrocarburizing, the effect of carbon and nitrogen concentration on the structure formation of the compound layer was clarified.

## 2. Experimental method

### 2.1 Test material and nitrocarburizing

SCM435H (JIS G 4052) was used as a test material. Chemical compositions are shown in Table 1. The commercially available SCM435 was quenched at 850°C,

tempered at 600°C, and processed into 10 mm square  $\times$  100 mmL specimens, and then conducted to various nitrocarburizing, gas nitriding, and plasma nitriding. The processing conditions are shown in Table 2.

The nitrocarburizing was based on  $NH_3$  gas, and  $CO_2$  and  $C_2H_2$  gas were added. In addition, since it has been reported that the structural change of the compound layer is mainly due to the decarburization phenomenon<sup>5</sup>), a nitrocarburizing with air was also carried out in anticipation of the decarburization effect. The gas nitriding was carried out in order to confirm the formation of the compound layer when the added gas was not used.  $K_N$  control is not conducted for both nitrocarburizing and gas nitriding.

The plasma nitriding is relatively easy to control the structure of the compound layer. Therefore, the  $\gamma'$  phase was intentionally generated by adjusting the added gases ( $N_2$  and  $H_2$ ) to guide the structural control of the compound layer in this paper.

Table 1 Chemical compositions of test materials (mass%)

C	Si	Mn	P	S	Cr	Mo
0.35	0.20	0.82	0.014	0.014	1.04	0.17

Table 2 Processing conditions of nitriding

Symbol	Heat pattern	Gas
GSN-CO (Nitrocarburizing with $CO_2$ )	150min at 570°C	$NH_3$ $CO_2$
GSN-CH (Nitrocarburizing with $C_2H_2$ )	150min at 570°C	$NH_3$ $C_2H_2$
GSN-Air (Nitrocarburizing with Air)	150min at 570°C	$NH_3$ Air
GN (Gas-Nitriding)	150min at 570°C	$NH_3$
PN (Plasma- Nitriding)	600min at 500°C	$N_2$ , $H_2$

### 2.2 Material evaluation

Each nitrided specimen was cut and subjected to SEM (Scanning Electron Microscope) observation and EBSD (Electron Back Scatter Diffraction) analysis to confirm the phase structure of the compound layer. EPMA (Electron

Probe Micro Analyzer) analysis was also performed to identify the carbon and nitrogen concentrations in the compound layer.

### 3. Result and discussion

#### 3.1 Compound layer structure

The cross-sectional observation results of each processed product are shown in Figure 1. In every cases, the formation of compound layer was confirmed in the surface layer. The compound layer thickness of nitrocarburizing product was about 20 $\mu$ m for GSN-CO with CO<sub>2</sub> addition, about 13 $\mu$ m for GSN-Air with air addition, and about 4 $\mu$ m for GSN-CH with C<sub>2</sub>H<sub>2</sub> addition. Gas nitriding (GN) without added gas was about 1 $\mu$ m. This is thought to be due to the long time required for compound layer formation by nitriding<sup>6)</sup>. On the other hand, nitrocarburizing with the addition of CO<sub>2</sub> and air forms oxides and carbides on the surface, and which is expected to promote nitriding by using them as catalysts<sup>7)</sup>. Therefore, it is considered that a relatively thick compound layer was obtained even under the same treatment conditions. The compound layer thickness of the nitrocarburizing with the addition of C<sub>2</sub>H<sub>2</sub> (GSN-CH) was as thin as about 4 $\mu$ m, which may be due to the fact that H<sub>2</sub> released during the decomposition of C<sub>2</sub>H<sub>2</sub> decreased the nitriding potential (K<sub>N</sub>). As a result of EBSD analysis, the phase composition of the compound layer of each treatment was mainly composed of the  $\epsilon$  phase. Plasma nitriding (PN), which was also carried out to guide the  $\gamma'$  phase control, produced a compound layer of about 6 $\mu$ m of  $\gamma'$  single phase.

#### 3.2 Carbon and nitrogen concentrations

The results of EPMA analysis to investigate the carbon and nitrogen concentrations in the compound layer of each treated product are shown in Figure 2. In the nitrogen distribution, the concentration region which can be attributed to the compound layer formed on the surface of each treatment is confirmed. On the other hand, there was a significant difference in carbon concentration between treatments. In nitrocarburizing with CO<sub>2</sub> and Air, and plasma nitriding, there was a clearly tendency of decarburization toward the surface. On the other hand, carburizing tendency was confirmed in the nitrocarburizing with C<sub>2</sub>H<sub>2</sub> addition. This is thought to be because C<sub>2</sub>H<sub>2</sub> is a highly carburizable gas.

From the results of EPMA analysis, the concentrations of carbon and nitrogen in the surface layer and near the interface of the compound layer were extracted respectively, and the results were arranged together with the phase structure of the compound layer, and are shown in Figure 3. It can be seen that a low carbon concentration and a low nitrogen concentration are necessary to make the compound layer a  $\gamma'$  phase. Therefore, in order to achieve  $\gamma'$  phase control of the compound layer by nitrocarburizing, it is considered necessary to reduce the nitrogen concentration of the compound layer with respect to the compound layer produced by nitrocarburizing with CO<sub>2</sub> addition.

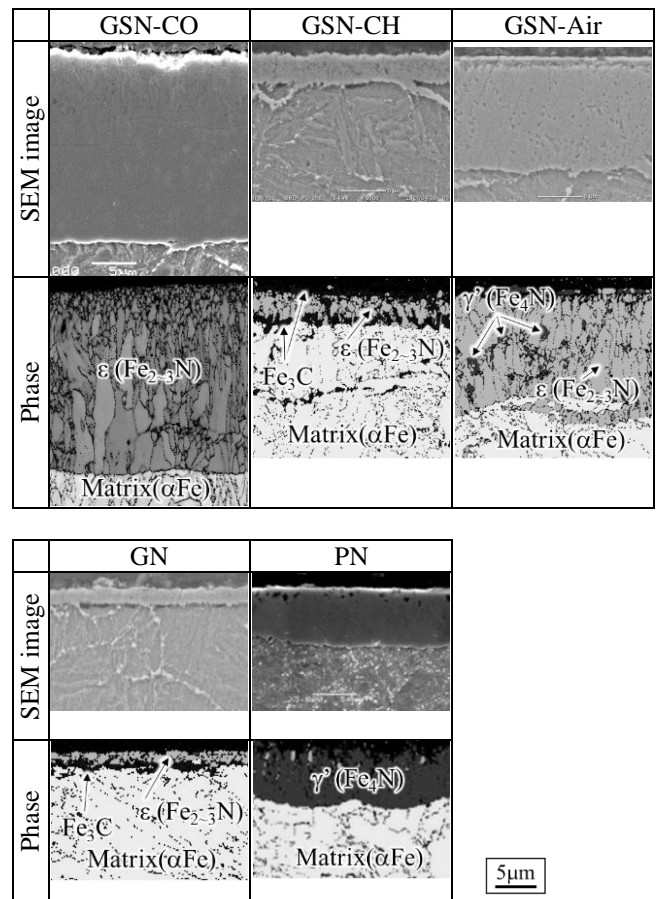


Figure 1 SEM images and phase map by EBSD patterns

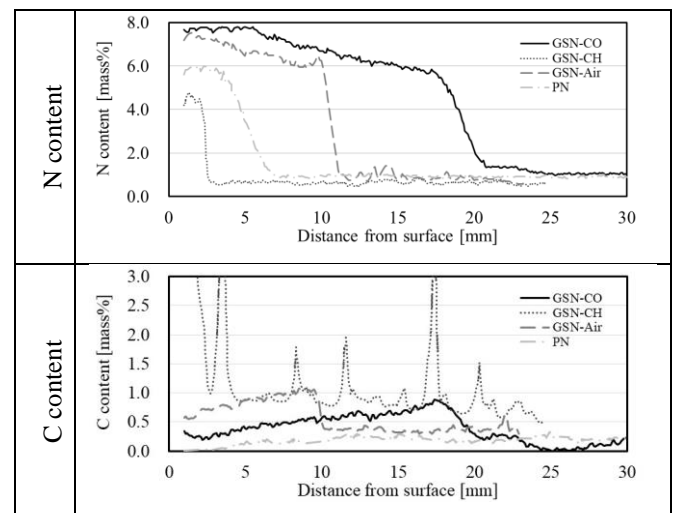


Figure 2 Profiles of Nitrogen and Carbon content by EPMA

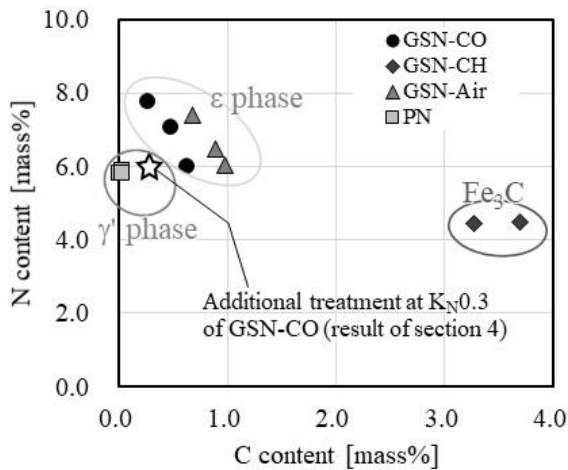


Figure 3 Effect of Nitrogen and Carbon on the phase structure

#### 4. Control of compound layer in nitrocarburizing

Before the detailed process examination, it was confirmed whether the compound layer can be  $\gamma'$  phase by controlling nitrogen concentration in nitrocarburizing using  $\text{CO}_2$  gas. Specifically, the GSN- $\text{CO}_2$  specimen was conducted to an additional treatment of 60min under a nitriding potential of  $K_N0.3$  in order to reduce the surface nitrogen concentration. The results are shown in Figure 4. The  $\gamma'$  phase was confirmed on the surface of the compound layer. The results of EPMA analysis shown in Figure 5 show that the nitrogen concentration in the surface layer is decreasing. The  $\gamma'$  phase is considered to be formed by the phase transformation from the  $\epsilon$  phase. From the above results, it was confirmed that the phase structure of the compound layer was dominated by the concentration of carbon and nitrogen in the surface layer. If the compound layer is formed by nitrocarburizing using  $\text{CO}_2$  as an additive gas, the compound layer can be controlled to  $\gamma'$  phase by denitriding under  $K_N$  control. We plan to continue to conduct verification in a consistent process.

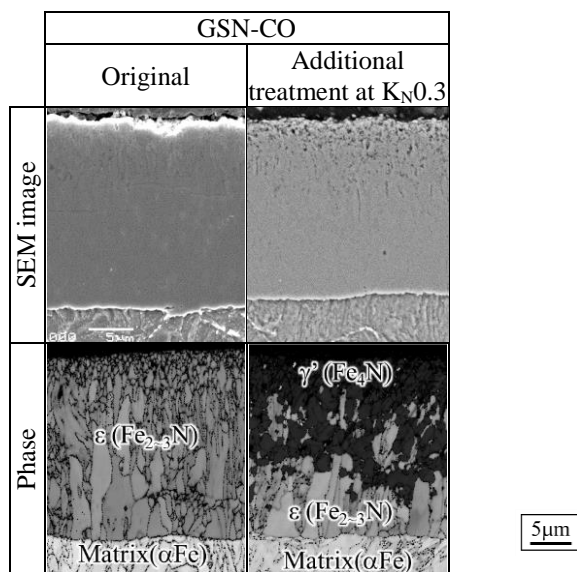


Figure 4 SEM images and phase map by EBSD patterns

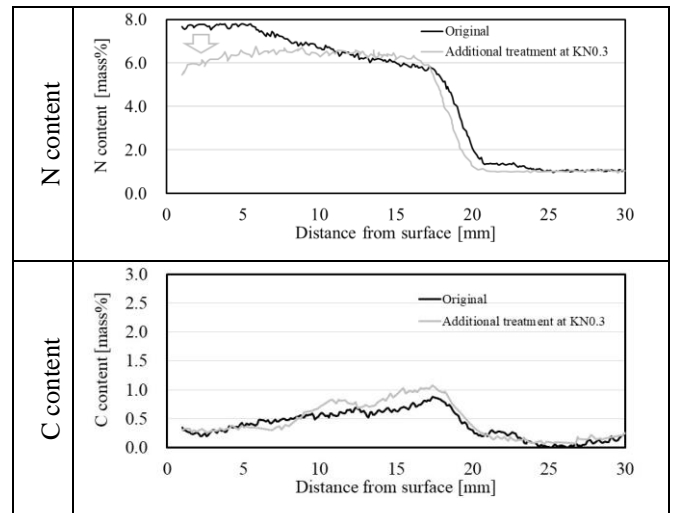


Figure 5 Profiles of Nitrogen and Carbon content by EPMA

#### 5. Conclusions

In the nitrocarburizing, it was found that the carbon concentration distribution in the compound layer differed depending on the type of added gas. To control the compound layer to the  $\gamma'$  phase, carbon and nitrogen concentrations must be optimally balanced. Therefore, in order to make the compound layer  $\gamma'$  phase by nitrocarburizing, it is important to optimize the nitrogen concentration of the compound layer produced by nitrocarburizing using  $\text{CO}_2$  as an additive gas.

#### 6. References

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