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The Effect of the Transformation of ε -Fe₂₋₃N into γ' -Fe₄N Phase on the Fatigue Strength of Gas-Nitrided Pure Iron

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Abstract: The effect of the iron nitride phases, ε -Fe₂₋₃N and γ' -Fe₄N, on the fatigue strength was investigated. Pure iron was used to observe only the effect of nitride, excluding the effects of factors, such as residual stress, depending on the alloy composition and microstructural change according to working on the fatigue strength. In this work, ε and γ' phases were respectively grown at a time on the surface of the pure iron specimens using the appropriate nitriding potential K_N, the mixture rates of ammonia and hydrogen gases, at same temperature of 570 °C according to the Fe-N Lehrer diagram. Another γ' phase was prepared by first growing the ε phase and then transformed from ε phase into γ' phase by changing the K_N at the same temperature of 570 °C in the 2-stage gas nitriding. The fatigue strengths of the iron nitride consisted of ε and γ' phases, γ' phase, and γ' phase grown by the 2-stage gas nitriding were evaluated, respectively. As a result, first, it can be seen that the diffusion layer of ε phase was deeper than γ' phase, but fatigue strength was lower. On the other hand, fatigue strength of both the γ' phases are higher than that of the ε , and the fatigue strength of γ' phase nitride grown by 2-stage gas nitriding was almost similar to that of γ' phase nitride grown at a time, i.e., fatigue strength was not significantly related to diffusion depth and depended on nitride phases in this study. Secondly, we cannot clearly conclude that there was the difference in fatigue strength according to the thickness of nitride layer consisted of γ' phase. However, it is clear that when ε phase was transformed to γ' phase, fatigue strength had the same level as γ' phase formed at one time.

Keywords: 2-stage gas nitriding; phase-controlled gas nitriding; phase transformation; Lehrer diagram; fatigue strength

1. Introduction

Gas nitriding is of considerable technological importance, because it can make a pronounced improvement on the fatigue strength, the wear, and the corrosion resistance of metals [1–3]. In gas nitriding, the surface properties of metals are improved by iron nitride layer and diffusion zone formed through the following mechanisms: Nitrogen (N) atoms are transferred into the iron and steel during the nitriding [4–6]. These N atoms were dissolved in the interstitial sites of the ferrite lattice and its mobility on the substrate of the interstitial sites can be large and its interaction with matrix atoms also be strong because of its surrounding strain fields accommodating the misfit of the size between



the atom and the interstitial site [7,8]. Through these mechanisms, the iron nitrides layer, consisting of ε -Fe₂₋₃N (ε) and/or γ' -Fe₄N (γ') phases, is formed with thickness of 10–20 μ m on the surface of metals and largely depends on nitrogen concentration and presented in the Fe-N phase diagram [9,10]. The diffusion layer is also formed at a depth of about 0.2 to 1.0 mm from surface.

Recently, many studied have been conducted to improve the fatigue strength of irons and steels by gas nitriding [11–14]. However, most of these studies have documented that thick diffusion layers have a significant effect on increased fatigue strength [15,16]. On the other hand, some studies report that iron nitride formed on the surface rather than diffusion layer affects the fatigue strength [17,18]. In particular, some studies indicate that nitride consisting of only γ' phase has higher fatigue strength than nitrides consisting of only ε phase or ε/γ' mixed phases.

Therefore, this study was conducted to confirm the following two points.

#1: It was investigated whether the fatigue strength was affected by iron nitrides formed on surface, not improved by diffusion layer. To this end, iron nitrides were formed by gas nitriding on pure iron, unlike the existing literature [11–14]. The reason for using pure iron was to minimize the effect of other factors of steels, which are such as residual stress depending on the alloy composition and microstructural change according to working, on the fatigue strength. In addition, the iron nitrides formed on the surface was subjected to a phase-controlled gas nitriding to accurately form ε or γ' phases. Phase-controlled gas nitriding is a gas nitriding technology capable of controlling and forming iron nitrides corresponding to a stable iron nitride phase region by gas nitriding with conditions of a specific temperature and nitriding potential, K_N, in the Fe-N Lehrer diagram.

#2: As mentioned in #1, it was reported that the fatigue strength of irons and steels was improved when iron nitride consisted of only γ' phase rather than ε phase. In addition, in the literature, it is shown that that fatigue strength was improved when γ' phase in iron nitride layer occupied a large portion [12]. Therefore, this study is intended to investigate the effect on fatigue strength by forming thick iron nitride consisted of γ' phase through 2-stage gas nitriding. 2-stage gas nitriding is a technique in which an iron nitride layer consisting of ε phase, which is grown to be relatively thick compared to that consisting of γ' phase at the same time, is first formed on the surface, and then iron nitride consisting of ε phase is phase-transformed to γ' phase by controlling K_N [12,19,20]; i.e., it was intended to investigate how the fatigue strength of iron nitride consisting of γ' phase, which was phase-transformed in ε phase through 2-stage gas nitriding, and that of iron nitride consisting of γ' phase, grown at one time through phase-controlled gas nitriding, are compared.

2. Materials and Methods

A commercial pure iron shaped into a round bar was used as a nitriding specimen and its chemical composition is presented in Table 1. The pure iron round bar was cut out from a round bar with the dimensions of about 30 mm in diameter and 8 mm in thickness, and annealed at 870 °C for 2 h in a pure H_2 gas atmosphere. Before nitriding, the surface of specimen was polished with a final stage of 1 μ m diamond.

Table 1. Chemical composition of pure iron in this study.

Composition	С	Si	Mn	Р	S	Fe
Pure iron	0.0036	0.026	0.257	0.0111	0.0053	Bal.

Phase-controlled and 2-stage gas nitriding were carried out in a horizontal quartz tube furnace with a diameter of 50 mm. The temperature was controlled within ±3 °C in about 5 cm of the uniformity temperature zone in furnace, where the specimen was placed by a ceramic boat. The flow of NH₃ and H₂ was controlled by a separate mass-flow controller. Phase-controlled gas nitriding to form iron nitrides consisted of ε phase or γ' phase on the surface of pure iron were controlled accurately with operating parameters at 570 °C, K_N = 1.4 atm^{-1/2} for 240 min and at 570 °C, K_N = 0.38 atm^{-1/2} for

240 min. 2-stage gas nitriding was performed to transform the ε phase into γ' phase with operating parameters as follows: at first stage, 570 °C and K_N = 1.4 atm^{-1/2} for 240 min, at second stage, 570 °C and K_N = 0.38 atm^{-1/2} for 30, 60, and 120 min. All of gas nitriding conditions are summarized in Table 2.

Gas Nitriding	Phase-Controlled	l Phase-Controlled	Phase-Transformed γ' -Fe ₄ N	
Conditions	ε-Fe ₂₋₃ N	γ' -Fe ₄ N	1st Stage	2nd Stage
Temperature (°C)	570	570	570	570
Time (min)	240	240	240	120
Nitriding potential (K_N , atm ^{-1/2})	1.41	0.38	1.41	0.38
Pressure (atm)	1	1	1	1

Table 2. Experimental conditions of gas-nitrided pure iron with various nitriding potential.

The furnace was first preheated and purged at temperature of 250 °C for about 100 min, and then raised the nitriding temperature of 570 °C. K_N was periodically measured to achieve the accurate values which depends on ammonia dissociation percentage during gas nitriding. In this work, the value of in-process K_N was little changed during gas nitriding because of very low ammonia dissociation in a horizontal tube furnace that was less than 3 vol.% of the input ammonia gas. As soon as the gas nitriding is completed, the specimen was cooled by blowing cold air continuously to prevent peeling off of the nitride layer when it was dropped into water for quenching. The cooling time at which the specimens reached room temperature was less than 5 min as sample size was small.

After gas nitriding, the microstructure of iron nitride was observed by the optical microscopy and FESEM. Additionally, the gas-nitrided specimens were characterized by high-resolution electron back-scatter diffraction (EBSD). Data were recorded and analyzed with the TSL OIM analysis software. To index the ε and γ' phases, these phases are defined using the lattice constants and phase–space groups reported in the literature [21]. The hexagonal closed-packed ε phase belongs to the P63/mmc space group and has the nominal lattice parameters of a = 2.529 Å and c = 4.107 Å [8,21]. The face-centered cubic γ' phases belong to the Pm3m space group and has lattice parameter of a = 3.798 Å [9,22].

X-ray diffraction (XRD) analysis was performed using a Rigaku Ultima 4 X-ray diffractometer to determine iron nitride phases. Measurements were made using Cu K α (λ =0.15406 nm) radiation at 40 kV and a dual position graphite monochromator for Cu in the diffracted beam. The range of the diffraction angle (2 θ) was from 20° and 80° with a step size of 0.02°. XRD patterns were recorded from the surface of the all the nitrided specimen. To identify the nitride phase from the position of the diffraction peaks, data from ICSD database were used.

The Knoop micro hardness test was conducted using a Mitutoyo HM-109 hardness tester with a load of 50 gf to determine the hardness of iron nitride and diffusion layer. For each nitride specimen, the hardness profile was plotted by the mean value of three measurements of hardness.

For the rotary bending fatigue test, the hourglass shaped test specimens were prepared according to the specification as shown in Figure 1, and then gas nitriding was performed under the planned conditions, and a sample was separately prepared to confirm the phase of the nitrided specimens. The fatigue test specimens were fixed with a jig during gas nitriding to minimize distortion. As a result, dimensional changes did not occur in all specimens after gas nitriding.



Figure 1. Geometry dimensions and photo of rotating bending fatigue test specimen which used in this study.

The main purpose of most engineering fatigue tests is to determine the relationship between the applied load and the number of load applications to cause failure, and to obtain some estimate of the probability of failure under specified loading conditions. Fatigue strength was determined using a rotating bending fatigue machine. Rotating bending fatigue tests were conducted under the conditions of load frequency of 3000 rpm in a laboratory air atmosphere. Hence, the tensile and compressive stresses on the specimen surface were calculated by Equation (1).

$$\sigma = \frac{32 \times \frac{WL}{2}}{\pi \times x^3} \tag{1}$$

where x (= 12 mm) is the diameter of the effectiveness region in the specimen, W(kgf) is the applied load on the specimen and L is the distance between the points of load application. In each test, the number of cycles to fatigue failure was noted; the tests were terminated if no failure had occurred after 10^7 cycles. ASTM E466 standard was employed to determine fatigue strength [23].

3. Results and Discussion

3.1. Calculated Thermodynamics

The Fe-N Lehrer diagram, which is well known in other literature [10,24–26], was absolutely necessary to accurately control iron nitrides, ε and γ' phases, with a suitable K_N in this study. In this study, the Fe-N Lehrer diagram was calculated using Equations (2) and (3) and nitrogen activity obtained from the Fe-N phase diagram database of Commercialized Thermo-Calc. Program (CHALPAD) and shown in Figure 2. The Fe-N Lehrer diagram showed almost similar patterns to that of H. Du. because of Gibbs energy of equations (4) given in the research of H. Du [24].

$$P_{N_2}^{1/2} = \frac{P_{NH_3}}{P_{H_2}^{3/2}} exp\left(\frac{\Delta G^0}{RT}\right)$$
(2)

 $\frac{P_{NH_3}}{P_{H_2}^{3/2}}$ is usually regarded as nitriding potential (K_N) or N potential in experiments.

$$\Delta G^0 = -G^0_{NH_3} + \frac{3}{2}G^0_{H_2} \tag{3}$$

$$\Delta G^{0} = -G_{NH_{3}}^{0} + \frac{\frac{3}{2}G_{H_{2}}^{0} + G_{N}^{0}}{= +45,403 - 128.94 \times T - 10.785}$$
(4)

$$\times TlnT\left(\frac{J}{mol}\right)$$

 ΔG^0 is the standard Gibbs energy of the NH₃ dissociation reaction.

From this calculation, in order to stably control nitride phases based on the above calculation results, in the Fe-N Lehrer diagram of Figure 2, K_N to form iron nitride consisted of ϵ phase was selected as 1.4 atm^{-1/2} and K_N to form iron consisted of γ' phase was selected 0.38 atm^{-1/2} at 570 °C.



Figure 2. Lehrer diagram which are calculated with Thermo-Calc. software in this study.

3.2. Microstructural and Phase Analysis

3.2.1. OM, FESEM, and GDS Analysis

The cross-sectional optical images of the microstructures of pure irons each subjected to gas nitriding with various K_N at temperature of 570 °C mentioned in the previous experimental method are shown in Figure 3. The nitride layers were observed with various thicknesses near the surface, but diffusion zone usually in gas-nitrided iron and steel was not clearly visible in these optical micrographs. The thickness of the nitride layer on the surface decreased with decreasing in K_N . The pure iron nitrided at 1.4 atm^{-1/2} of nitriding potential has a thickness of 20.6 ± 5 µm and there are many pores in the layer as shown in Figure 3a. On the other hand, the pure iron nitrided at 0.3 atm^{-1/2} of nitriding potential has a thickness of 7 ± 2 µm and there were a few pores in the layer as shown in Figure 3b. The pure iron nitrided with phase transformation with changing 1.4 atm^{-1/2} to 0.38 atm^{-1/2} of the nitriding potential has a thickness of 13.0 ± 5 µm and there were many pores in the layer as shown in Figure 3c.



Figure 3. Cross-sectional OM images from the gas-nitrided pure iron with various nitriding potential: (a) 1.4, (b) 0.38 and (c) 1st stage: 1.4 and 2nd stage: 0.38 of nitriding potential at 570 °C for (a), (b) 4 h and (c) 1st stage: 4 h and 2nd stage: 2 h.

The FESEM cross-sectional images of the nitrided pure irons are shown in Figure 4, and the GDS analysis results are also superimposed on the images and plotted in the direction of nitrogen diffusion from the surface into inside. The thicknesses of all nitrides layers are almost similar to those observed with optical microscope and the values are $19 \pm 2 \,\mu m$ for 1.4 atm^{-1/2} of the nitriding potential, $8 \pm 1 \,\mu m$

for 0.38 atm^{-1/2} of the nitriding potential, and $13 \pm 2 \mu m$ for phase transformation with changing 1.4 atm^{-1/2} to 0.38 atm^{-1/2} of the nitriding potential. The maximum nitrogen concentration in nitride layer of the pure irons nitrided by 1.4 and 0.3 atm^{-1/2} of the nitriding potential and phase-transformed with changing 1.4 atm^{-1/2} to 0.38 atm^{-1/2} of the nitriding potential were about 9.4 wt.%, 5.8 wt.% and 5.9 wt.%, respectively. The nitride phases on the surface can be deduced through qualitative relation between the Fe-N phase diagram and the nitrogen concentration depth profile results. As a result, the nitride layer of Figure 4a may consist of ε/γ' phases and the nitride layers of (b) and (c) may consist of γ' phase, i.e., it can be seen that ε and γ' corresponding to Figure 4a,b are controlled with an accurate nitride phases by the nitriding potentials selected in the Lehrer diagram of Figure 2b. In addition, the γ' nitride phase corresponding to Figure 4c can also be inferred that the phase transformation from ε to γ' phase, which is the objective in this study, has occurred almost completely by the 2-stage gas nitriding. In addition, a small number of pores were observed in nitride consisting of γ' phase, which is mentioned in the optical microscopic image.



Figure 4. Cross-sectional SEM micrograph obtained perpendicular to the nitrogen diffusion direction from the gas-nitrided pure iron with various nitriding potential and the change in the nitrogen weight percent from GDS analysis: Gas-nitrided pure iron with (**a**) 1.4, (**b**) 0.38 and (**c**) 1st stage: 1.4 and 2nd stage: 0.38 of nitriding potential at 570 °C for (**a**), (**b**) 4 h and (**c**) 1st stage: 4 h and 2nd stage: 2 h.

3.2.2. XRD and EBSD Analysis

Although the nitride phases were inferred from the results of cross-sectional observation and N concentration analysis, XRD analysis was performed to observe the more accurate nitride phases. The XRD results of pure iron nitrided with 1.4 and 0.3 atm^{-1/2} of nitriding potential and the 2-stage gas nitriding were presented in Figure 5. It can be seen that the peaks (33.401, 41.22, 47.97, 70.180, 84.76, 89.51) [27] of the γ' phase were detected in all nitrided pure irons. On the other hand, the peaks (38.338, 43.768, 57.625, 77.037) [28] of the ε phase were detected only in pure iron nitrided with 1.4 atm^{-1/2} of nitriding potential and 2-stage gas nitriding. From the results, it can be seen that nitride formed by gas nitriding with 0.3 atm^{-1/2} of nitriding potential was completely controlled in γ' phase. However, in 2-stage gas nitriding, ε phase was detected, and it was considered that ε phase was not completely phase-transformed into γ' phase and remained. The reason for this was estimated that the initial ε phase nitride layer was thick and there was not enough time to completely transform all the ε nitride phase.

In addition, magnetite peaks were observed, which are thought to be formed by oxygen that was not removed when nitrogen was cooled after gas nitriding.



Figure 5. The XRD patterns for Gas-nitrided pure iron with 1.4, 0.38 and 1st stage: 1.4 and 2nd stage: 0.38 of nitriding potential at 570 °C.

Although the nitride phases were analyzed by XRD, the nitride phases were observed by the EBSD analysis to further increase accuracy. In particular, it was intended to measure the distribution and amount of ε phase nitride remaining in the nitride layer after the 2-stage gas nitriding. The EBSD results are shown in Figure 6. The FESEM images were taken with back-scattered electron (BE) contrast from the nitride layer shown in left side of Figure 6. The phase map from the EBSD analysis of the same area in right side of Figure 6 corresponds perfectly with the BE image of the FESEM but with a better contrast showing the grain boundaries and the change in the microstructure from the surface into the bulk material.

In EBSD analysis results, the nitride layer consists of bilayer of ε and γ' phases in Figure 6a: the thick outer layer consisted of the ε phase nitrides (yellow color) and the thin inner layer consisted of γ' phase nitrides (red color), and there was mono layer consisting γ' phase nitrides in Figure 6b. In Figure 6c, it was observed that ε phase nitrides detected in XRD analysis were very slightly distributed only in the grain boundary, and most of the phase was transformed into γ' phase. It was presumed that the remaining ε phase nitride was present on in the grain boundary because the relatively high N concentration was maintained by the movement and diffusion of N atoms despite the lowering of nitriding potential in the second stage of 2-stage gas nitriding.

The thicknesses of the nitride layers observed in the EBSD analysis were almost similar to those of nitride layers measured by OM and FESEM. The thicknesses of nitride layer shown in Figure 6a consisting of ε and γ' phases were 17.5 ± 2 µm and 4.1 ± 1 µm, respectively. The thicknesses of the nitride layers shown in Figure 6b,c consisting of γ' phase were 6 ± 2 µm and 12 ± 3 µm, respectively. Compared to the growth thickness of nitrides, it can be seen that ε phase nitride was grown faster layer than γ' phase nitride at the same time, i.e., the result that can be inferred through the previous analysis will be possible to form in a short time the thicker single γ' phase nitride layer by phase transformation of 2-stage gas nitriding as suggested in the literature [12].



Figure 6. (Right side) phase map from the cross-sectional gas-nitrided pure iron, with the phase relevant color coded map, α -Fe or α -Fe[N] (red), γ' phase (green), ε phase (yellow), (Left side) Cross-sectional SEM images from EBSD area: Gas-nitrided pure iron with (**a**) 1.4, (**b**) 0.38 and (**c**) 1st stage: 1.4 and 2nd stage: 0.38 of nitriding potential at 570 °C for (**a**), (**b**) 4 h and (**c**) 1st stage: 4 h and 2nd stage: 2 h.

3.3. Phase-Transformation Gas Nitriding

In this study, ε phase nitride, which had not been transformed and left a small amount, was not a significant problem to confirm whether the γ' phase nitride of thick layer, which was formed by 2-stage gas nitriding, was able to improve fatigue strength compared to γ' phase nitride formed at once. According to the literature [12,19,20], however, it was recorded that the ε phase formed in the first stage was mostly transformed into γ' phase in the second stage for a short time. Nevertheless, this study showed different results than expected that ε phase formed in the first stage would be completely transformed into the γ' phase in the second stage during 2-stage gas nitriding. Therefore, additional 2-stage gas nitriding experiments were conducted to confirm whether ε phase would not be transformed into γ' phase due to insufficient time mentioned above.

In this part, the degree of γ' phase transformation according to the change of the second stage process times of 30 min, 60 min, and 120 min during 2-stage gas nitriding was observed by analyzing with EBSD measurements and the results are shown in Figure 7. In all the 2-stage gas nitriding, the nitride layers, which consisted of ε and γ' phases, were almost transformed to monolayers consisting of γ' phase nitride and a small amount of ε phase nitride was observed at 30 min and 120 min. However, it was confirmed that the ε phase was not observed at 60 min, and it was completely transformed into the γ' phase. In addition, the result of observation was that thickness of nitride decreased from 21.6 µm to 10 ± 1 µm in gas nitriding for a relatively short time of 30 min to transform the ε phase formed at one time into a γ' phase. The thickness of the nitride, which was phase-transformed for 60 min by increasing the time of second stage, was 10 ± 2 µm, which was similar to the result of 30 min. However, it can be seen that the thickness of nitride the phase-transformed nitride for 120 min in the second stage was about 12 ± 3 µm, which was about 2 µm thicker than that of the previous two nitrides, but the amount of ε phase mentioned several times earlier also increased.

According to these results, it cannot be concluded whether there was ε phase that has not been transformed due to lack of time, but was not observed in the result of 60 min or the change in the concentration of nitride occurring due to excessive diffusion and/or movement of already accumulated N atoms at the boundary of 60 min. No further analysis and discussion will be made in this study. This is because [28] explained the mechanism of phase transformation to γ' phase through annealing after gas nitriding, but this cannot be clearly indicated because the process conditions such as transformation

process time and temperature were different to explain the mechanism of phase-transformation gas nitriding conducted in this study with reference.



Figure 7. (Right side) phase map from the cross section gas-nitrided pure iron, with the phase relevant color coded map, α -Fe or α -Fe[N] (red), γ' phase (green), ε phase (yellow); (Left side) Cross-sectional SEM images from EBSD area: Gas-nitrided pure iron with 1st stage: 1.4 atm^{-1/2} of nitriding potential for (**a**) only 1st stage: 240 min, and 2nd stage: 0.38 atm^{-1/2} of nitriding potential at 570 °C for (**b**) 1st stage: 240 min and 2nd stage: 30 min, (**c**) 1st stage: 240 min and 2nd stage: 60 min and (**d**) 1st stage: 240 min and 2nd stage: 120 min.

3.4. Micro Hardness Profiles

The cross-sectional hardness of ε/γ' , γ' and γ' with a small amount ε phase of nitrides defined by the previously analyzed results, was measured five times in total to present the average value and the hardness depth profiles are shown in Figure 8. The maximum hardness values and locations of ε/γ' , γ' and γ' with a small amount of ε phase nitrides were about 526 ± 25 HK_{0.05}, 400 ± 5 HK_{0.05} and 415 ± 15 HK_{0.05} at 10 µm, 5 µm and 3 µm, respectively. The maximum hardness of γ' nitride with a small amount ε phase formed by 2-stage gas nitriding was relatively lower than that of ε nitride and higher than that of γ' nitride, which means that most of the ε phase have been transformed into γ' phase. In addition, the maximum hardness of γ' nitride with a small amount ε phase was slightly higher than that of γ' nitride formed at a time because, as can be seen from the previous EBSD analysis, there was a small amount of ε phase that was not transformed, i.e., the fact that can be confirmed through the hardness results was that even when ε phase was transformed to γ' phase, it can have almost the same hardness as γ' phase formed at a time. This means that the hardness values of nitrides depended only on the nitrogen concentration. The core hardness value of pure iron was about 160 ± 2 HK_{0.05}.



Figure 8. Microhardness profiles obtained on gas-nitrided pure iron with 1.4, 0.38 and 1st step: 1.4 and 2nd step: 0.38 of nitriding potential at 570 °C.

3.5. Fatigue Test Results

The fatigue strengths of nitrides consisting of ε and γ' phase, γ' phase, and transformed γ' phase were measured by rotating bending fatigue tests, and the results are shown in Figure 9. The fatigue limit stresses (failure strength of 10^7 cycles) of all nitrides are shown in Table 3.

It is generally known that the absolute value of fatigue strength is related to diffusion layer of nitrided irons and steels [15,16]. According to the hardness results, it can be seen that the diffusion layers of γ' phase and transformed γ' phase nitrides are almost the same, and only that of ε and γ' phase nitride was relatively thick. In the fatigue test result, however, it can be seen that the diffusion layer of nitride consisting of ε phase was the thickest while fatigue limit stress at 10⁷ cycles was the lowest except for the specimen without gas nitriding.

This study conducted a rotary bending fatigue test of nitrides formed by phase-controlled and phase-transformation gas nitriding to confirm that nitride consisting of γ' phase was superior to that consisting of ε phase, and the results are shown in Figure 9. The fatigue test was conducted in both low-cycle and high-cycle fatigue, and the fatigue limit stress did not cause fracture in the high-cycle of 10⁷. There is research that suggests the thicker the diffusion layer formed by gas nitriding, the better the fatigue strength [12]. However, in this study, it can be seen that the ε phase had a thicker diffusion layer than the γ' phase, but the fatigue strength was much lower. According to the results of fatigue tests, the fatigue strength differed depending on the phase of the nitride compared to the diffusion layer. In the literature [21], fracture of materials occurs due to surface crack nucleation which is originated from surface defects such as grooves, scratches, pores, holes, or metallic inclusions, even for low stresses.

The second purpose of this study is to verify the fact that fatigue strength is improved when γ' phase is thick. However, as expected, in the fatigue strength result, the nitride transformed from the ε phase to the γ' phase does not show a significant difference in fatigue strength although it is thicker than the γ' phase nitride formed at one time.

The fatigue strength of nitrided pure iron was higher than that of untreated pure iron from the fatigue test results, and the fatigue strength was improved when γ' phase layer was formed on the surface. According to the literature [29], the fatigue limit increases with increasing hardness up to hardness of about 400 HV, but fatigue limit is lowered when nonmetallic inclusions (carbide, nitride, etc.) exist in pure iron or steels with over 400 HV. In addition, when ε phase with high hardness and brittleness existed on the surface, fatigue crack occurred and propagated due to plastic deformation

due to repeated compression and tensile. The nitride consisted of thick ε phase cannot withstand the tensile stress in fatigue test because of directly passing the crack propagation through ε phase layer. On the other hand, γ' phase slowed down the initiation and propagation of cracks to the inside because compressive stress of γ' phase layer presented much larger than that of the ε phase layer [30]. When the compressive residual stress on the surface of the material was higher, the generation of the initial crack was delayed because the tensile stress generated in the fatigue test was relaxed. In addition, it was shown that nitride improves fatigue life because it plays a role of preventing progress and accumulation of dislocation. It seems to be due to ε phase remaining completely untransformed in the second stage nitriding.

Specimen	Failure Strength of 10 ⁷ Cycles		
Phase-controlled ε-Fe ₂₋₃ N	535		
Phase-controlled γ' -Fe ₄ N	570		
Phase-transformed γ' -Fe ₄ N	580		

Table 3. Fatigue limit stress of the nitride phases formed on surface of pure iron after gas nitriding.



Figure 9. S-N curve diagram of pure iron after gas nitriding with various nitriding conditions.

4. Conclusions

In this study, gas nitriding was conducted to examine the following two points. First, this study was conducted to observe if nitride consisting of γ' phase formed by gas nitriding can improve the fatigue strength compared to one consisted of ε phase. Secondly, it was shown that the thicker γ' phase nitride layer, the better the fatigue strength. Therefore, 2-stage gas nitriding, which was known to be able to formed thicker γ' phase relatively than one formed at one time during same time, was conducted and fatigue strength of nitride consisting of γ' phase formed by 2-stage gas nitriding was compared to one consisting of γ' phase formed at one time through fatigue testing. In addition, we observed how the fatigue strength of nitride consisted of γ' phase-transformed from ε phase changed.

The experimental results of this study were as follows:

(1) As planned in this study, nitrides consisting of ε phase and γ' phase were accurately formed by phase-controlled gas nitriding using appropriate 1.4 atm^{-1/2} and 0.38 atm^{-1/2} of nitriding potential in Fe-N Lehrer diagram at 570 °C, and a thick γ' phase could be formed through phase transformation in 2-stage gas nitriding. The γ' phase formed through phase-transformation nitriding was not completely phase-transformed in this study, and a small amount of ε phase remained at the grain boundaries.

(2) In the second stage of 2-stage nitriding, the phase-transformation process time was changed to 30 min, 60 min, and 120 min, and the process of phase transformation from ε phase to γ' phase was observed. A small amount of ε phase was observed at the results of 30 and 120 min in the second stage.

In addition, at 30 and 60 min, the thickness was slightly reduced compared to the thickness of nitride before phase transformation, but at 120 min, it tended to increase again.

(3) As previously known, nitride consisting of ε phase had a maximum hardness value of 526 HK_{0.05} and γ' phase and phase γ' transformed by 2-stage gas nitriding had relatively low hardness values of 400 HK_{0.05} and 415 HK_{0.05}, respectively. In addition, the hardness profile tended to be very similar to the nitrogen concentration profile, and it can be seen that the maximum hardness values and location of nitrides also depended on the nitrogen concentration.

(4) To examine the fact that the target both nitrides consisting of γ' phases in this study had higher fatigue strength than one consisted of ε phase, the fatigue limit stresses of nitrides were compared with each other at 10⁷ cycles.

(5) Second, it was predicted that thick nitride consisting of γ' phase-transformed through 2-stage gas nitriding would have better fatigue strength than one consisting of γ' phase formed at one time, but results of the fatigue strength were almost similar. In addition, fatigue strength of nitride consisting of γ' phase-transformed were significantly improved as compared to that of one consisted of ε phase. However, despite the relatively thick nitride consisting of γ' phase being transformed, as shown in a literature, the tendency for the thicker nitride consisting of γ' phase being higher with the fatigue strength did not appear in this study.

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