Effect of the ion nitriding surface hardening process on fatigue behavior of AISI 4340 steel

Sule Yildiz Sirina, Kahraman Sirinb, Erdinc Kalucc,⁎

a Vocational School of Asim Kocabiyik, Kocaeli University, 41800 Hereke- Kocaeli, Turkey
b Noksel Steel Pipe Industries Company, 54300 Hendek- Sakarya, Turkey
c Department of Mechanical Engineering, Engineering Faculty, Kocaeli University, 41040 Kocaeli, Turkey

ARTICLE DATA

Article history:
Received 12 June 2006
Received in revised form
24 January 2007
Accepted 30 January 2007

Keywords:
Ion nitriding
Fatigue strength
Mechanical properties
AISI 4340 steel

ABSTRACT

Ion nitriding is a glow discharge surface modification technique, which is primarily used to increase the fatigue strength, wear, corrosion resistance and surface hardness of steels. Because of the formation of high compressive residual stresses in the case region, increasing surface hardness and case depth cause remarkable improvement in fatigue properties of steels. In this study, the ion nitrided properties of quenched and tempered AISI 4340 low alloy steel were investigated under different process parameters including time and temperature. It has been found that the ion nitriding surface treatment improves the fatigue strength and increases the fatigue limit depending on the case depth. Up to 91% improvement in fatigue strength of the steel has been attained by ion nitriding. It was determined that, the subsurface ‘fish eye’ type formation is the dominant fatigue crack initiation mechanism in ion nitrided AISI 4340 steel in high cycle region and its origin was nonmetallic inclusions.

© 2007 Elsevier Inc. All rights reserved.

1. Introduction

Plasma or ion nitriding is a method of surface hardening using d.c. glow discharge technology to introduce elemental nitrogen to the surface of metal part for subsequent diffusion into the material [1,2]. The ion nitriding process which is applied to ferrous materials has been extensively used to improve the surface properties, wear, fatigue, corrosion and friction properties and also load bearing capacity of dynamically loaded components [3-6]. During the ion nitriding process, the nitriding reaction not only occurs at the surface but also in the subsurface owing to the long distance diffusion of nitrogen atoms from the surface towards the core. Diffused nitrogen into a steel surface is combined with alloying elements to form a fine dispersion of alloy nitrides. As a result, a thin iron nitride layer, which consists of γ-Fe₄N and/or ε-Fe₂,₃N intermetallic phases, referred to as the ‘white layer’ because it is not attacked by alcoholic nitric acid etch [7,8] is produced on the surface together with a relatively thick and strong diffusion zone in the subsurface of a steel component, which gradually reduces the hardness and nitrogen concentration towards the core, resulting in a diffuse case-core interface [9]. The hardness and the depth of the diffusion layer depend mainly on the amount of nitride-former elements in the steels [10-12], the time and the temperature of the ion nitriding treatment [12-14], the specific gas composition used in an ion nitriding system [15] and the initial microstructure of the material to nitriding [16,17]. Ion nitriding has recently received considerable industrial interest owing to its characteristic of faster nitrogen penetration, short treatment time, low process temperature, minimal distortion, clean specimens, low energy use and easier control of compound and diffusion layer formation compared with conventional techniques such as gas and liquid nitriding [8,18-21]. Nitriding has grown especially from the fact that, through nitriding very hard surface layers may be obtained without substantial modification of the bulk material.

⁎ Corresponding author. Tel.: +90 262 335 11 48x1147; fax: +90 262 335 28 80.
E-mail address: ekaluc@kou.edu.tr (E. Kaluc).

1044-5803/$ - see front matter © 2007 Elsevier Inc. All rights reserved.
doi:10.1016/j.matchar.2007.01.019
properties [12]. This led to numerous applications of the process in industries such as the manufacture of machine parts for plastics and food processing, packaging and tooling, as well as pumps and hydraulic machine parts, crankschafts, rolls and heavy gears, motor and car construction, cold and hot working dies and cutting tools [1,2].

Fatigue strength can be significantly improved by nitriding, and this improvement attributes to increasing surface hardness and residual stress distribution [22–28] or increasing case depth [9,14,24,25,29]. At the first model, improvement in fatigue behavior attributes to alterations of the surface properties and some other causes, as follows. The formation precipitate particles can hinder dislocation motion and, therefore, slip band penetration through the nitrided layer. A diffusion layer, which is formed during ion nitriding process, causes compressive residual stresses on or near the surface of machine parts, as well as low tensile residual stress in the core. Both increased residual stress and surface hardness result in improvements in the fatigue life because the thin, hard layer prevents plastic flow. Therefore, the slip bands can only be activated by very high stresses in this region. This means that plastic deformation, which is the source of crack initiation, probably appears in the subsurface. The suppression of slip band formation at the surface delays crack initiation and propagation, thus being responsible for the improvement of fatigue life [27]. At the other model, the effect of increasing the case depth on the fatigue limit can be viewed as effectively moving the fatigue crack initiation site further into the core. This means that a greater applied bending stress will be required at the surface to create a sufficiently high level of stress at the case–core interface to initiate failure [24]. In the high cycle regime, it was also determined that the subsurface fatigue crack formation was dominant in failure initiation mechanism in ion nitrided steels [8,9,14,24–30]. Not only the level of applied stress and the thickness of nitrided case of specimens, but also the inclusion size, type and orientation are very effective in the determination of crack origins [14,31,32].

There have been studies of the effects of plasma nitriding on the rotating bending fatigue strength of AISI 1045 [30,33], AISI 4140 [14,28], AISI 5140 [34], EN40B [9,24], 34CrNiMo6 [16], AISI 15B21H steel [35,36] and AISI 304L [37]. AISI 316 [38], 3CR12 stainless steel [38]. However, there has not been any information about fatigue behavior of ion nitrided AISI 4340. Therefore, in the present research AISI 4340 low-alloy steel was ion nitrided in NH3 under different conditions including time (2, 4, 8, 16 h) and temperature (500, 520, 540 °C) and their effect on the rotating bending fatigue strength was investigated.

### 2. Experimental Work

A standard medium carbon, CrMoNi low alloy AISI 4340 steel, widely used in the production of automotive crankschafts, rear axle shafts, aircraft crankschafts, connection rods, propeller hubs, gear, drive shafts, landing gear parts and heavy duty parts of rock drills, has been used in this study. The chemical composition of the steel is given in Table 1. The 22 mm diameter steel was received in normalized condition and austenitized at 840 °C for 30 min, quenched in oil and than tempered at 600 °C for 1 h. The hourglass shaped, rotating bending fatigue test specimens were machined according to DIN 50113 (Fig. 1) at CNC turning lathe with very sensitive temperature in the vacuum chamber. Microhardness measurements were used to determine diffusion layer case depth as well as to aid in characterizing the physical properties of the case as a function of depth [12,13]. The microhardness of the ion-nitrided surface was evaluated using a FutureTech microhardness tester with a load of 200 g. The hardness at 20 μm depth was chosen for comparison of surface hardness, so that any possible effects from a compound layer would be negligible. The case depth was defined as the depth at which the hardness is 10% Vickers above the core hardness. For each specimen’s hardness, profile was determined by three measurements at the cross section and the average was plotted. Optical microscopy was used for the determination of the white layer thickness and also phase analysis was conducted with a Shimadzu XWXRD-6000 X-ray diffractometer using Cu X-ray tube (λ = 1.5405 Å). The fracture surfaces of specimens were examined under optical microscopy and scanning electron microscopy in order to evaluate the crack initiation and growth characteristics of the materials in the core and nitrided case regions of specimens. 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 by using a rotating bending fatigue machine. Rotating bending fatigue test were conducted by being use a sinusoidal load of frequency 95 Hz (5700 rpm) in laboratory air atmosphere and load ration R = –1. Specimens ranging from 14 to 20 were used to obtain the fatigue strength for each

### Table 1 – Chemical composition of AISI 4340 steel

<table>
<thead>
<tr>
<th>Elements</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.38</td>
<td>0.26</td>
<td>0.69</td>
<td>0.014</td>
<td>0.001</td>
<td>0.80</td>
<td>0.22</td>
<td>0.69</td>
<td>Bal</td>
</tr>
</tbody>
</table>
condition of nitriding. In each test, the number of cycles to fatigue failure was noted; the tests were terminated if no failure had occurred after $10^6$ cycles and at least two specimens were tested for one stress level. Statistical analysis using four or five stress levels was done after the fatigue test results. ASTM E739-91 standard was employed to determine fatigue strength.

3. Results and Discussion

The microstructure of the nitrided material is shown in Fig. 2 which is ion nitrided at 500 °C for 4 h. The compound layer thicknesses, case depth, surface hardness, and fatigue strength of ion nitrided specimens are given in Table 2 with some dimensionless parameters like relative case area $A_R$, relative case depth $t/D$ and $D/(D−2t)$, together with details of applied heat treatments.

The thickness of compound layer increases parabolically with time [5] and the growth rate of compound layer is changed by temperature [21,34]. For example, increasing the treatment time from 2 to 16 h resulted in an increase in compound layer thickness from 2.6 to 4.8 μm at 500 °C; this increase in compound layer thickness was 4.8 to 8.8 μm at 540 °C (Fig. 3). Time and temperature can influence not only the thickness of the compound layer, but also which phases it consists of. Fig. 4 shows the diffraction patterns obtained for ion nitrided AISI 4340 low alloy steel at different temperature and time. The X-ray analysis shows the existence of a $\gamma$-Fe$_4$N phase in all compound zones on the surface at all temperature and time. The compound layer consisted of $\gamma$-Fe$_4$N, $\varepsilon$-Fe$_2$N, and $\varepsilon$-Fe$_{2.3}$N phases, and proportion of $\gamma$-Fe$_4$N to $\varepsilon$-Fe$_{2.3}$N and the thickness of this layer increased with increased time at 500 °C. The compound layer consisted of only $\gamma$-Fe$_4$N phase at 540 °C and the thickness of this layer increased with increased time.

Table 2 – Structural and fatigue properties of ion nitrided AISI 4340 steels

<table>
<thead>
<tr>
<th>Treatment details</th>
<th>Surface hardness, HV$_{0.2}$</th>
<th>Comp. layer, μm</th>
<th>Case depth, t μm</th>
<th>Relative case depth, (t/D)</th>
<th>$D/(D−2t)$</th>
<th>Relative case area, $A_R$</th>
<th>Fatigue strength, σ MPa</th>
<th>% improvement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austenitized 840 °C, 30 m/oil</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Q.T. and 2 h, 500 °C ion nitried</td>
<td>510</td>
<td>2.6</td>
<td>280</td>
<td>0.070</td>
<td>1.163</td>
<td>0.352</td>
<td>822</td>
<td>54</td>
</tr>
<tr>
<td>Q.T. and 4 h, 500 °C ion nitried</td>
<td>526</td>
<td>3.0</td>
<td>300</td>
<td>0.075</td>
<td>1.176</td>
<td>0.384</td>
<td>838</td>
<td>57</td>
</tr>
<tr>
<td>Q.T. and 8 h, 500 °C ion nitried</td>
<td>634</td>
<td>4.0</td>
<td>360</td>
<td>0.090</td>
<td>1.220</td>
<td>0.487</td>
<td>912</td>
<td>70</td>
</tr>
<tr>
<td>Q.T. and 16 h, 500 °C ion nitried</td>
<td>609</td>
<td>4.8</td>
<td>470</td>
<td>0.118</td>
<td>1.307</td>
<td>0.709</td>
<td>924</td>
<td>73</td>
</tr>
<tr>
<td>Q.T. and 4 h, 520 °C ion nitried</td>
<td>537</td>
<td>3.9</td>
<td>395</td>
<td>0.099</td>
<td>1.246</td>
<td>0.553</td>
<td>902</td>
<td>69</td>
</tr>
<tr>
<td>Q.T. and 2 h, 540 °C ion nitried</td>
<td>553</td>
<td>4.8</td>
<td>315</td>
<td>0.079</td>
<td>1.187</td>
<td>0.409</td>
<td>830</td>
<td>55</td>
</tr>
<tr>
<td>Q.T. and 4 h, 540 °C ion nitried</td>
<td>580</td>
<td>5.4</td>
<td>420</td>
<td>0.105</td>
<td>1.266</td>
<td>0.602</td>
<td>922</td>
<td>72</td>
</tr>
<tr>
<td>Q.T. and 8 h, 540 °C ion nitried</td>
<td>610</td>
<td>6.9</td>
<td>450</td>
<td>0.113</td>
<td>1.290</td>
<td>0.665</td>
<td>954</td>
<td>78</td>
</tr>
<tr>
<td>Q.T. and 16 h, 540 °C ion nitried</td>
<td>549</td>
<td>8.8</td>
<td>510</td>
<td>0.127</td>
<td>1.342</td>
<td>0.802</td>
<td>1024</td>
<td>91</td>
</tr>
</tbody>
</table>
The hardness profiles of nitrided cases are given in Fig. 5. The hardness decreases from surface to core, since the concentration of metal nitrides decreases towards core. The surface hardness of specimens is in the range of 560–634 HV$_{0.2}$, while the core hardness remains unchanged, 315 HV$_{0.2}$. The surface hardness is a strong function of temperature [39]. The hardness decreases can be explained in terms of precipitate size and density. Although the surface hardness of nitrided specimen increases with increasing time and temperature, the reduced surface hardness was observed at longer nitriding times.

Fig. 4 – X-ray diffraction patterns from the surface of specimens ion nitrided for (a) 500 °C, 2 h; (b) 500 °C, 16 h; (c) 540 °C, 2 h; (d) 540 °C, 16 h.

Fig. 5 – Microhardness profiles obtained on AISI 4340 after various nitriding treatments.
time because of the formation of larger nitride precipitates which are less effective in increasing hardness than smaller ones [5]. At the lower temperature (500 °C) and shorter treatment time (2 h), nitrogen uptake and precipitate density are low; therefore the amount of hardening is also low. At the higher temperature (540 °C) and longer treatment time (16 h), precipitate growth and tempering of matrix become important. The decrease in surface hardness is related to both precipitate density and size. The maximum surface hardness is obtained at temperature of 500 °C and a time of 8 h owing to optimum precipitate density and size. The reduction of surface hardness related to longer treatment time was more effective at high temperature (Fig. 6).

The effective cases depth of nitrided specimens based on 340 HV0.2 are given in Table 2. It can be seen from Table 2 that the case depth increased linearly with increased treatment time at both temperatures as indicated in some earlier studies [5,9,12,13] and the generation of nitrided case was much faster at 540 °C than 500 °C (Fig. 7) as expected for diffusion-controlled growth. It can be seen from Fig. 8 that higher treatment temperatures produce deeper cases at the same treatment times. While the maximum case depth obtained at temperature of 540 °C and a time of 16 h is 510 μm, the minimum case depth obtained at temperature of 500 °C and a time of 2 h is 280 μm. While the nitrided case depth increased strongly with temperature, the surface hardness decreased at longer nitriding time because of the formation of larger nitride precipitates [12,13]. It is interesting that the maximum hardness and the maximum case depth do not coincide [8,40].

Fig. 6 – Effect of nitriding temperature and time on surface hardness.

The S–N curves obtained from rotating bending fatigue tests are given in Fig. 9. Different fatigue limit properties were observed for all types of materials considered in this study, and an absolute progress in fatigue limit with increasing case depth was observed like previous investigations [9,14,24,25,29]. The effect of increasing the case depth on the fatigue limit can be viewed as effectively moving the fatigue crack initiation site further into the core. This means that a greater applied bending stress will be required at the surface to create a sufficiently high level of stress at the case–core interface to initiate failure [24]. Table 2 gives the fatigue strength improvements as a result of the ion nitriding treatment. Increase in case depth caused higher fatigue resistance, and an improvement of 91% in fatigue strength was developed for 16 h, 540 °C ion nitrided specimens, which had the heaviest nitrided case in the study, when the fatigue performance of quenched and tempered steel was assumed to be the reference. It was seen that a linear relationship was obtained between fatigue strength of steel and case depth (Fig. 10) [41].

The section size is one of the most important factors determining the fatigue performance of machine parts. Some dimensionless parameters are defined to express the proportion of nitrided case zone in the whole cross-section such as, relative case area \( A_R \), which is defined as the ratio of case area to core area, the ratio of specimen diameter to core diameter \( D/(D−2t) \) and the ratio of specimen diameter to specimen diameter \( t/D \) for round specimens [14,24,25]. The change in fatigue performance and the improvement in fatigue strength, as functions of dimensionless parameters \( D/(D−2t) \), \( t/D \) and \( A_R \) are given in Table 2, referring to the

Fig. 7 – The relationships between case depth and treatment time at 500 and 540 °C.

Fig. 8 – The relationships between nitriding temperature and case depth at 4 h.
fatigue performance in quenched and tempered condition of steel. There is a relationship between fatigue strength and dimensionless parameters.

Macroscopic examinations of the fracture surface of fatigued ion nitrided samples have shown that the fracture surface can be characterized into two groups. In the first group, the fatigue cracks initiated at the surface in the low-cycle/high stress region and in the second group cracks initiate at the subsurface, case-core transition zone in the high-cycle/low stress region. All the fractured specimens were observed to have failed by the ‘fish eye’ phenomenon with fatigue crack originating from nonmetallic inclusion in the high-cycle/low stress region. In this type, the cracks are originated at inclusions in the core and at positions near the case/core interface [14,24–27,29]. The fish eye cracks are circular in shape, which is a direct consequence of pure rotating bending type of cyclic stressing [14]. Scanning electron micrograph (SEM) fractograph of a typical subsurface fish eye crack formation is given in Fig. 11. The specimen was ion nitrided at 540 °C for 16 h and loaded under 1100 MPa cyclic stress up to complete fracture at the failure cycle of $1.87 \times 10^5$.

Higher magnification of Fig. 11 reveals that such nuclei have their origin at a non-metallic inclusion, but it could not be determined what it was. On the other hand, the fatigue cracks

![Fig. 11 – SEM fractograph of a typical subsurface fish eye crack formation.](image)
4. Conclusions

1. The thickness of the compound layer increased with increasing treatment time and temperature. The structure of the compound layer formed during the ion nitriding of AISI 4340 steel shown a transformation from ε-Fe₂₃N₃ nitride to γ-Fe₃N nitride with increasing treatment time and temperature. While γ-Fe₃N phase was formed at all temperatures and times, ε-Fe₂₃N₃ was not formed at high temperatures.

2. The case depth increased with increasing time and temperature for volume diffusion controlled growth. For this reason, the progress of case depth in ion nitriding can be expressed by a power function of process time.

3. The surface hardness increased with increasing time and temperature of ion nitriding treatment. But for extended nitriding periods, a significant loss of surface hardness was observed at both nitriding temperatures as a result of the formation of large nitride precipitates.

4. The surface hardness of AISI 4340 steel was increased by ion nitriding. It is interesting that the maximum hardness and the maximum case does not coincide. While the maximum surface hardness was obtained at 500 °C for 8 h, the largest case depth thickness formed at 540 °C for 16 h.

5. The fatigue strength of AISI 4340 steel was increased by up to 91% by ion nitriding process. The improvement of 91% in fatigue strength was developed for 16 h, 540 °C ion nitrided specimens, which had the heaviest nitried case in the core region from nonmetallic inclusions.

6. In the high cycle fatigue regime, fish eye crack initiation is one of the main characteristics of fractured ion nitried specimens. The subsurface crack initiation may take place in the core region from nonmetallic inclusions.

**REFERENCES**