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Influence of initial conditions on the mechanical behavior of ion nitrided AISI 4340 steel

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ABSTRACT

The influence of ion nitriding with surface roughness and initial microstructure on fatigue behavior and mechanical strength of AISI 4340 steel have been investigated under different process parameters including time and temperature. The first group fatigue and tension test specimens were not heat treated before ion nitriding and received in hot-rolled condition while the second group specimens were quenched and tempered. While the surface of the reduced section was being mechanically polished for the fatigue specimens, some specimens in the second group were not mechanically polished before ion nitriding to obtain the third fatigue test specimens group. While the effects of initial microstructure, surface roughness and ion nitriding conditions on fatigue behavior were investigated, the effects of initial microstructure and ion nitriding conditions on mechanical properties were investigated. First improvement in fatigue and mechanical properties were obtained with quenched and tempered. The obtained case depth of QT AISI 4340 steel is thicker than that of the hot-rolled AISI 4340 steel at all ion nitriding conditions and a linear relationship was obtained between fatigue strength of steel and case depth for both steels. Although the subsurface fatigue crack formation is dominant in failure initiation mechanism in ion nitrided steels, surface roughness effect on fatigue strength continued in a decreasing rate.

1. Introduction

The ion nitriding is a plasma-activated thermodynamic process for the production of case hardened surface layer not only for ferrous metals but also for an increasing number of nonferrous metals. A few hundred volts dc is applied between the grounded container wall (positive) and an insulated center post supporting the work (negative) to be nitrided. A glow discharge is created in the ionized gas accelerating positive nitrogen ions to the work. During the ion nitriding process, the nitriding reaction occurs not only at the surface but also in the subsurface owing to the long-distance diffusion of nitrogen atoms from the surface towards the core. As a result, a thin iron nitride layer, which is called compound layer or white layer, is produced on the surface together with a relatively thick and strong diffusion layer in the subsurface of a steel component. The case layer produced by ion nitriding shows the typical profile exhibited by thermal diffusion processes. The concentration of nitrogen peaks at the surface, and then decreases exponentially as a function of depth into the surface. The case structure of a nitrided steel, which may include a diffusion layer with or without a compound layer, depends on the type and concentration of nitride former alloying elements in the steels, the time-temperature exposure of a particular nitriding treatment [1], and the gas composition used for ion nitriding [2]. As with other diffusion methods, the initial microstructure can also influence the response of a material to nitriding. In case of alloy steels, a quenched and tempered structure is considered to produce the optimum nitriding results [1].

The ion nitriding process is more economical than all the other commercial nitriding processes because of faster nitrogen diffusion, lower nitriding temperature, shorter treatment times and lower gas consumption [3]. Easier control of compound and diffusion layer formation and also suppressed compound layer formation, obtainable at very hard surface layers without substantial modification of the bulk material properties, are the other advantages of ion nitriding [4–7]. Thanks to a number of advantages, the ion nitriding process, which is extensively used to improve the surface properties, wear, fatigue, corrosion, friction properties, and also load bearing capacity of dynamically loaded components, is widely used in manufacturing and machining industries primarily to treat engine components, tools for hot/cold work, dye castings, machine tools, and tribocomponents.

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Fatigue strength in high cycle region can be significantly improved by ion nitriding. The formation of precipitates in the diffusion layer tends to increase the surface hardness and introduces beneficial high compressive residual stresses on or near the surface of machine parts, as well as low tensile residual stresses in the core [4]. These beneficial stresses lower the magnitude of the applied tensile stresses; and therefore, increase the fatigue life of the component [8]. While some investigations on ion nitrided steels have revealed that fatigue limit increases with increasing case depth of nitrided layers [4,5,9–13], the others have revealed that fatigue limit increases with the strength and residual stress distribution in the cross-section of specimens [14–16]. Contrary to low cycle fatigue region, it has also been found out that in high cycle fatigue region the subsurface fatigue crack formation is dominant in failure initiation mechanism in ion nitrided steels [4,5,9–17]. 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 on determination of crack origins [4,18].

Since the nitrided case is extremely thin and very hard, it should be supported by the strong and tough core. Mechanical characteristics of metals are changed by ion nitriding [17]. In as much as ion nitriding changes the chemical composition of the surface layer, the transformation kinetics and tempering responses are different in the regions of the resulting composite. As a result, it leads to a core and a case with different microstructures and different responses to deformations. Thus, the surface hardened component is considered as a composite which consists of a high strength–low ductility case and low strength–high ductility core [17,19]. Considering nitrided steel as a composite material, mechanical characteristics of such composite will depend on the strength, toughness and hardness of both the substrate and the nitrided layer [9,10,20]. The surface layer roughness, which is conditioned by manufacturing processes, particularly by finishing treatment, has a very important effect on the fatigue strength. Surface roughness causes local stress concentrations and reduces the fatigue strength [21]. Although the subsurface fatigue crack formation in high cycle region is dominant in failure initiation mechanism in ion nitrided steels, surface roughness effect on fatigue strength continues in a decreasing rate [22].

Heat treatment of steel substrates influences their microstructure which alters nitrogen diffusivity during ion nitriding and the obtained nitrided layers may differ in composition, thickness and mechanical properties. There have been many studies related to the effects of ion nitriding on the fatigue strength of quenched and tempered steels. However, there has not been any information about the effect of initial microstructure, which is related to heat treatment of the substrate before ion nitriding, on the fatigue and mechanical properties of the component. And also the effect of surface roughness condition related to finishing treatment before ion nitriding on the fatigue strength has not been studied. In this study, the influence of ion nitriding with surface roughness condition and initial microstructure on fatigue behavior and mechanical strength of AISI 4340 low alloy steel were investigated under various process conditions as temperature and time of nitriding process by using a microhardness tester, optical microscope, surface profilometer, scanning electron microscope (SEM), rotating bending fatigue tester, and tensile test machine.

2. Experimental work

Low alloy AISI 4340 steel has been used in this study and the chemical composition of the steel is given in Table 1. The 22 mm diameter steel bar was received in hot-rolled condition. The hourglass shaped, rotating bending fatigue test specimens were machined according to DIN 50113 (Fig. 1a) at CNC turning lathe with very sensitive dimension. Fatigue specimens were divided into three groups. The first group specimens were not heat treated before ion nitriding and received in hot-rolled condition. The second group specimens were initially heat treated by austenitizing to evaluate the influence of the initial microstructure on the structure and properties of the nitrided layers, at 840 °C for 30 min followed by oil quenching and tempering at 600 °C for 1 h, QT. While the surface of the reduced section was being mechanically polished by using alumina slurry to an average roughness below approximately 0.3 μm for the fatigue specimens, some specimens in the second group were not mechanically polished before ion nitriding and received CNC turning lathe condition to obtain the third specimens group. Thus, the effects of surface roughness condition, initial microstructure and ion nitriding conditions on fatigue behavior were investigated.

Table 1
Chemical composition of AISI 4340 steel.

C	Si	Mn	P	S	Cr	Mo	Ni	Fe
0.38	0.26	0.69	0.016	0.004	0.80	0.22	0.69	Bal.

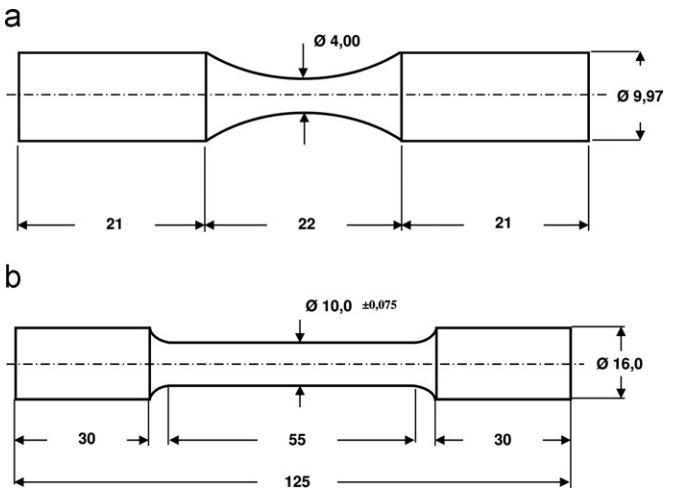

Fig. 1. a. Fatigue test specimen, dimensions in mm, b. Mechanical test specimen, dimensions in mm.

Tension test specimens were machined according to EN10002 (Fig. 1b) at CNC turning lathe with very sensitive dimension. Tension test specimens were divided into two groups. The first group covers the specimens which were not heat treated before ion nitriding and received in hot-rolled condition. The second group specimens were initially heat treated by austenitizing at 840 °C for 30 minutes, followed by oil quenching and tempering at 600 °C for 1 h before nitriding, QT, similar to fatigue specimens. Thus, the effects of initial microstructure and ion nitriding conditions on mechanical properties were investigated.

The specimens were degreased in trichloroethylene and then placed in an industrial DC glow discharge ion nitriding furnace. The ion nitriding furnace employed in this study is similar to that described by Genel et al. [4]. The specimens were employed as the cathode. The furnace was evacuated by the vacuum pump and then pure NH_3 (ammonia) treatment gas was admitted into the furnace through a gas distribution valve. The specimens were ion nitrided for 2 h, 4 h, 8 h, and 16 h at a temperature of 500 °C, 520 °C or 540 °C, voltage between 480 V and 540 V depending on the desired temperature. Temperatures of specimens were measured by thermocouples, which were embedded in the specimens. The treatment time was accounted after treatment temperature was reached. After ion nitriding, the specimens were cooled to room temperature in the vacuum chamber.

Surface profilometer was used to determine the surface roughness before and after ion nitriding. Surface roughness was characterized by measuring the average roughness Ra, according to the ISO 4287:1997 standard [23]. During the surface roughness measurements, the measurement length was chosen as 0.75 mm. For each specimen, surface roughness was determined by 10 measurements and the average was plotted.

Microhardness measurements were used to determine the diffusion layer thickness as well as to aid in characterizing the physical properties of the diffusion layer as a function of depth. The surface hardness and the microhardness profile of ion-nitrided specimens were evaluated using a microhardness tester with a load of 200 g, according to the ASTM E384-99 standard [24]. 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 hardness profile of each specimen was determined by three measurements at the cross section and the average was plotted. The case depth was defined as the depth at which the hardness was 10% HV above the core hardness.

The main purpose of the most engineering fatigue tests is to determine the relationship between the applied load and the number of load applications to cause failure, and to estimate the probability of failure under specified loading conditions. Rotating fatigue tests were performed on a cantilever bending test machine, at a frequency of 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 condition of ion nitriding. The tests were carried out until complete fracture of specimens occurred. The tests were concluded if no failure had occurred after 10^6 cycles and at least two specimens were tested for one stress level. In each test, the number of cycles to fatigue failure was noted. 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. The fracture surfaces of specimens were examined under optical microscope and scanning electron microscope in order to evaluate the crack initiation and growth characteristics of the materials in the core and nitrided case regions of specimens.

Mechanical properties of ion nitrided AISI 4340 steel under different conditions were measured by using a tensile test machine. For each group, mechanical properties of specimens were determined by three measurements and average values were taken into consideration.

3. Results and discussion

Microstructures of the first group specimens not heat treated before ion nitriding, received in hot-rolled condition and the second group specimens initially heat treated by austenitizing at 840 °C for 30 min, followed by oil quenching and tempering at 600 °C for 1 h are given in Fig. 2. The hardness of hot-rolled AISI 4340 steel has increased from 210 $Hv_{0,2}$ to 315 $Hv_{0,2}$ by quenching and tempering.

The hardness profiles of ion nitrided cases are given in Figs. 3 and 4 in respectively for QT AISI 4340 steel and hot-rolled AISI 4340 steel. The hardness profile of both hot-rolled AISI

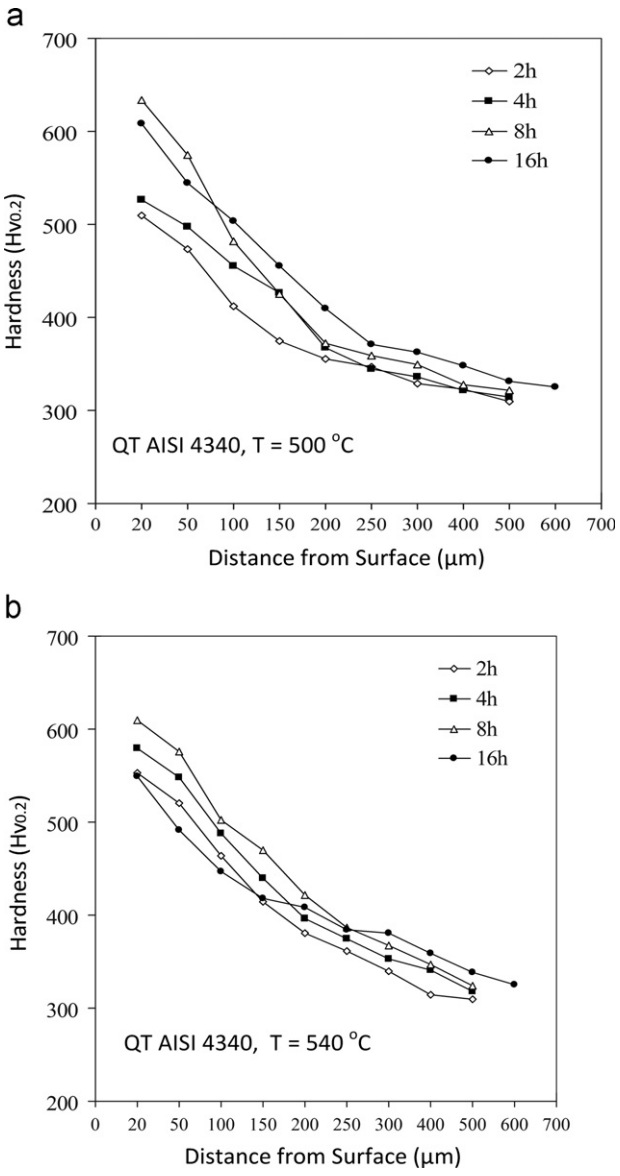

Fig. 3. Microhardness profiles of QT AISI 4340 steel ion nitrided at different process parameters.

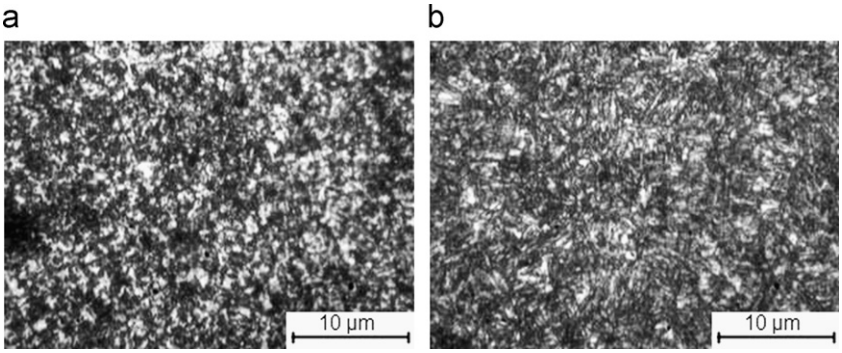

Fig. 2. a. The microstructure of hot-rolled AISI 4340 steel, and b. The microstructure of QT AISI 4340 steel.

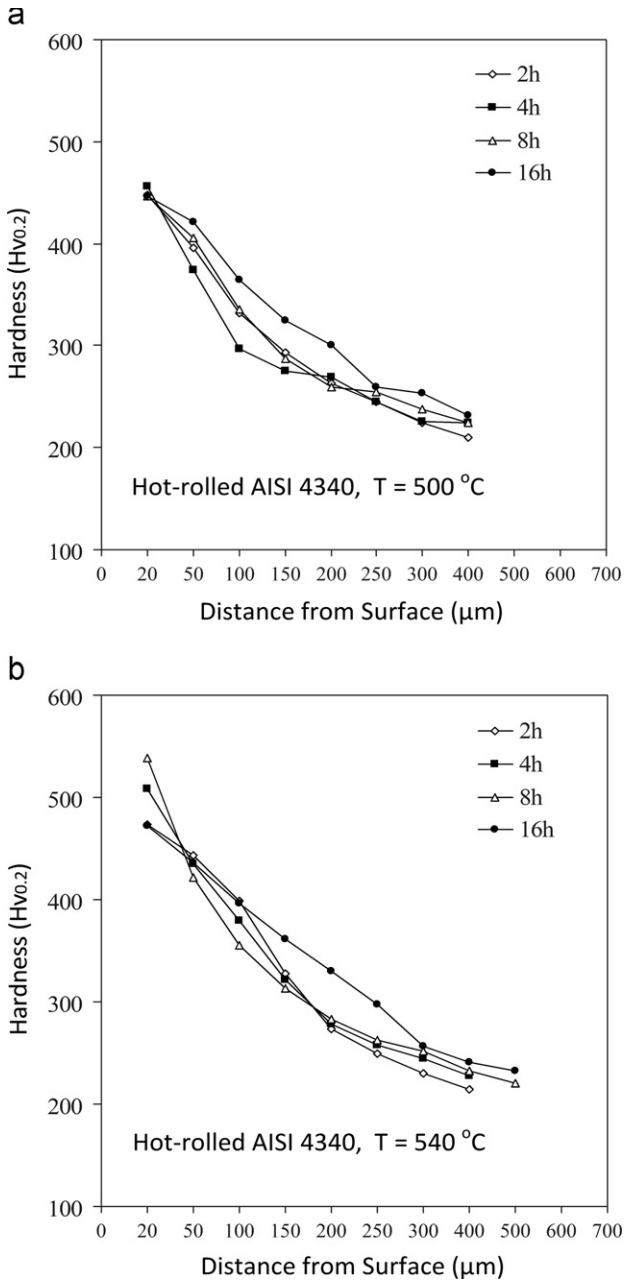

Fig. 4. Microhardness profiles of hot-rolled AISI 4340 steel ion nitrided at different process parameters.

4340 steel specimens and QT AISI 4340 steel specimens decreases gradually from the surface toward the core. The surface hardness of ion nitrided QT AISI 4340 steel specimens is in the range of 510–634 $Hv_{0,2}$, while the core hardness remains unchanged, 315 $Hv_{0,2}$. The surface hardness of ion nitrided hot-rolled AISI 4340 steel specimens is in the range of 447–539 $Hv_{0,2}$, while the core hardness remains unchanged, 210 $Hv_{0,2}$. In other words, ion nitriding increases the surface hardness of hot-rolled AISI 4340 steel specimens up to the core hardness values of QT AISI 4340 steel specimens. The nitrided layer on the QT AISI 4340 steel specimens exhibit a hardness higher than that of the nitrided layer on the hot-rolled AISI 4340 steel specimens at all ion nitriding conditions. The highest surface hardness is about 634 $Hv_{0,2}$ for QT AISI 4340 steel ion nitrided at 500 °C, 8 h and it is about 539 $Hv_{0,2}$ for hot-rolled AISI 4340 steel ion nitrided at 540 °C, 8 h, as shown in Table 2. There is a maximum hardness increase of about 634 $Hv_{0,2}$ and 539 $Hv_{0,2}$, which correspond to a 101% increase for the QT AISI 4340 specimens and a 157% increase for the hot-rolled AISI 4340 specimens, respectively. While the core hardness of QT AISI 4340 steel is higher than the core hardness of hot-rolled AISI 4340 steel, the increase in the percentage of the surface hardness of hot-rolled AISI 4340 specimen is higher than the increase in the percentage of the surface hardness of QT AISI 4340 specimen at all ion nitriding conditions, as shown in Table 2. Although the surface hardness of nitrided specimen increases with increasing time and temperature, the reduced surface hardness is observed at longer nitriding time due to the formation of larger nitride precipitates which are less effective in increasing hardness than smaller ones. This reduction of surface hardness is more effective at high temperature (Fig. 5a and b). Hardness increasing after ion nitriding is attributed to the solid solution hardening by *N* atoms, the formation of fine precipitates of alloy nitrides acting as barriers against movement of dislocations leading to the dispersion-hardening and the presence of compressive residual stresses in the nitrided layer. The presence of alloy nitrides in the iron matrix and the formation of solid solution due to the nitrogen solution in Fe lattice cause some structural misfit resulting in compressive residual stresses in the nitrided layer [20].

The effective case depth of nitrided specimens based on 10% Hv above that of the core hardness is given in Table 2 together with the details of applied heat treatment. It can be seen in Fig. 6a and b that the case depth increases with increasing ion nitriding time at both temperatures because of the increased nitrogen diffusivity as indicated in some earlier studies [5,6] and the generation of nitrided case is much faster at 540 °C than 500 °C in both QT AISI 4340 steel and hot-rolled AISI 4340 steel. The obtained case depth of QT AISI 4340 specimen is a few thicker than that of the hot-rolled AISI 4340 specimen at all ion nitriding conditions, as shown in Fig. 6a/b and in Table 2. The difference in nitrided depth should be due to the nitrogen transport processes inside the substrate.

The statistical evaluation of fatigue test data is given in Table 3, in which the extreme values of endurance limit satisfying 95% confidence limit are given together with the average values for all conditions of heat treatments. First improvement in fatigue properties and increase in hardness were obtained from quenching and tempering of hot-rolled AISI 4340 steel. The fatigue strength of hot-rolled AISI 4340 steel was increased from 350 MPa to 534 MPa by quenched-tempering and this improvement was about 52.5% (Table 4). It is believed that significant improvements in fatigue properties are obtained with ion nitriding and this improvement is attributed to increasing surface hardness and residual stress distribution [14–16] or increasing case depth [4,5,9–13]. In the first model, improvement in fatigue behavior is attributed to alterations of the surface properties and some other causes, as follows. The formation precipitate particles can hinder dislocation motion and, therefore, slip band penetrates through the nitrided layer. A diffusion layer, which is formed during ion nitriding process, causes compressive residual stress at the surface. 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 it causes the improvement of fatigue life [15]. In 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 more heavily applied bending stress will be required at the surface to create a

Table 2
Heat treating details and structural properties of AISI 4340 steel.

Treatment details	Surface hardness[a] ($Hv_{0,2}$)	Effect of ion nitriding on max surface hard(%)	Case depth[b], t (μm)
Hot-rolled[c]	210	–	–
Hot-rolled and ion nitrided at 500 °C for 2 h	448	113	280
Hot-rolled and ion nitrided at 500 °C for 4 h	456	117	285
Hot-rolled and ion nitrided at 500 °C for 8 h	447	112	315
Hot-rolled and ion nitrided at 500 °C for 16 h	447	112	395
Hot-rolled and ion nitrided at 540 °C for 2 h	474	126	290
Hot-rolled and ion nitrided at 540 °C for 4 h	508	142	365
Hot-rolled and ion nitrided at 540 °C for 8 h	539	157	400
Hot-rolled and ion nitrided at 540 °C for 16 h	472	125	500
Austenitized 840 °C, 30 min/oil quenched,tempered at 600 °C for 1 h. (QT)[d]	315	–	–
QT and ion nitrided at 500 °C for 2 h	510	62	280
QT and ion nitrided at 500 °C for 4 h	526	67	300
QT and ion nitrided at 500 °C for 8 h	634	101	360
QT and ion nitrided at 500 °C for 16 h	609	93	470
QT and ion nitrided at 540 °C for 2 h	553	76	315
QT and ion nitrided at 540 °C for 4 h	580	84	420
QT and ion nitrided at 540 °C for 8 h	610	94	450
QT and ion nitrided at 540 °C for 16 h	549	74	510

[a] The hardness at 20 μm depth is chosen for surface hardness.
[b] The case depth is defined as the depth at which the hardness is 10% Hv above the case hardness.
[c] Hot-rolled: not heat treated before ion nitriding and received in hot-rolled condition.
[d] QT: austenitized 840 °C, 30 min, oil quenched and tempered at 600 °C for 1 h, heat treatment before ion nitriding.

sufficiently high level of stress at the case–core interface to initiate failure [11]. The S–N curves obtained from rotating bending fatigue tests are given in Figs. 7 and 8 for all nitrided and QT AISI 4340 steels and in Figs. 9 and 10, for all nitrided and hot-rolled AISI 4340 steels. Distinct fatigue limit characteristics were observed for all types of materials considered in the study, and an absolute progress in fatigue limit with increasing case depth was observed. It is clear that there is a linear relationship between fatigue strength of steel and case depth for both QT AISI 4340 steel and hot-rolled AISI 4340 steel, as shown in Figs. 11 and 12. Increase in case depth causes higher fatigue resistance, and an improvement of 91.7% in fatigue strength was developed for 16 h at 540 °C ion nitrided specimens, which have the heaviest nitrided case in the study, when the fatigue performance of QT AISI 4340 steel is assumed to be the reference. When the fatigue performance of hot-rolled AISI 4340 steel is assumed to be the reference, 110% improvement in fatigue strength was developed for 16 h at 540 °C ion nitrided specimens, which have the heaviest nitrided case in the study, while 58.8% improvement in fatigue strength was developed for 4 h at 500 °C ion nitrided specimens, which have the thinnest nitrided case depth observed for fatigue behavior. Maximum surface hardness and fatigue strength of hot-rolled AISI 4340 steel and QT AISI 4340 steel ion nitrided at different conditions are shown in Table 4 with the effects of heat treatment before ion nitriding.

Macroscopic examination of the fracture surface obtained by fatigue test shows that the fracture surface of ion nitrided specimens can be characterized into two groups. In the first group, the fatigue cracks initiate at the surface in the high stress/low cycle fatigue region and in the second group cracks initiate at the subsurface, case–core transition zone in the low stress/high cycle fatigue region. All the fractured specimens have been observed to have failed by the 'fish eye' phenomenon with fatigue cracks originating from nonmetallic inclusions in the core and at positions near the case–core interface in the low stress/high cycle fatigue region [4,9–11,14,15]. The fish eye cracks are in circular shape, which is a direct consequence of pure rotating bending type of cyclic stressing. Scanning electron microscope (SEM) fractograph of a typical subsurface fish eye crack formation is given in Fig. 13a,b and c. The specimen received QT condition 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 1.87×10^5 is shown in Fig. 13a. The specimen received hot-rolled condition was ion nitrided at 540 °C for 16 h and loaded under 730 MPa cyclic stress up to complete fracture at the failure cycle of 1.235×10^6 is shown in Fig. 13b. Higher magnification of Figs. 13a and b reveal 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 crack was developed at inclusions on the surface of specimens in hot-rolled and QT structure (Fig. 13c) and also in ion nitrided components bended in high stress/low cycle fatigue region.

The influence of ion nitriding with surface roughness condition on fatigue behavior has been investigated. Polished and not-polished QT AISI 4340 steel specimens were ion nitrided at 500 °C for 8 h. Since the surface layer roughness causes local stress concentrations, the fatigue strength reduces. When surface roughness was decreased from 0.96 μm. to 0.25 μm by polishing, the fatigue strength of QT AISI 4340 steel was increased from 458 MPa to 536 Mpa, which brings about 16.6% improvements (Table 5). It can be seen in Fig. 14 that, while the fatigue strength of polished QT AISI 4340 steel is increased from 534 MPa to 912 MPa by ion nitriding at 500 °C for 8 h, the fatigue strength of not-polished QT AISI 4340 steel is increased from 458 MPa to 855 MPa by ion nitriding at 500 °C for 8 h. The fatigue strength of both polished QT AISI 4340 steel and not-polished QT AISI 4340 steel are increased respectively 70.8% and 86.6% by ion nitriding at 500 °C for 8 h. It can be said that after ion nitriding, the increase in the percentage of the fatigue strength of not-polished QT AISI 4340 steel is higher than the increase in the percentage of the fatigue strength of polished QT AISI 4340 steel. Although the subsurface fatigue crack formation in the high cycle region is dominant in failure initiation mechanism in ion nitrided steels, surface roughness effect on fatigue strength continues in a decreasing rate. After ion nitriding, the surface roughness effect on fatigue strength of QT AISI 4340 steel was observed to decrease from 16.6% to 6.6% (Table 5). It is possible to say that the surface roughness effect on fatigue strength continues in a decreasing rate even after ion nitriding.

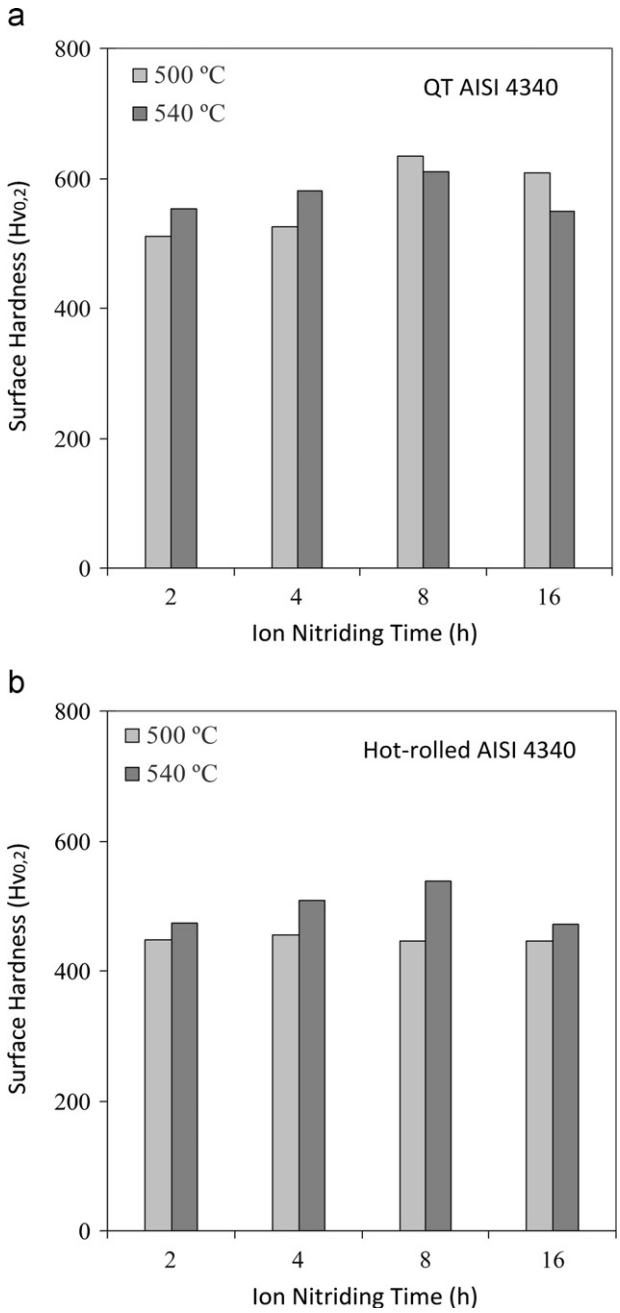

Fig. 5. Effect of ion nitriding temperature and time on surface hardness of, a. QT AISI 4340 steel and b. hot-rolled AISI 4340 steel.

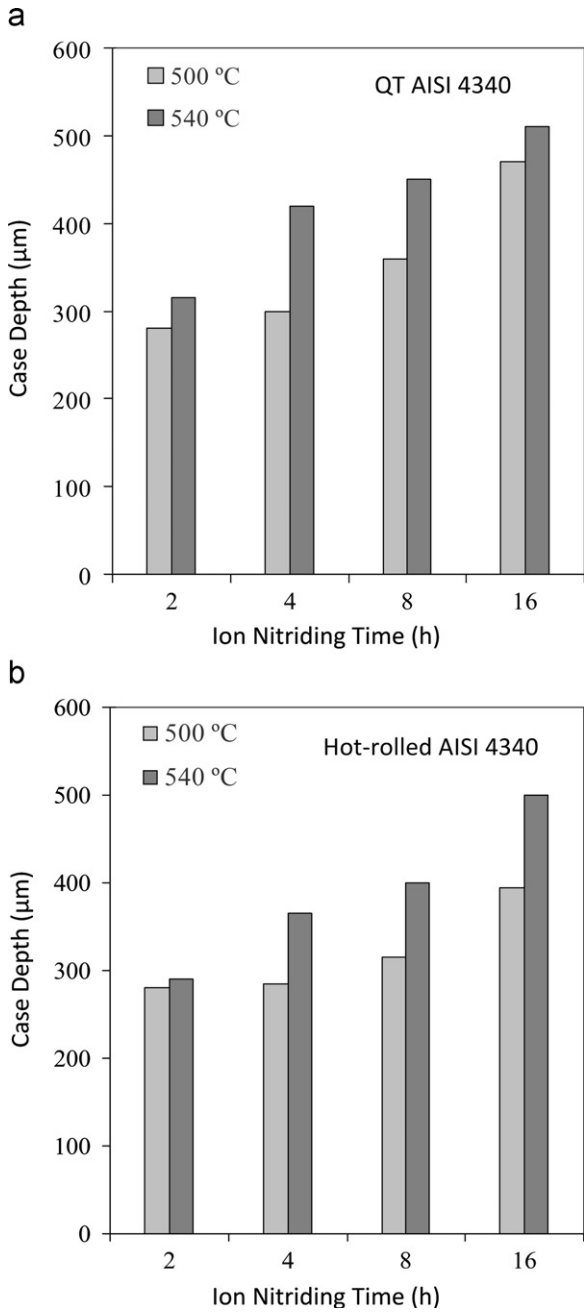

Fig. 6. The relationship between case depth and treatment time at 500 °C and 540 °C for; a. QT AISI 4340 steel and b. hot-rolled AISI 4340 steel.

The improvement of mechanical properties depends on a great variety of metallurgical and mechanical consideration. Since ion nitriding alters the chemical composition of the surface layer, the transformation kinetics and tempering responses are different in the regions of the resulting composite, which leads to a core and a case, with different microstructures and different responses to deformations. In addition, it is widely known that fatigue strength of materials increases roughly in proportion to the yield strength and tensile strength [22]. The stress–strain properties obtained from this test are listed in Tables 6, 7 and 8. It can be seen from Table 6 that the yield strength of hot-rolled AISI 4340 steel is increased from 412 MPa to 901 MPa by quenching and tempering, which brings about 118.6% improvement. The yield strength of QT AISI 4340 steel is increased in the range of 1.4%–4.3% by ion nitriding in different time–temperature conditions. This increase is in the range of 12.1%–17.0% for hot-rolled AISI 4340 steel. Although the increase in yield strength is small, it is continuously increasing by ion nitriding. The ultimate strength of hot-rolled AISI 4340 steel is increased from 636 MPa to 1008 MPa by quenching and tempering, which brings about 58.5% improvement. While yield strength is continuously increasing, ultimate strength of the ion nitrided AISI 4340 steel shows fluctuation in all ion nitriding conditions compared with the core material (Table 7). The ductility of the material has greatly reduced through both the quenching-tempering and ion nitriding process. According to Table 8, while the percentage reduction in the area is 23% for the hot-rolled AISI 4340 steel, it has reduced to only 14% by quenching and tempering. The percentage reduction in area of ion nitrided hot-rolled AISI 4340 steels has decreased in the range of 17.4%–47.8% when compared with hot-rolled AISI 4340 steel.

Table 3
Statistical analysis of fatigue test results and equations of S–N curves of AISI 4340 steel.

Treatment details	Equations of S–N curves	95% confidence limit for 10^6 cycles (MPa)	Mean fatigue strength (MPa)
Hot-rolled	log N = 46.01213 − (15.72772 × log S)	335–361	350
Hot-rolled.500.4	log N = 76.82088 − (25.79802 × og S)	536–569	556
Hot-rolled.500.16	log N = 66.72464 − (21.55558 × log S)	627–674	656
Hot-rolled.540.4	log N = 144.09591 − (49.28663 × og S)	622–641	634
Hot-rolled.540.16	log N = 92.49864 − (30.17747 × log S)	708–751	735
QT	log N = 52.35073 − (16.99379 × log S)	517–546	534
QT.500.2	log N = 64.87321 − (20.19778 × log S)	802–837	822
QT.500.4	log N = 64.22978 − (19.91905 × log S)	807–858	838
QT.500.8	log N = 60.37357 − (18.36958 × log S)	892–928	912
QT.500.16	log N = 55.03044 − (16.53242 × log S)	902–941	924
QT.540.2	log N = 68.61950 − (21.45213 × log S)	794–851	830
QT.540.4	log N = 100.91624 − (32.01631 × log S)	883–942	922
QT.540.8	log N = 68.38907 − (20.94021 × log S)	913–979	954
QT.540.16	log N = 64.54446 − (19.44699 × log S)	995–1045	1024
Not-polished [a] QT	log N = 28.42560 − (8.42772 × log S)	439–473	458
Not-polished QT.500.8	log N = 51.12692 − (15.39246 × log S)	820–879	855

[a] The surface of the reduced section was not mechanically polished before ion nitriding and received CNC turning lathe condition.

Table 4
The effect of initial microstructure and ion nitriding condition on max surface hardness and fatigue performances of AISI 4340 steel.

Heat treating details	Max surface hardness ($Hv_{0,2}$)	Effect of initial microstructure on max surface hard. (%)	Fatigue strength (MPa)	Effect of initial microstructure on fatigue strength (%)	Improvement in fatigue strength by ion nitriding (%)
Hot-rolled	210	50	350	52.5	–
QT	315		534		–
Hot-rolled.500.4	456	15.3	556	50.7	58.8
QT.500.4	526		838		57.0
Hot-rolled.500.16	447	36.2	656	40.8	87.0
QT.500.16	609		924		73.0
Hot-rolled.540.4	508	14.1	634	45.4	81.1
QT.540.4	580		922		72.6
Hot-rolled.540.16	472	16.3	735	39.3	110.0
QT.540.16	549		1024		91.7

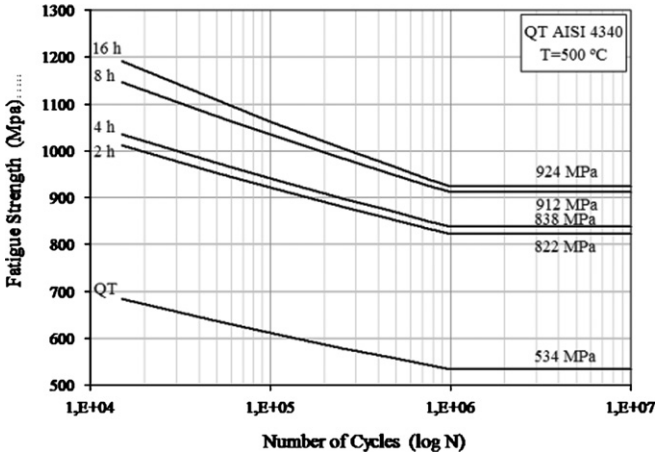

Fig. 7. Fatigue curves of QT AISI 4340 steel ion nitrided at 500 °C for 2, 4, 8, 16 h.

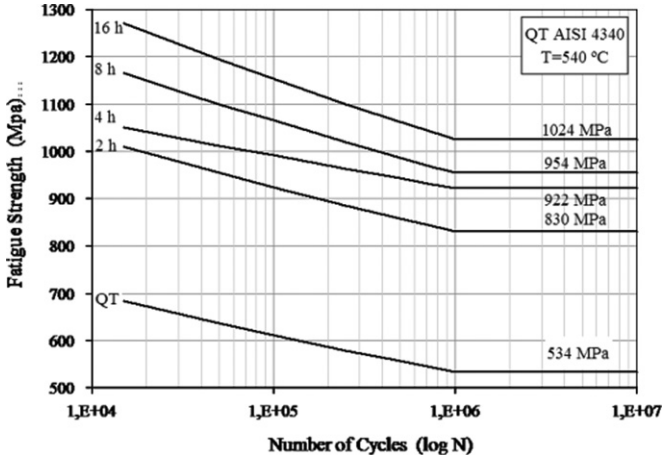

Fig. 8. Fatigue curves of QT AISI 4340 steel ion nitrided at 540 °C for 2, 4, 8, and 16 h.

This decrease is in the range of 14.3%–57.1% for QT AISI 4340 steel. This is thought to be caused by the nonhomogeneity of the ion nitrided specimen and the resulting multiaxial stresses. The stress multiaxiality is due to different Poisson's ratios when the core material behaves plastically while the case layer deforms elastically. This results in an early failure of the case layer, which in turn serves as stress concentration for the core, leading to failure without much elongation [17]. In addition, it is known that the elastic modulus E is strongly influenced by nitriding in different nitriding conditions, and the stress–strain curves are consequently different [19]. But the shape of the stress–-strain curve of the ion nitrided specimen is similar to that of the core material. Since ion nitriding process only changes the behavior of the thin surface layer, the strength properties of ion nitride specimen are still controlled by the bulk core material [17].

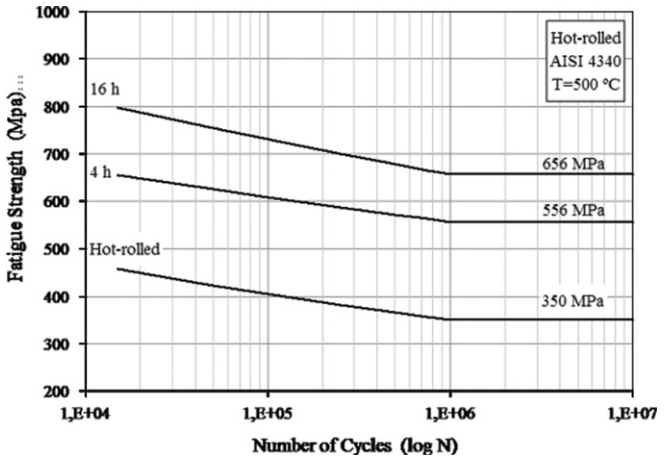

Fig. 9. Fatigue curves of hot-rolled AISI 4340 steel ion nitrided at 500 °C for 4, and 16 h.

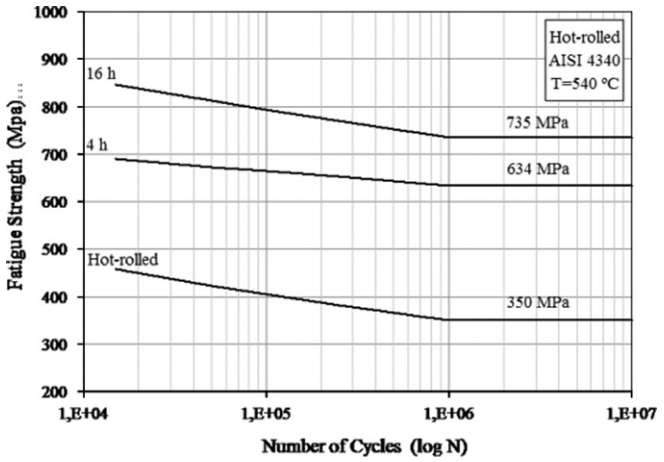

Fig. 10. Fatigue curves of hot-rolled AISI 4340 steel ion nitrided at 540 °C for 4, and 16 h.

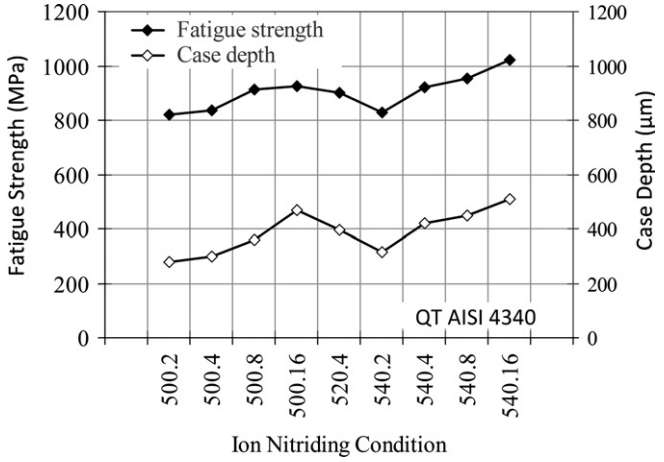

Fig. 11. Relationship between case depth and fatigue strength of ion nitrided QT AISI 4340 steel [12].

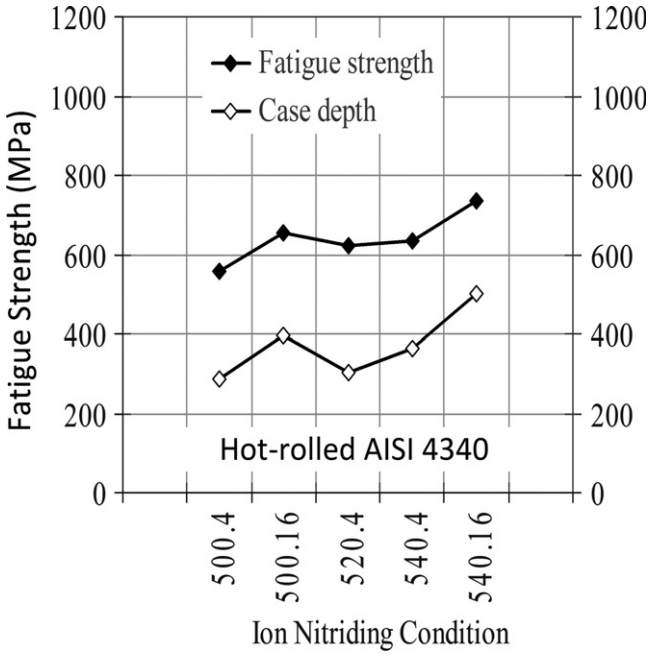

Fig. 12. Relationship between case depth and fatigue strength of ion nitrided hot-rolled AISI 4340 steel [21].

4. Conclusions

(1) The initial microstructure can influence the response of a material to nitriding. The obtained case depth of QT AISI 4340 steel is thicker than that of the hot-rolled AISI 4340 steel at all ion nitriding conditions. The quenched and tempered structure is considered to produce the optimum nitriding results.

(2) The case depth increased with increasing ion nitriding time at both 500 °C and 540 °C, and the generation of nitrided case was much faster at 540 °C than 500 °C in both QT AISI 4340 steel and hot-rolled AISI 4340 steel.

(3) Despite the surface hardness of nitrided specimen increased with increasing time and temperature, the reduced surface hardness was observed at longer nitriding time due to the formation of larger nitride precipitates. This reduction of surface hardness was more effective at 540 °C than 500 °C. Although the surface hardness of ion nitrided QT AISI 4340 steel specimen is higher than that of the hot-rolled AISI 4340 specimen at all ion nitriding conditions, the percentage increase in surface hardness of hot-rolled AISI 4340 specimen is higher than the percentage increase in surface hardness of QT AISI 4340 specimen.

(4) First improvement in fatigue properties and increase in hardness were obtained with quenching and tempering of hot-rolled AISI 4340 steel and this fatigue improvement was about 52.5%.

(5) A linear relationship was obtained between fatigue strength of steel and case depth for both QT AISI 4340 steel and hot-rolled AISI 4340 steel. Increase in case depth caused higher fatigue resistance and an improvement of 91.7% in fatigue strength was developed at 540 °C for 16 h ion nitrided specimens, which had the heaviest nitrided case in the study, when the fatigue performance of quenched–tempered steel was assumed to be the reference. When the fatigue performance of hot-rolled steel was assumed to be the reference, 110% improvement in fatigue strength was developed at 540 °C for 16 h ion nitrided specimens. Initial microstructure of AISI 4340 steel effects on fatigue properties continued even after ion nitriding at different time–temperature conditions. Although the fatigue strength of ion nitrided QT AISI 4340 steel specimen is higher than that of the hot-rolled AISI 4340 specimen at all ion nitriding conditions, the percentage increase in fatigue strength of hot-rolled AISI 4340 specimen is higher than the percentage increase in fatigue strength of QT AISI 4340 specimen.

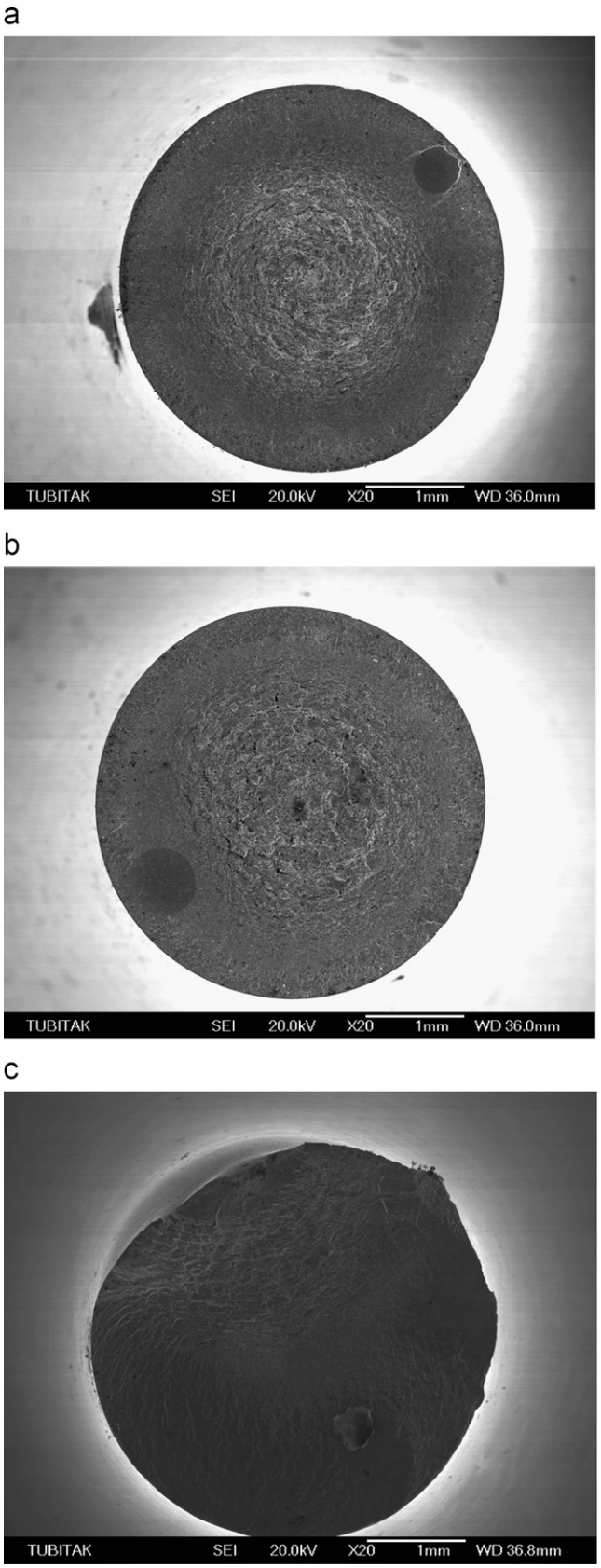

Fig. 13. SEM fractograph of a. QT and ion nitrided at 540 °C for 16 h AISI 4340, exposed to 1100 MPa cyclic stress, resulting 1.87×10^5 cycles of fatigue life, b. hot-rolled and ion nitrided at 540 °C for 16 h AISI 4340, exposed to 730 MPa cyclic stress, resulting 1.235×10^6 cycles of fatigue life, c. QT AISI 4340 structure, exposed to 560 MPa cyclic stress, resulting 4.98×10^5 cycles of fatigue life.

Table 5
The effect of surface roughness on fatigue strength of QT and ion nitride AISI 4340 steel.

Surface roughness and heat treatment conditions	Fatigue strength (MPa)	Improvement in fatigue strength by polishing (%)	Improvement in fatigue strength by ion nitriding (%)
QT-not polished (surface roughnes: 0.96 μm)	458	16.6	–
QT-polished(surface roughness: 0.25 μm)	534		–
QT-not polished-ion nitrided 500 °C, 8 h	855	6.6	86.6
QT-polished-ion nitrided 500 °C, 8 h	912		70.8

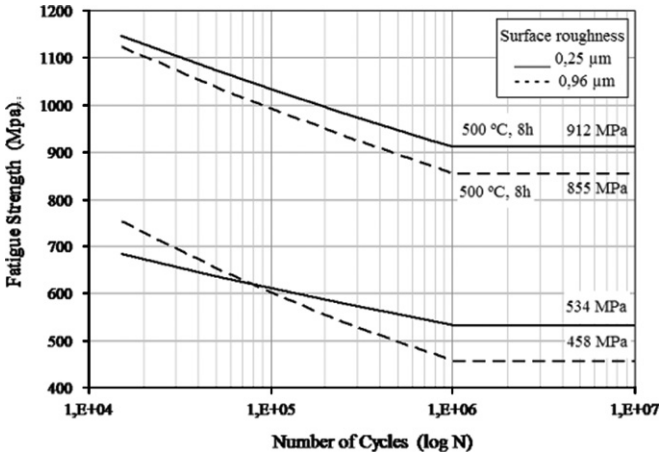

Fig. 14. The effect of surface roughness condition on fatigue strength of QT and ion nitrided at 500 °C for 8 h AISI 4340 steel.

Table 6
The effect of initial microstructure and ion nitriding conditions on yield strength ($R_{0,2}$) of AISI 4340 steel.

Ion nitriding condition	$R_{0,2}$ in hot-rolled condition (MPa)	$R_{0,2}$ in QT condition (MPa)	Increase in $R_{0,2}$ by QT (%)	Increase in $R_{0,2}$ by ion nitriding of hot-rolled AISI 4340 (%)	Increase in $R_{0,2}$ by ion nitriding of QT AISI 4340 (%)
AISI 4340-not nitriding	412	901	118.6	–	–
500 °C,2 h	462	914	97.8	12.1	1.4
500 °C,4 h	468	920	96.5	13.6	2.1
500 °C,8 h	470	931	98.0	14.1	3.3
500 °C,16 h	471	940	99.5	14.3	4.3
540 °C,2 h	466	920	97.4	13.1	2.1
540 °C,4 h	472	922	95.3	14.5	2.3
540 °C,8 h	481	927	92.7	16.7	2.8
540 °C,16 h	482	936	94.1	17.0	3.8

(6) Macroscopic examinations of the fracture surface of ion nitrided specimens show that cracks initiate at the subsurface, case–core transition zone by the 'fish eye' phenomenon in the high cycle/low stress region.
(7) When surface roughness causing local stress concentrations decreased, the fatigue strength of quenched–tempered AISI 4340 steel increased. Both mechanically polished AISI 4340 steel and received CNC turning lathe condition AISI 4340 steel specimen's fatigue strength increased by ion nitriding and the

Table 7
The effect of initial microstructure and ion nitriding conditions on ultimate strength (R_m) of AISI 4340 steel.

Ion nitriding condition	R_m in hot-rolled condition (MPa)	R_m in QT condition (MPa)	Increase in R_m by QT (%)	Changing in R_m by ion nitriding of hot-rolled AISI 4340 (%)	Changing in R_m by ion nitriding of QT AISI 4340 (%)
AISI 4340- not nitriding	636	1008	58.5	–	–
500 °C, 2 h	647	1003	55.0	+1.70	−0.49
500 °C, 4 h	642	982	52.9	+0.90	−2.57
500 °C, 8 h	633	987	55.9	−0.47	−2.08
500 °C, 16 h	646	1001	54.9	+1.50	−0.70
540°C, 2 h	639	995	55.7	+0.47	−1.28
540°C, 4 h	635	980	54.3	−0.15	−2.70
540°C, 8 h	629	979	55.6	−1.10	−2.80
540°C, 16 h	607	984	62.1	−4.55	−2.38

Table 8
The effect of initial microstructure and ion nitriding conditions on the percentage reduction in area (%A) of AISI 4340 steel.

Ion nitriding condition	% A in hot-rolled condition	% A in QT condition	Decrease in %A by QT (%)	Decrease in %A by ion nitriding of hot-rolled AISI 4340 (%)	Decrease in % A by ion nitriding of QT AISI 4340 (%)
AISI 4340-not nitriding	23	14	39.1	–	–
500 °C, 2 h	19	11	42.1	17.4	21.4
500 °C, 4 h	19	11	42.1	17.4	21.4
500 °C, 8 h	17	9	47.0	26.0	35.7
500 °C, 16 h	17	7	58.8	26.0	50.0
540°C, 2 h	19	12	36.8	17.4	14.3
540 °C, 4 h	18	10	44.4	21.7	28.5
540 °C, 8 h	16	8	50.0	30.4	42.8
540°C, 16 h	12	6	50.0	47.8	57.1

increase in the percentage of the fatigue strength of not-polished QT AISI 4340 steel is higher than the increase in the percentage of the fatigue strength of polished QT AISI 4340 steel. Although the subsurface fatigue crack formation in the high cycle region is dominant in failure initiation mechanism in ion nitrided steels, surface roughness effect on fatigue strength continued in a decreasing rate. After ion nitriding at 500 °C for 8 h, surface roughness effect on fatigue strength of QT AISI 4340 steel decreased from 16.6% to 6.6%.

(8) Mechanical characteristics of AISI 4340 steel, $R_{0,2}$, R_m and % A changed with prior heat treatment quenching–tempering and ion nitriding. Since the surface hardened component is considered as a composite consisting of a high strength-low ductility case and low strength–high ductility core, mechanical characteristics can change. While the yield strength was continuously increasing and the percentage reduction in area was continuously decreasing in all nitriding conditions compared with the core material, ultimate tensile strength of the ion nitrided AISI 4340 steel showed fluctuation.

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