

# The effect of nitriding on fatigue strength of structural alloys

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## 1. Introduction

By changing surface layer properties of machinery parts it is possible to increase considerably their load – capacity. One of the ways to obtain high complex surface physical and mechanical properties of metallic materials such as hardness, wear resistance, contact fatigue, corrosion resistance and others is chemical heat treatment method – nitriding [1-5]. Also, the nitriding is one of the basic methods for increasing fatigue strength of structural metallic materials [6-14].

There are sufficiently many publications on the methods and modes of nitriding and also in using this chemical heat treatment method for increasing load – capacity of structural metallic materials [1, 3, 15, 16]. For example, ion nitriding method proved itself very well for parts such as gears, working at dynamical – loads and friction conditions simultaneously [4, 17]. The use of nitriding process for strengthening of structural metallic materials was restricted by some shortages: toxicity of some technological processes, comparatively not great depth of strengthened surface layer (thoroughly till 0.5 – 7.0 mm), duration of nitriding process and other factors.

However, the latest significant progress in elimination of these problems was made and that allowed to look with optimism at broad use of different nitriding methods for structural steels strengthening. So, the technology of nitriding stepped far forward: from old methods of nitriding in the gaseous medium to ion nitriding. At present old and new methods of nitriding are used in connection with the creation of nontoxic cyanate containing baths [18]. In the articles [1, 4, 18-20] some modern technologies of nitriding are reviewed and in the articles [21, 22] are offered new principles for the analysis of nitrided layer formation processes, which allow to achieve the controllability of diffusion process saturation of iron (steel) by nitrogen.

Mostly the influence of nitriding on wear resistance and contact fatigue of structural steels was studied [23]. Besides, structural state of the strengthened nitrided layer, its hardness and residual stresses distribution in surface layers were estimated. Although in the P. Forrest monograph [24] the data that nitriding of carbon and alloy steels increases the fatigue limit of smooth specimens up to 13 – 105 % and the specimens with stress concentrator up to 29 – 230 % are presented already, the basic regularities of static strength, low cycle fatigue and high cycle fatigue strength of structural steels after treatment by nitriding were not studied enough. However, there are not many investigations about the effect of nitrided layer thickness on characteristics of static and fatigue strength of structural

metallic materials. This work presents a short review of experimental data and the analysis of basic factors which influence on the regularity of fatigue cracks initiation and propagation in nitrided structural steels.

## 2. Static strength of structural steels after nitriding

To investigate the effect of nitriding processes on fatigue strength of structural metallic materials it is necessary to dwell briefly on the peculiarities of static mechanical properties change after nitriding, because of the existence of some empirical relationships between static mechanical properties (for example, tensile strength and hardness) and fatigue limit.

The nitriding results in substantial increase of surface layers hardness of carbon and alloy structural steels. At temperature below 590 °C during surface layer saturation of iron by nitrogen at first the phase  $\alpha$  – solid solution of nitrogen in iron, after that the layer of the phase  $\gamma$  with face – centered cubic lattice (FCC) and with ordering arrangement of nitrogen atoms in the centers of elementary cells is formed. At further saturation by nitrogen the nitriding process can be completed by phase  $\varepsilon$  formation on the surface layer with hexagonal close – packed lattice (HCP) and with ordering arrangement of atoms in a wide interval of nitrogen concentration. At slow cooling, due to variable solubility of nitrogen in the phases  $\alpha$  and  $\varepsilon$ , precipitation of the second – phase  $\gamma_{II}$  occurs and the structure of nitrided zone (from surface to core) becomes as follows:  $\varepsilon + \gamma_{II} \rightarrow \gamma' \rightarrow \alpha + \gamma'_{II} \rightarrow \alpha$  [25]. Only for the phase  $\gamma'$  and nitrogenous martensite  $\alpha'$  the high hardness of nitrided layer is explained not only by the processes of precipitation and the formation of nitrides during nitriding process but also by higher solubility of nitrogen in the ferrite alloy by transitional elements. The dissolved nitrogen results in the development of high microstresses which relaxation is burdened at lower threshold of recrystallization. At nitriding hardness of surface layer reaches 1000 HV and more [3].

Besides that thin stronger nitrided layer influences static strength characteristics of nitrided steels, also compressive residual macrostresses arising in surface layers of the metal during the process of chemical heat treatment have an effect too. However, between residual macrostress level and hardness of nitrided surface layer there is no direct correlation [26].

Despite of comparable small depths of diffusion layer, nitriding essentially influences yield strength and appreciably reduces plasticity and viscosity. The presence of hydrogen during furnace nitriding may have an influence on mechanical properties. After nitriding strength properties of carbon steel 40 increases by 142 MPa and of

alloy steel 40X decreases by 158 MPa. Plastic properties after nitriding of these steels always decrease. So, for steel 40 elongation decreases from 27.3 up to 20.6 % and relative reduction of area from 55.0 up to 37 %. For grade 40X steel elongation decreases from 20.0 up to 1.3 % and relative reduction of area from 52.0 up to 1.3 %. After soaking of nitrided specimens for 2688 hours at the temperature of 20 °C slight increase of strength and plastic properties in both steels is observed [27].

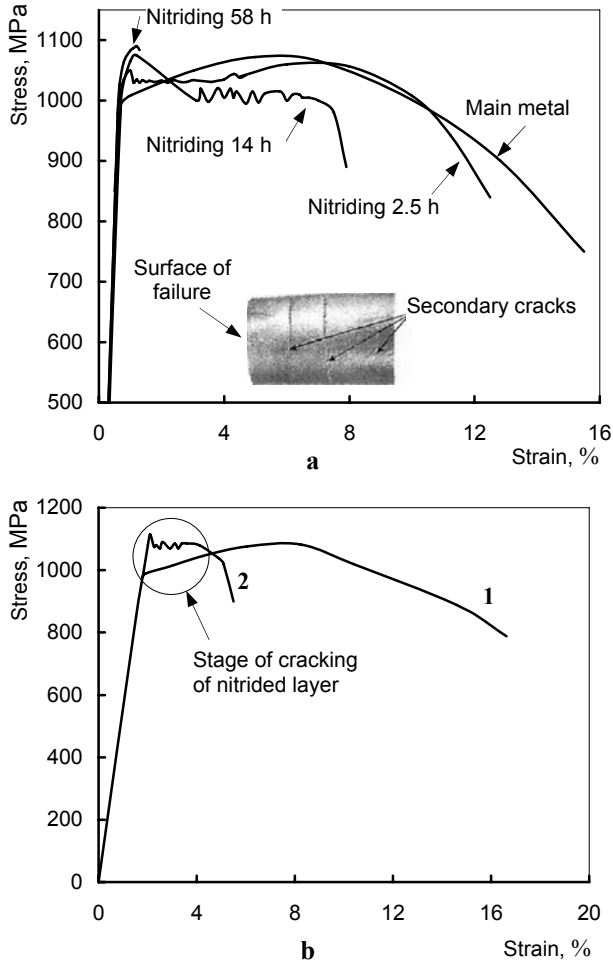


Fig. 1 Peculiarities of tensile diagrams of nitrided steels AISI 4140 (a) and 42CrMo4 (b)

In the work [6] static strength characteristics of gas nitrided cylindrical smooth specimens of low – alloy steel AISI 4140 in the conditions of static tension were investigated. Nitriding durations of the specimens after quenching and tempering were 2.5, 14 and 58 hours and total depths of diffusion layer (tempered martensite and fine – dispersion precipitations of CrN) were accordingly – 400, 600 and 1000  $\mu\text{m}$ . Vickers hardness after nitriding increased from 355 (initial state) up to 640 HV. The depth of nitriding was practically identical to all modes of nitriding and was about 1000  $\mu\text{m}$ . However, hardness of nitrided layer was higher for the specimens, which were nitrided 58 hours. Compressive residual stresses on a surface of nitrided specimens achieved the maximal value 380 MPa. In Fig. 1, a characteristic shape of stress–strain curve in an initial state and after various modes of nitriding is submitted. The initial stress–strain curve has no physical yield zone. In case of nitrided specimens the yield point is observed when the first brittle crack in nitrided layer is

formed. The subsequent multitudinous cracking occurs practically at constant stress. For specimens nitrided 2.5 hours the yield point and yield plateau appear and then the stage of strain hardening is observed. For specimens nitrided 14 hours after cracking at once there comes a stage of dishardening. By increasing the time of nitriding the material plasticity is reduced. Failure of specimens nitrided during 58 hours occurs right after the first crack occurrence in nitrided layer. Similar results on specimens from grade 42CrMo4 steel after ion nitriding were received (Fig. 1, b) [12]. Presence of yield plateau for nitrided steels is connected with the fact that cracking of nitrided layer occurs practically at constant stress.

### 3. Fatigue strength of nitrided steels

At present nitriding for increasing fatigue strength of structural metallic materials more widely is used [6-14, 28-32], although, in some cases, it results in negative effect (for example, nitriding of high – strength bearing steel SUJ2 resulted in decrease of fatigue strength [30]). The results of experimental data on the influence of nitriding on fatigue strength will be considered below.

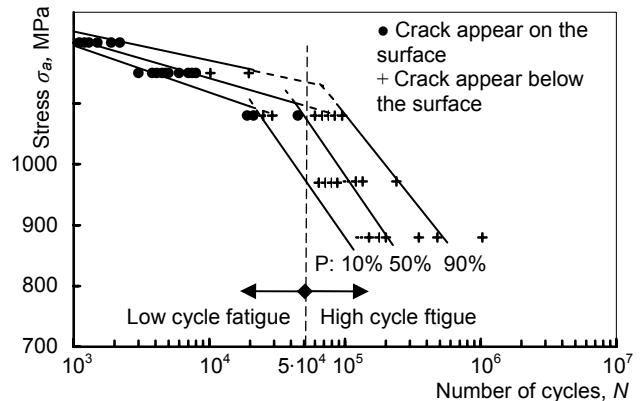


Fig. 2 Cyclic strength of nitrided grade 20MnCr5V steel specimens (the depth of nitrided layer – 0.5 mm) in transition from low cycle area of fatigue to high cycle fatigue with various probability of failure  $P$

*Low cycle fatigue.* At low cycle fatigue (base of tests up to  $5 \times 10^4 - 10^5$  cycles of loading) at which rather high stresses operate, durability to failure, basically, is connected with the processes of cracks propagation after cracking of the stronger surface nitrided layer [29]. In the work [31] it is shown that in high – strength grade 20MnCr5V steel after nitriding up to the depth of 0.5 mm the sharp bend of transition from low cycle fatigue area to high cycle fatigue appears (Fig. 2). Besides, in the area of low cycle fatigue the cracks initiate on the surface of specimens and in the area of high cycle fatigue – in near surface layer at the interface between basic metal and nitrided layer. Since the initiation of surface cracks in nitrided layer is fast, the durability of nitrided specimens at low cycle fatigue often is lower than the durability of specimens not processed by chemical heat treatment method [8]. In the work [12] it is shown that ion nitriding up to the depth of 350  $\mu\text{m}$  of specimens from alloyed grade 42CrMo4 steel reduces durability in the area of low cycle fatigue and at the same time increases fatigue limit to 30%.

Processes of microcracking on the surface of nitrided layer at low cycle fatigue are determined by a num-

ber of factors such as multiaxial stressed state of specimen (part), the kind of fatigue loading (tests in conditions of constant stress or constant strain in the cycle, uniaxial loading or bend), level of mechanical properties of nitrided layer, thickness of nitrided layer and the value and distribution of residual stresses in near surface layers (their relaxation during cyclic deformation). A major factor is fast cracking of the stronger nitrided layer at high cyclic stresses in the area of low cycle fatigue. In the work [8] an example of specimens from the normalized grade SAE 1045 steel featuring static and low cycle fatigue after nitriding is considered (with use of composition model).

*High cycle fatigue.* In the work [29] the influence of gas nitriding (at temperature 525 °C during 24 hours) and glow-discharge nitriding on fatigue strength of specimens from grade En 41B steel was studied. It was shown that various methods of nitriding result in identical values and distribution of residual stresses. At high stresses (durability is less than  $10^4$  cycles) fatigue failure is caused by microcracking on the surface of material in brittle nitrides. At lower stresses the initiation of fatigue microcracks occurs under strengthened nitrided layer at non-metallic inclusions.

Influence of soft gas nitriding on fatigue strength of structural grade SMn435 steel (wt %: 0.33C; 0.26Si; 0.15Cr; 1.34Mn; 0.015P; 0.021S) is investigated in work [14]. The obtained results were compared with the data received on grade SCM415 steel (Fig. 3). As a result of gas nitriding surface hardness of specimens from grade SMn435 steel increased from 250 up to 450 HV. The depth of the most strengthened layer was about 18  $\mu\text{m}$ . It is seen (Fig. 3) that nitriding considerably increases the durability and fatigue limit of specimens from grade SMn435 steel. However for grade SCM415 steel nitriding considerably increases the durability, but fatigue limit practically does not change in comparison with initial state. The fatigue failure of nitrided specimens begins with cracking of the strengthened near surface layer at achieving 80 % from expected durability.

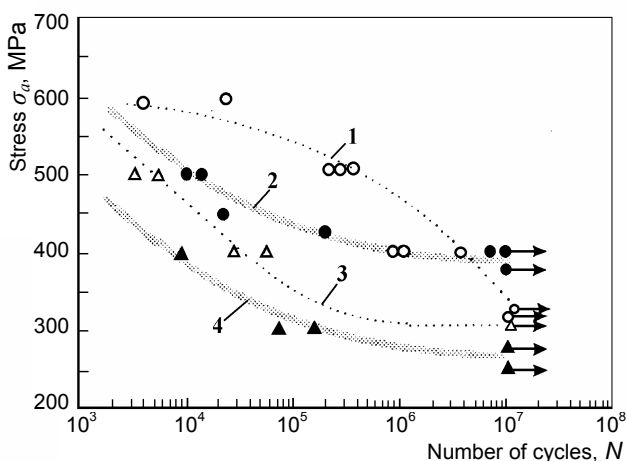


Fig. 3 Fatigue diagrams of structural steels after gas nitriding: 1 – grade SMn415 steel after nitriding, 2 – grade SMn435 steel after nitriding, 3 – grade SMn415 steel, initial material; 4 – grade SMn435 steel, initial material

In the work [6] the influence of gas nitriding on characteristics of static and fatigue strength of specimens

from medium carbon chromium – molybdenum low-alloy grade AISI 4140 steel was investigated. The fatigue strength in conditions of axial tension – compression and static tension of cylindrical smooth specimens was investigated. Nitriding durations of specimens after quenching and tempering were 2.5, 14 and 58 hours and total depth of diffusion layer (tempered martensite and fine – dispersion precipitations of CrN) were accordingly – 400, 600 and 1000  $\mu\text{m}$ . Vickers hardness after nitriding increased from 355 (initial state) up to 640 HV (Fig. 4). Although the depth of nitriding was practically identical for all modes of nitriding and was about 1000  $\mu\text{m}$ , however hardness of nitrided layer was higher for the specimens, which were nitrided during 58 hours. The compressive residual stresses on a surface of nitrided specimens achieved the maximal value 380 MPa.

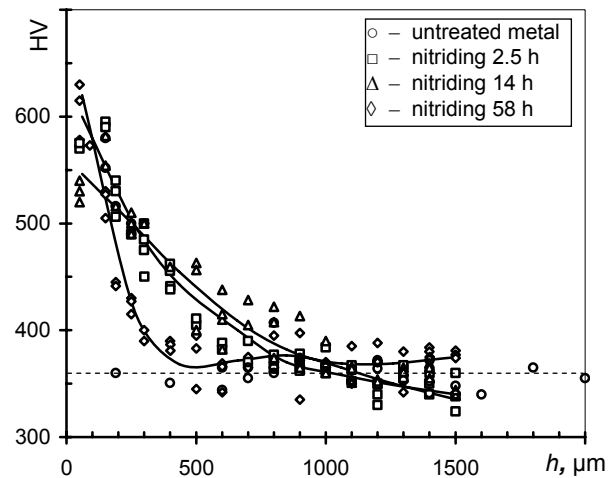


Fig. 4 Dependence of Vickers hardness of nitrided grade AISI 4140 steel specimens on layer depth

In Fig. 5 the results of fatigue tests in conditions of axial tension – compression with the frequency of 10 Hz are presented. It is seen, that nitriding essentially increases fatigue strength. The fatigue limit has increased from 500 MPa (initial material) up to 550 – 600 MPa (nitrided specimens). However, for nitrided specimens the wide scatter of experimental data of durability is observed. This wide scatter is connected with the presence of surface defects. For nitrided specimens the initiation of fatigue cracks, as a rule, occurs at the interface (nitrided layer – basic metal) at nonmetallic inclusions, and that results in formation of the surface of fatigue fracture such as “fish eye”. Thus, we see that increase of gas nitriding time, results in the increase of hardness of a nitrided layer, however plasticity of the basic metal sharply decreases what may result in sudden brittle failure. Therefore not always it is necessary to seek for maximum hardness on a surface of structural steels after nitriding. Also it is necessary to take into account operating conditions of nitrided parts because the conditions of damaging are various, for example fatigue or wear.

The process of gas nitriding is technological and well controllable which allows to receive the given parameters of diffusion layer. In the work [32] the influence of gas nitriding on fatigue strength of specimens from normalized steel 45 (wt %: 0.48C; 0.56Mn; 0.2Si; 0.12Cr; 0.1Ni) was investigated. For nitriding the installation consisting of the system of programmed composition control

of saturating atmosphere (CIP – 901) and laboratory furnace (CIIIOJI) was used. Saturating atmosphere – a mix of ammonia ( $\text{NH}_3$ ) and dissociated ammonia ( $\text{N}_2 + \text{H}_2$ ) was used. The phase composition of nitrated layer was determined by metallographic and X-ray diffraction methods. The structure of nitrated layer consisted of several phases. On the surface there is high-nitrogenous  $\varepsilon$  – phase ( $\text{Fe}_{2.3}\text{N}$ ), and under it heterogeneous structure, consisting of nitrides  $\text{Fe}_{2.3}$  and  $\text{Fe}_4\text{N}$ , which hardness 7 – 8 GPa is formed (Fig. 6). In diffusion sublayer well expressed eutectoid ( $\text{Fe}_4\text{N}$  and  $\alpha$  – the phase) was formed. The zone of internal nitriding with increased hardness after nitriding on mode 1 was 0.4 mm and after nitriding on mode 2 was 1.0 mm. The maximum hardness of nitrated layers was in the depth 30  $\mu\text{m}$  and it surpassed initial hardness of the normalized specimens from grade 45 steel more than twice (Fig. 6).

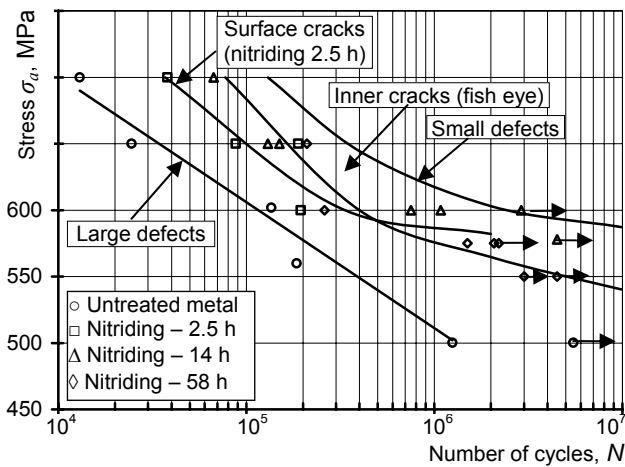


Fig. 5 Fatigue diagrams of untreated AISI 4140 steel specimens and after various regimes of nitriding

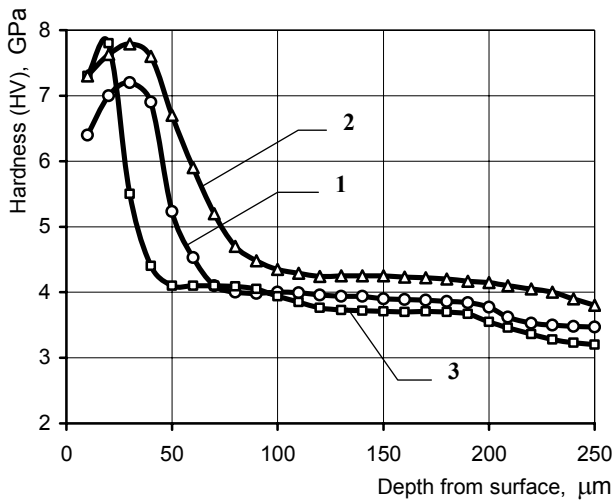


Fig. 6 The change of nitrated layer hardness of grade 45 steel after various regimes of nitriding: 1 – nitriding at 630°C during 2 hours; 2 – nitriding at 600°C during 7 hours; 3 – nitriding at 600°C during 3 hours

Three sets of specimens from grade 45 steel nitrated by three modes were investigated: 1 – nitriding at 630°C during 2 hours, 2 – nitriding at 600°C during 7 hours and 3 – nitriding at 600°C during 3 hours. In comparison, the fatigue strength of initial normalized specimens from this steel also was investigated. The fatigue tests have shown (Fig. 7) that nitriding on all three modes

sharply raises fatigue strength of grade 45 steel (curves 1-3) in comparison with the normalized state (curve 4).

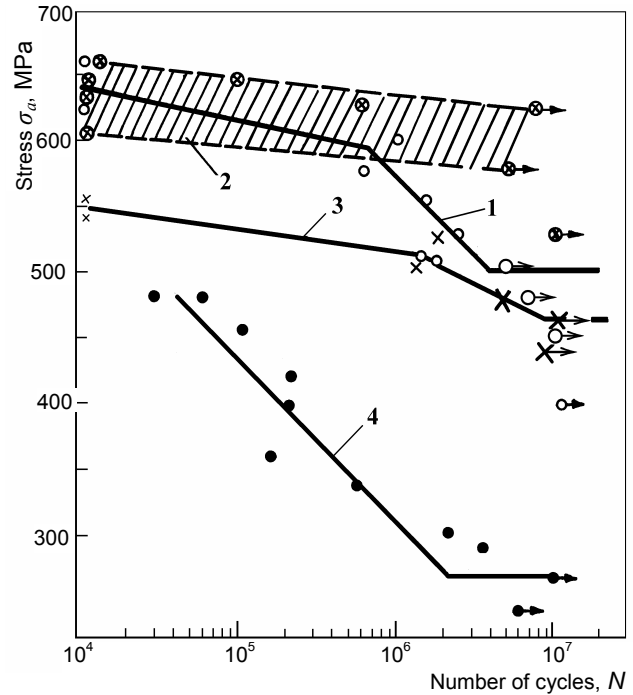


Fig. 7 Influence of nitriding regimes on fatigue limit of steel 45; 1 – nitriding at 630°C during 2 hours; 2 – nitriding at 600°C during 7 hours; 3 – nitriding at 600°C during 3 hours; 4 – initial state of steel

The fatigue limit of the specimens nitrated on the first mode (curve 1) increased up to 85% and nitrated on the second (curve 2) and the third modes (curve 3) – up to 114% and 70% accordingly (Fig. 7). Fatigue limit of steel processed on the second mode is taken according to the lowest level of nonfractured specimens because in this case the data of fatigue tests are obtained as a strip of the scatter. The specimens nitrated on the first mode at high loads fractured practically at once in the range of durability  $10^3 - 5 \times 10^3$  cycles, and then with lowering of cyclic load, the specimens fractured only after  $\sim 10^6$  cycles. This fact testifies that nitriding on the first mode results in creation of the strengthened near surface layer which well resists to fatigue load. The specimens nitrated on the second mode though show higher level (more high level) of fatigue limit, may fracture practically at once at stresses near the fatigue limit. This testifies that the metal has high sensitivity to defects resulting in the initiation and propagation of fatigue crack. The second and the third modes differ only in duration of the process (7 and 3 hours) and therefore at identical maximum level of Vickers hardness, the specimens processed on the third mode have lower depth of strengthened layer (Fig. 6). As it is seen from Fig. 7 fatigue strength of the specimens of this series is lower, than the others. The study of the fatigue fractures by scanning electron microscope showed that in initial normalized specimens from grade 45 steel the crack initiates in an usual way on the surface of the metal and after nitriding – under strengthened nitrated layer. The characteristic near the surface area of fatigue crack propagation arises which is called “fish eye”. In Fig. 8, a the scheme of fatigue fracture of nitrated specimen with near surface initiation of a crack [23] and fracture surface of nitrated specimens from grade

42CrMo4 steel tested on fatigue at low (Fig. 8, b) and high (Fig. 8, c) levels of cyclic stresses [7] are presented. At high levels of stresses the initiation of cracks occurs in the surface nitrided layer. The study of a place of fatigue crack initiation showed that structural stress concentrators inside of "fish eye" are big nonmetallic inclusions.

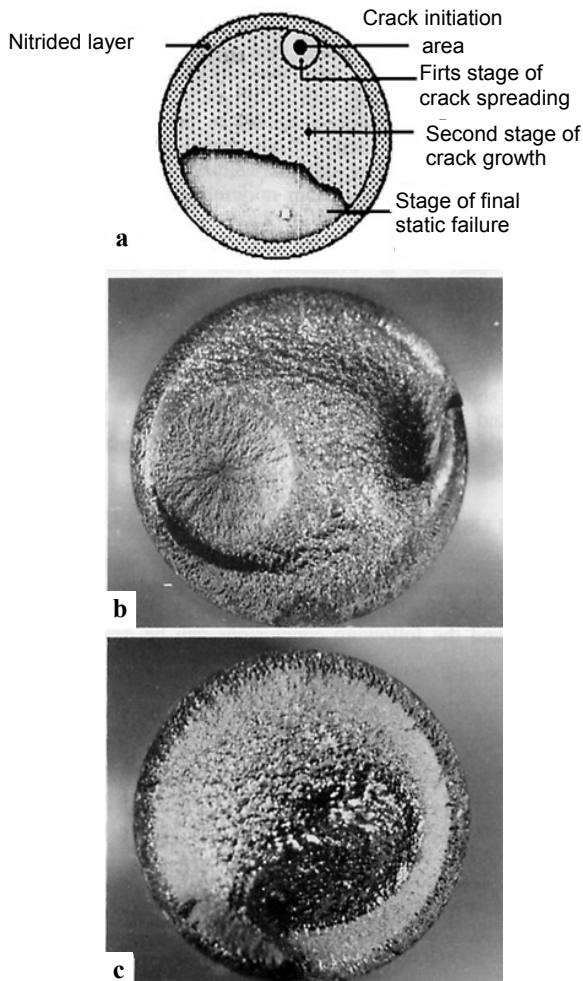


Fig. 8 Character of macrofailure of nitrided specimens: a – the scheme of fatigue fracture with near surface initiation of a crack; b – fracture surface of grade 42CrMo4 steel tested at low cycle fatigue; c – fracture surface of the same steel tested at high cycle fatigue

The ion nitriding essentially increases fatigue resistance of all structural steels due to the saturation of metal surface by nitrogen and causing in surface layer compressive stresses. So, the fatigue limit of steel such as 30XMΦA after ion nitriding increases up to 80 % (Fig. 9) [4, 16]. The study results of the influence of various phase composition of nitrided layer (after saturation in various technological mediums) on plastic resistance of grade 38XMIOA steel at alternating loads are given in Fig. 10. The greatest effect is received after combined saturation which can be explained by the more advanced diffusion zone formed at the first stage of nitriding and also more homogeneous its structure without excessive carbidic – nitridic precipitations [4, 15]. The fatigue resistance of steel 38XMIOA in solution of NaCl gives more appreciable effect (Fig. 10, b): fatigue limit is increased 5.4 times. In this case the value of  $\varepsilon$  – nitride presence on surface of

steel is high. It has high corrosive resistance. Besides, after combined saturation nitridic zone consisting of  $\text{Fe}_{2-3}(\text{N},\text{C})$  has the higher thickness than the zone consisting of  $\text{Fe}_{2-3}\text{N}$ .

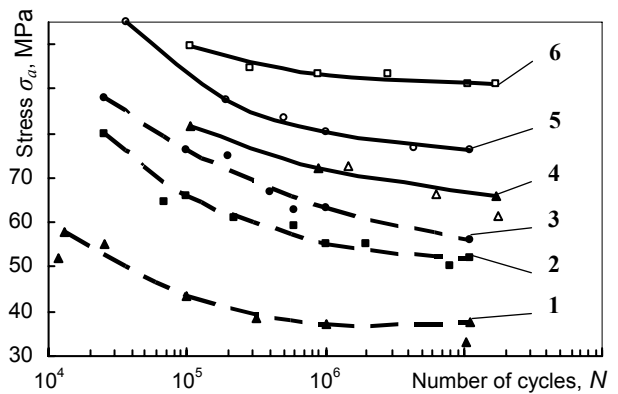


Fig. 9 Influence of ion nitriding on steels' fatigue resistance: hatched lines – after quenching and high temperature tempering; continuous line – after nitriding; 1, 4 – grade Ck45 steel (the same as grade 45 steel); 2, 6 – grade 30CrMoV9 steel (the same as grade 30XMΦA steel); 3, 5 – grade 42CrMo4 steel (the same as grade 35XM steel)

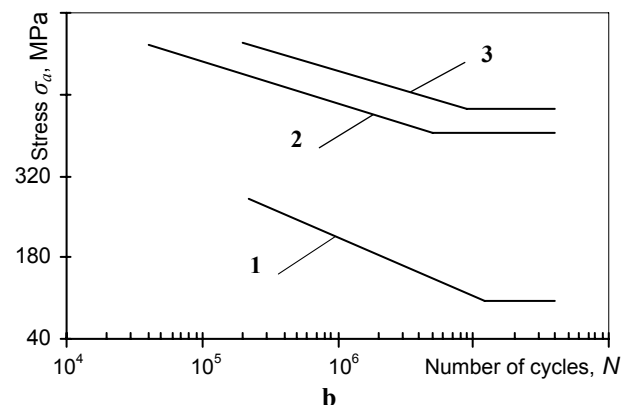
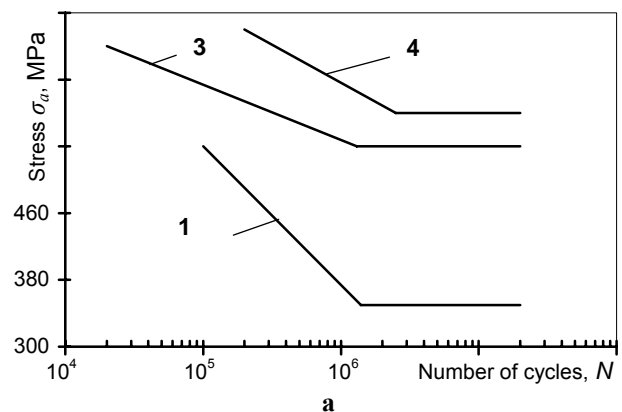


Fig. 10 Fatigue strength of grade 38XMIOA steel in air (a) and in solution of 3% NaCl (b); 1 – after quenching and high temperature tempering; after ion nitriding at 520°C temperature during 3 hours in various environment: 2 – 100% of  $\text{NH}_3$ ; 3 – 90% (wt)  $\text{NH}_3$  + 10% (wt)  $\text{C}_3\text{H}_8$ ; 4 – after dual nitriding in 10% (wt)  $\text{NH}_3$  + 90% Ar during 1.5 hours and 90% (wt)  $\text{NH}_3$  + 10% (wt)  $\text{C}_3\text{H}_8$  during 1.5 hours

In [11] the influence of various modes of low-temperature plasma nitriding on fatigue strength of cast

iron with spherical graphite CSN (ISO 1083–76) (wt %: 3.30C; 0.55Mn; 2.60Si; 0.08Cr; 0.18Ni; 0.014P; 0.024S; 0.06Mn) was investigated. During nitriding by the mode A1 the temperature in plasma was 430 °C, holding time – 24 hours, cooling in 10 – 12 hours in the gaseous medium of ammonium (purity 99.99 %). At nitriding by the mode A2 double nitriding was carried out on mode A1. Nitriding on mode B (standard gas nitriding) was carried out at temperature 510 °C during 2 hours, holding time at this temperature 9 hours, cooling 2 hours. The fatigue tests were carried out in the condition of pure bending with the frequency of 130 Hz. In Fig. 11 the data of fatigue strength of initial normalized specimens and specimens after various modes of nitriding are presented. The nitriding considerably increases the fatigue limit of cast iron: initial state – 290 MPa, mode A1 – 325 MPa, mode A2 – 408 MPa and mode B – 380 MPa. Also after nitriding by mode A2 surface of the metal had maximum Vickers hardness (Fig. 11). Authors [11] consider that both types of nitrides  $\epsilon$  and  $\gamma$  had an influence on the increase of fatigue strength after plasma nitriding.

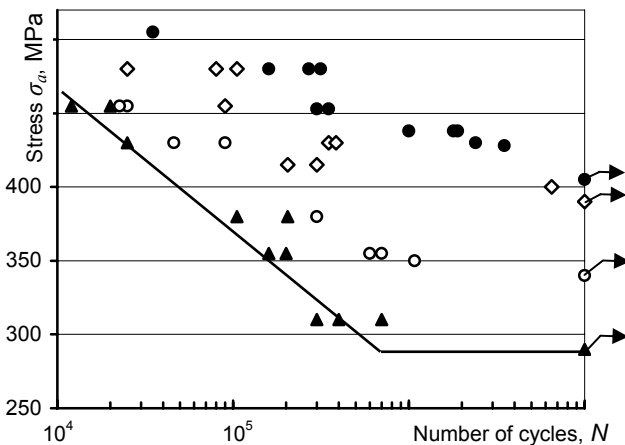


Fig. 11 Cyclic strength of untreated cast iron CSN 42 2305 (series BM) specimens and after various regimes of nitriding:  $\blacktriangle$  – BM;  $\circ$  – A1;  $\bullet$  – A2;  $\diamond$  – B

In [9] the influence of the modes of plasma nitriding on fatigue strength of structural grade 709M40 steel (En19) (wt %: 0.33C; 1.16Cr; 0.4Mo; 0.86Mn) in bending conditions rotating smooth specimens and specimens with stress concentrator (recess: width of 0.5 mm and depth of 0.2 mm) were studied. Plasma nitriding was carried out on the installation (40 kW Klöckner) at 500 °C, in the medium of gaseous mix (25 %  $N_2$  and 75 %  $H_2$ ) at 5 mbar pressure during 10 hours and 40 hours. The maximum compressive residual stresses on the surface of metal after nitriding during 10 hours were 498 MPa, and after nitriding 40 hours were 385 MPa. At the same time Vickers hardness (HV 0.1) of surface for both sets of specimens was approximately identical and was within the limit 650 – 620 HV 0.1. However, at higher distance from the surface (up to 500  $\mu$ m) hardness of nitrided layer was higher for the specimens which were longer nitrided. From the results of fatigue tests presented in Fig. 12, a it is seen that nitriding of smooth specimens during 10 and 40 hours result in sharp increase of the fatigue limit from 530 MPa (initial state) up to maximum value 1040 MPa (nitriding during 40 hours). For nitrided specimens with stress concentrator (Fig. 12, b) the fatigue limit increases from 280 MPa (ini-

tial state) up to 580 MPa (nitriding duration 10 hours). Thus, we see, that in case of specimens with stress concentrator it is impossible to aspire to big duration of nitriding because there is a danger of brittle crack occurrence at lower stresses. It is possible to note one more interesting regularity. If in the case of smooth specimens after nitriding the bend of fatigue curves is displaced aside the greater number of cycles of loading (that is connected with later initiation of fatigue cracks) in the case of specimens with stress concentrator the reverse picture is observed. This is connected with the presence of stress concentration though the stress of crack initiation increases after nitriding, but at the same time it results in faster initiation of fatigue crack at the stresses near to the fatigue limit.

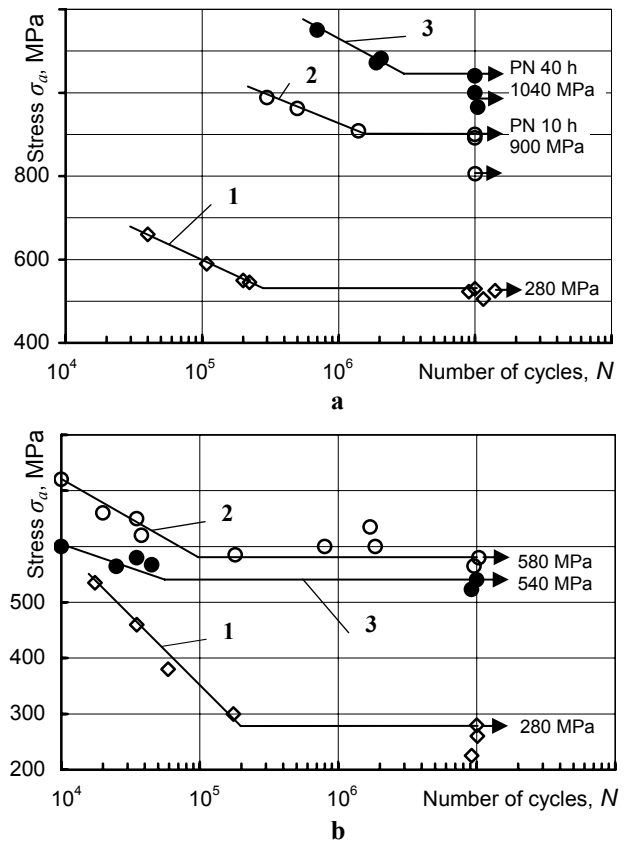


Fig. 12 Fatigue diagrams of untreated specimens of grade 709M40 steel (En19) and after various regimes of plasma nitriding: a – smooth specimens; b – specimens with concentrator; 1 – untreated specimens, 2 – nitriding during 10 h, 3 – 40 h

In [28] the influence of ion nitriding on the change of fatigue strength of grade ČSN16420.6 steel (wt %: 0.10 – 0.17C; 0.3 – 0.6Mn; 0.17 – 0.37Si; up to 0.035P; up to 0.035S; 0.6 – 0.9Cr; 3.20 – 3.70Ni) was investigated. Cylindrical specimens with the diameter and gauge length of 6.74 mm and 24 mm accordingly were used. The nitriding was carried out at the temperature of 450 °C during 20 hours in the flow of  $NH_3$  at the pressure of 200 Pa and at anode voltage – 580 V. The nitrided specimens were cooled in vacuum up to 100 °C. Fatigue limit of nitrided specimens increased from 450 MPa up to 630 MPa (increased on 40 %), and the resistance of low cycle fatigue (on the base of  $10^5$  cycles) increased from 605 MPa up to 865 MPa (increased on 43%). The initiation of fatigue cracks occurred under the strengthened surface due to

stronger surface layer and the presence of compressive residual stresses.

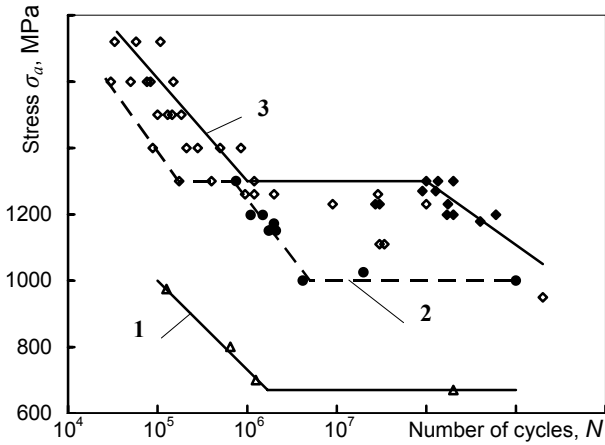


Fig. 13 Fatigue diagram of bearing grade SUJ2 steel specimens: 1 – annealing; 2 – nitriding; 3 – quenching + tempering (○ – appearance of cracks on the surface; ● – cracks below the surface)

The investigations of the influence of ion nitriding on characteristics of fatigue of bearing grade SUJ2 steel (wt %: 1.01C; 0.23Si; 0.36Mn; 0.012P; 0.007S; 1.45Cr; 0.06Cu; 0.04Ni; 0.02Mo) in conditions of circular bend were carried out in the work [10]. Ion nitriding was carried out at the temperature of 400 °C in the medium  $H_2 = NH_3$  at the pressure of 1 atmosphere. The thickness of nitrided layer was about 300  $\mu\text{m}$ . The compressive residual stresses on the surface were 540 MPa. In Fig. 13 the data of fatigue tests of the specimens in three states are presented: after annealing, quenching and tempering ( $\sigma_u = 2330$  MPa,  $\Psi = 1.3$  %) and after nitriding of the specimens which were preliminary quenched and tempered ( $\sigma_u = 1360$  MPa,  $\Psi = 0.34$  %). The fatigue limit minimum in the annealed state is observed. The specimens after quenching and tempering and after nitriding have higher level of fatigue strength and fatigue curves are stepped. The formation of steps is connected with the change of the mechanism of fatigue cracks initiation: from surface type (at high stresses) to near surface type such as “fish eye”. For the base tests  $\sim 2 \times 10^8$  cycles of loading the fatigue limits of these two sets of specimens are approximately identical ( $\approx 1000$  MPa), though in the area of limited durability cyclic fatigue properties of the specimens after quenching and tempering are higher.

In [12] the influence of ion nitriding at the temperature of 525 °C during 30 hours in the medium of nitrogen (20 %) and hydrogen (80 %) on fatigue strength of alloyed grade 42CrMo4 steel (wt %: 0.41C; 0.77Mn; 0.28Si; 0.026S; 1.02Cr; 0.019P; 0.16Mo; 0.16Ni; 0.04Al; 0.25Cu; 0.03Ti) was investigated. The depth of nitrided layer was 300  $\mu\text{m}$ . The fatigue tests were carried out in conditions of three – point bend with the concentrator such as the cut (radius in the top of cut was 4 mm). The distribution of residual stresses in surface layers is presented in Fig. 14 and fatigue curves of initial specimens (oil quenching, austenitization at 850°C and tempering at 580°C,  $\sigma_u = 1050$  MPa) and nitrided specimens ( $\sigma_u = 1070$  MPa) are presented in Fig. 15. It is seen that in the area of low cycle fatigue the durability of nitrided specimens is less than the durability of nonnitrided specimens. At high dura-

bilities to failure the durability of nitrided specimens is higher than the durability of nonnitrided specimens.

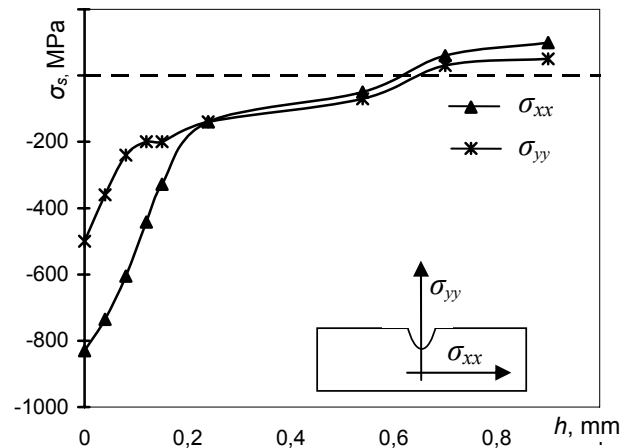


Fig. 14 Residual stress dependence on distance from surface of nitrided specimens of alloyed grade 42CrMo4 steel (the same as grade 42XMHД steel)

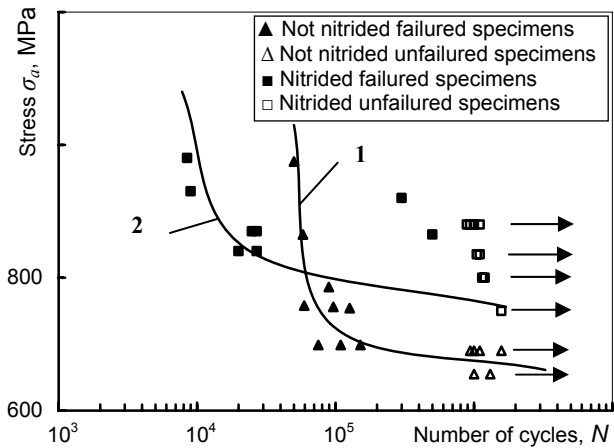


Fig. 15 Fatigue diagrams of grade 42CrMo4 steel (the same as grade 42XMHД steel): 1 – not treated specimens, 2 – nitrided specimens

The modification of surface layer by atoms of nitrogen even on small depth increases the high cycle fatigue of stainless grade X18H10T steel [33]. Ion beam treatment was carried out at the temperature of 650 K during 2 hours at the energy of nitrogen ions 1 – 3 keV and the density of ionic current was 2 mA/cm<sup>2</sup>. The increase of hardness of surface layer after such treatment was observed up to depth 15  $\mu\text{m}$ . In Fig. 16 the results of fatigue tests in the conditions of symmetric plane bending are presented. It is seen that the efficiency of ion beam treatment by nitrogen grows with the decrease of cyclic stresses and at high stresses the distinction of the durability of initial material and after nitriding is not observed. It is necessary to note, that there are many experimental data showing that after high – energy ion implantation (40 – 300 keV), when penetration depth of nitrogen ions is only tenths of micron the increase of fatigue strength of structural steels and titanium is observed [33–36]. But the analysis of such modification of surface of metallic materials by nitrogen demands special consideration.

There are not so many works in which the influence of nitriding on the regularity of fatigue cracks propa-

gation is studied [13]. It is clear that only small fatigue cracks can be considered. When the crack passes surface strengthened layer it propagates in the basic metal and general stressed state of the part (specimen) may have influence on its propagation only. In [13] the influence of soft nitriding (tufftriding process) in salt bath at the temperature of 570 °C during 90 min (the depth of nitrided layer was 1.0 mm) on the regularity of small fatigue cracks propagation in the plane specimens of low carbon steel (0.12 % C) with V – notch and thickness of 4 mm was investigated. It was shown, that propagation speed of small fatigue cracks in the nitrided layer is slowed down in comparison with annealed state. Besides the level of threshold factor of stress intensity  $K_{th}$  grows.

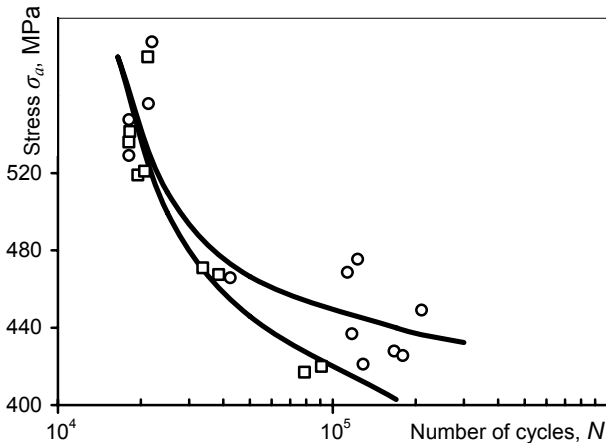


Fig. 16 Cyclic durability of grade 08X18H10T steel: (□) – untreated; (○) – nitrogen ion implantation at 650 K

*Influence of nitriding on corrosion fatigue.* Also, for the increase of corrosion fatigue of steel products the methods of diffusion saturation of surface layers by nitrogen are used. For example, it is possible to subject by anti-corrosive nitriding any steel including simple carbon steels. Saturation process was carried out at 600 – 700 °C during 0.5 – 1.0 hours. At such mode of saturation from gaseous ammonia the continuous layer of corrosive resistance phase –  $\epsilon$  is formed on a surface of product protecting metal from atmospheric corrosion, aggressive influence of water and other corrosion mediums.

In [37] the efficiency of the application of short – time nitriding to increase the resistance to corrosive – fatigue failure of medium carbon steel was studied. The investigations were carried out on smooth and notched specimens with working part diameter of 8 mm at cyclic pure bending with rotation. The nitriding was carried out in dehydrated and cleared ammonia during 7 hours at 550 °C and the degree of ammonia dissociation was about 30 %. At nitriding modes the depth of the layer was 45 – 55  $\mu$ m and Vickers hardness was about 6100 MPa. It is stated that nitriding results in the increase of fatigue limit of specimens from grade 45 steel approximately by 40% and sharp increase (almost 5 times) of conditional limit of corrosion fatigue. The surface of nitrided specimens after fatigue tests in the corrosion medium has not changed colour and products of corrosion were not revealed on it. The increase of fatigue resistance of nitrided steel in the corrosion medium occurred not only due to the corrosive layer creation on a surface of specimen interfering with fracture but also due to formation in it compressive residual stresses which maximum value was about 300 MPa. In details, the influ-

ence of nitriding on the resistance to corrosion fatigue is given in the monograph [37].

### 3.1. Major factors influencing fatigue strength increase of nitrided structural steels

*Influence of a layer thickness.* As a result of nitriding in structural steels in near surface layers of the metal a complex structure with increased hardness is formed. This results in essential increase of fatigue limit which in physical sense corresponds to the stress of fatigue crack initiation. The stronger surface layer (formed after nitriding) is a barrier to dislocations on the surface and this increases the stress of fatigue cracks initiation and fatigue limit [38].

There is inconsistent data on the influence of stronger nitrided layer thickness on fatigue limit. In [26] fatigue limit of structural steels grows with the increase of nitrided layer depth. For the parts without stress concentration the growth of fatigue limit is observed only at the relationship of the layer thickness ( $x$ ) to radius ( $R$ ) equal 0.1 – 0.2. At the presence of stress concentrators the maximal value of fatigue limit is achieved at  $x/R = 0.001$ . At further increase of the layer thickness the fatigue limit either does not change or decreases (on 10 – 20 %) due to the decrease of compressive stresses in the layer and increase of tensile stresses in a core and dishardening core. The failure begins under nitrided layer [2].

There is data which shows that the fatigue limit grows with depth of nitrided layer [24]. However in [39] is asserted that this regularity basically is due to the influence of the size of a small specimen. If the depth of nitrided layer makes significant part of the radius of a specimen, the failure occurs between the strengthened layer and the core where the stress is obviously lower. For big parts such strengthening will give insignificant effect. The author of [39] considers that the effect of various treatments should be compared according to stresses in the zone of failure. In conformity with this, in [39] it is shown that for chromium – molybdenum steel a thin nitrided layer (1.0 mm formed during 10 hours at 485 °C) gives almost the same fatigue resistance as a thick layer (3.56 – 5.1 mm, formed during 72 hours at 485 °C) and the first treatment for parts was recommended [24].

After nitriding additional treatment of nitrided layer is required quite often. Grinding to the depth of 0.05 mm (when common thickness of the layer is 0.4 – 0.5 mm) does not decrease the fatigue limit. The deeper grinding decreases the fatigue limit. The cut which depth exceeds the strengthened layer thickness, removes the strengthening caused by nitriding. The fatigue limit of nitrided products may be increased to 15 – 20 % using rollers [2]. Thus, it is obvious, that the required depth and structure of nitrided layer, in each concrete case demands careful analysis, depending on nitriding technology, scale factor, steel grade and concrete conditions of parts exploitation.

However it is possible to state, that in most cases to achieve the best characteristics of fatigue it is not necessary to seek for high nitrided layer depth but the presence of surface layer consisting mainly from nitride phase. In [40] on specimens from grade AISI4140 steel it was shown that repeated plasma nitriding after preliminary cyclic loading does not remove fatigue damage as already exist-



ing nitrided surface layer interferes in diffusion during secondary nitriding.

*Influence of residual macrostresses and microstresses.* One of major factors increasing characteristics of high cycle fatigue of nitrided steels is the presence of compressive residual macrostresses which are formed in the process of chemical heat treatment. The level of these stresses reaches up to 600 – 800 MPa [1, 2, 4, 41].

In [26] it was shown that increased fatigue strength of nitrided steel is connected not only with the presence of compressive residual macroscopic stresses (stresses of the first and second sort) but also with residual microscopic stresses (stresses of the second and third sort). The residual macroscopic stresses  $\varepsilon_{macro} = (\alpha_1 - \alpha_0) / \alpha_0$  were interpreted as the change of average size of crystal lattice ( $\alpha_1$  – average lattice size at the maximum of stress,  $\alpha_0$  – initial size of the lattice) and residual microscopic stresses  $\varepsilon_{micro} = \Delta\alpha / \alpha_1$  ( $\Delta\alpha$  – amplitude of lattice size change). In Fig. 17 and 18 the data on residual macrostresses and microstresses distribution in carbon grade Ck45 steel and alloy grade 24CrMo13 steel (grade En40B steel) after gas nitriding in an atmosphere of ammonium at the temperatures of 783 K and 853 K are presented. It is seen that the character of residual microstresses and macrostresses change with the increase of nitrided layer depth for the investigated materials is similar. The maximum residual stresses in both cases are observed at the depth of 300  $\mu\text{m}$ . The basic distinction is that if the compressive residual macroscopic stresses of nitrided steels at cyclic deformation practically are relaxed completely at the stresses which are near the fatigue limit so the residual surface microstresses in steels nitrided in salt baths do not change with the growth of the number of cycles of loading. Besides, it is known that the level of residual microstresses may even increase due to strain cyclic hardening [26, 42].

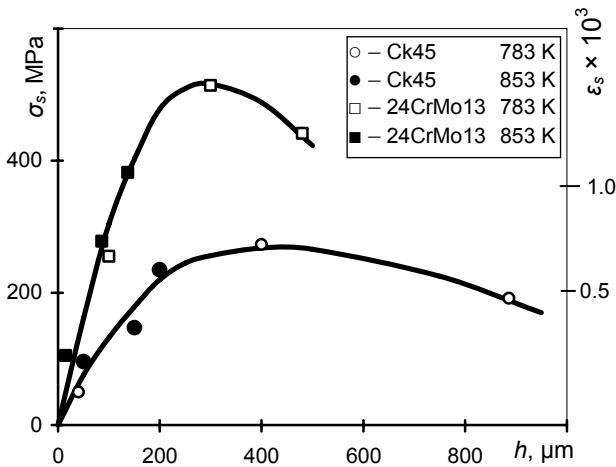


Fig. 17 Surface residual compression macrostress ( $\sigma_s$ ) and microstrain ( $\varepsilon_s$ ) dependence on the depth of nitrided layer of grade Ck45 and 24CrMo13 steels

Thus, both compressive macrostresses and microstresses arising during nitriding process of steels play the definite part in the formation of fatigue strength characteristics. It is important also that if the level of hardness of nitrided layer is not directly related with residual macrostresses, in case of microstresses the linear relationship between Vickers hardness and average surface microdeformation is observed [26].

Thus, the second important factor of fatigue limit

increase of nitrided steels is the presence of compressive residual macrostresses and microstresses in surface layers. If the first (macrostresses) during load cycling may completely relax, the level of the second (microstresses), on the contrary, may increase.

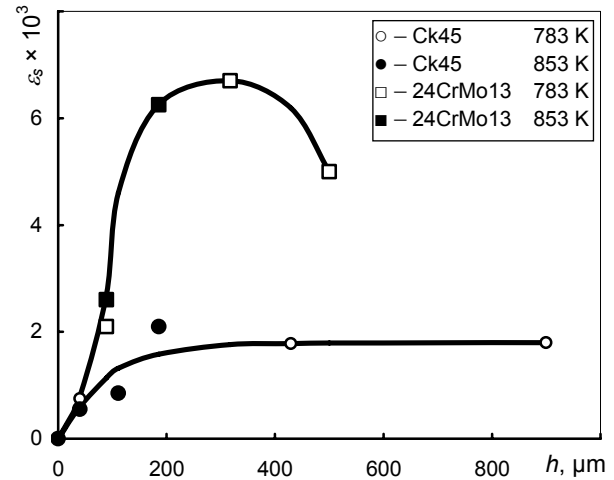


Fig. 18 Surface residual microstress ( $\varepsilon_s$ ) dependence on the depth of nitrided layer ( $h$ ) of grade Ck45 and 24CrMo13 steels

#### 4. Basic conclusions

1. Independently from nitriding method the characteristics of high cycle fatigue of the limited durability and fatigue limit of structural steels are improved. The initiation of fatigue crack, as a rule, occurs under surface layer at the interface of nitrided layer and a matrix or at non-metallic inclusions. In the area of low cycle fatigue the decrease of durability is observed more often and this is connected with stronger nitrided layer cracking at high repeated loadings.

2. In the case of nitriding of smooth specimens from structural steel the fatigue curves bend is displaced aside from the greater number of loading cycles (this is connected with later initiation of fatigue cracks), however, in the case of nitriding of the specimens with stress concentrator the reverse picture is observed. At the presence of stress concentration, though the stress of crack initiation increases as a result of nitriding but at the same time, it results in faster initiation of fatigue crack at stresses near to the fatigue limit.

3. The increase of high cycle fatigue strength is connected with the creation of stronger surface layer at nitriding, the creation of compressive residual macro and microstresses. The basic distinction is that if compressive residual macroscopic stresses of nitrided steels at cyclic deformation are completely relaxed at the stresses, near fatigue limit, the level of residual surface microstresses does not change with the growth of the number of loading cycles or even increase due to strain cyclic hardening.

4. The hardness of nitrided layer is not directly related with residual macrostresses. Also the increase of fatigue limit is not always directly related with the thickness of nitrided layer. The efficiency of chemical treatment of nitriding (with the purpose to increase fatigue strength) depends on many interconnected factors such as technology of the process, chemical composition of the steel, level of residual macro and microstresses, hardness, depth and

structural composition of nitrided layer.

### Acknowledgement

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#### AZOTINIMO ĮTAKA KONSTRUKCINIŲ LYDINIŲ NUOVARGIO STIPRIUI

##### Reziumė

Šiame straipsnyje pateikta eksperimentinių duomenų apžvalga apie skirtingų įazotavimo metodų įtaką konstrukcinių plienų cikliniam patvarumui, analizuojama įvairių faktorių įtaka nuovargio įtrūkių atsiradimui ir plitimui įazotintose konstrukcinėse metalinėse medžiagose. Tyrinėtas įazotintų konstrukcinių plienų statinis stipris, mažaciklis ir daugiacykliškas nuovargis.

Tyrimų rezultatai parodė, kad, nepriklausomai nuo panaudoto įazotavimo metodo, konstrukcinio plieno patvarumas daugiacykliam nuovargiui (sąlyginė patvarumo riba ir nuovargio riba) pagerėja, o patvarumas mažacykliam nuovargiui dažniausiai pablogėja. Konstrukcinio plieno ciklinio patvarumo padidėjimas daugiacyklio nuovargio srityje yra susijęs su tuo, jog po įazotavimo padidėja paviršinio sluoksnio stipris bei atsiranda makro ir mikro liekamieji gniuždymo įtempiai paviršiniame sluoksnyje. Ciklinio patvarumo sumažėjimas mažacyklio nuovargio srityje yra susijęs su tuo, jog veikiant didelėms kintamoms apkrovoms įazotintas sluoksnis sutrūkinėja.

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#### THE EFFECT OF NITRIDING ON FATIGUE STRENGTH OF STRUCTURAL ALLOYS

##### Summary

This article presents a review of experimental data of the effect of different nitriding methods on fatigue strength of structural steels, and the analysis of basic factors influence on regularity of fatigue cracks initiation and propagation in nitrided structural metallic materials. The basic regularities of static strength, low cycle fatigue and high cycle fatigue strength of structural steels after treatment by nitriding were studied.

The results of this study have shown that independently from a method of nitriding the characteristics of high cycle fatigue of the limited durability and fatigue limit of structural steels are improved. In area of low cycle fatigue more often decrease of durability is observed and that is connected with cracking stronger nitrided layer at high repeated loadings. The increase of fatigue strength at high cycle fatigue is connected with creation of stronger surface layer, creation of compressive residual macroscopic stresses and microstresses after nitriding.

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В. Квядарас, В. Чюплис, А. Чюплис, Й. Вилис

#### ВЛИЯНИЕ АЗОТИРОВАНИЯ НА ЦИКЛИЧЕСКУЮ ПРОЧНОСТЬ КОНСТРУКЦИОННЫХ СПЛАВОВ

##### Резюме

В настоящей работе дан обзор экспериментальных данных по влиянию различных методов азотирования на циклическую прочность конструкционных сталей и анализируются основные факторы, влияющие на закономерности зарождения и распространения усталостных трещин в азотированных конструкционных металлических материалах. Изучались основные закономерности статической прочности малоциклового и многоциклового усталостной прочности конструкционных сталей после обработки азотированием.

Результаты исследований показали, что вне зависимости от методов азотирования характеристики многоциклового усталости (ограниченная долговечность и предел выносливости) конструкционных сталей улучшаются. В области малоциклового усталости чаще всего наблюдается снижение долговечности, что связано с растрескиванием более прочного азотированного слоя при высоких повторных нагрузках. Повышение усталостной прочности при многоциклового усталости связано с созданием при азотировании более прочного поверхностного слоя, макроscopicких остаточных напряжений сжатия и микронапряжений.

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