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Influence of nitriding temperature on surface structural characteristics and fatigue strength of steel

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1. Introduction

It is known that initial acts of plastic flow are connected to the surface layer of the material. The fatigue behaviour in comparison with static loading has unusually high sensitivity to conditions of surface layer. The large influence of condition of the metal surface on fatigue strength is caused by earlier damage of the surface layer (in comparison with other volume of metal), i. e. in this layer occur earlier accumulation of critical density of dislocations. Here at strains, which are much below yield strength of material, the plastic deformation develops in thin surface layer (the size 1 – 2 grains). Fatigue cracks generally start at the free surface of a material or near the surface and propagate across the part [1-3]. Therefore, using surface strengthening (hardening) treatments it is possible to improve considerably the resistance to the fatigue crack initiation and increase fatigue life of metals and alloys. The material surface condition has a predominant effect on high-cycle fatigue and less important for low-cycle fatigue.

One of the ways to obtain high complex surface physical and mechanical properties of metallic materials such as hardness, corrosion resistance, contact fatigue, wear resistance and others is a chemical heat treatment method – nitriding. Also, nitriding is one of the basic methods for increasing fatigue strength of metals [4-7]. By using nitriding process it is possible to produce strong and shallow case with high compressive residual stresses on the surface of steel [8-10]. Independently of nitriding methods (gas, plasma and liquid) and modes the fatigue strength of structural steels are improved [11]. However, the efficiency of nitriding (with purpose to increase fatigue strength) depends on many factors such as technology of the process, chemical composition of the steel, level of residual stresses, hardness, depth and structural composition of nitrided layer. The main goal of this research was to study the influence of gas nitriding temperature on surface structural characteristics and fatigue strength of plain carbon steel.

2. Experimental

As object of research the structural plain carbon steel (grade C45) normalized at 850°C for 30 min was chosen. Chemical composition of steel is given in Table 1.

The fatigue samples were prepared from this material had a minimal diameter of 7.52 mm and gauge length of 20 mm. The surface of samples was grinded. The fatigue tests were carried out by a rotating – beam fatigue testing machine (MUI-6000) with a frequency of load alternation 3000 cycles/min. The hardness was measured by a microhardness testing tester (PMT-3) with a load of 100 gf. The microhardness measurements were performed on grinded and polished cross sections of the samples.

Table 1

Chemical composition of steel (in wt %)

C	Si	Mn	Cr	Ni	Fe
0.48	0.20	0.56	0.12	0.10	Rest

After the fatigue tests, the fracture surface of the samples was observed on the scanning electron microscope (ISU-3). Microstructures of the nitrided samples were observed by means of an optical metallographic microscope "Neophot" after etching polished surface with 4% nitric acid solution in ethanol. All tests were done at the room temperature.

The samples of steel (C45) were gas nitrided by different temperatures (550°C, 630°C and 700°C) at constant nitriding time – 3 hours. The gas nitriding process was carried out in saturating atmosphere of ammonia (NH_3) and dissociated ammonia (2N + 6H) gases.

At the initial stage of gas nitriding process, the samples were placed in a hermetic container, blown off with ammonia and placed in a furnace. After that, the samples were heated up to the temperature of nitriding process in the saturating atmosphere. The samples were cooled by ammonia flow to 100°C.

3. Results and discussion

3.1. Hardness and microstructure of nitrided layer

The metallographic examination shows that nitrided layer consists of two layers as displayed in Fig. 1. A compound (white) layer was formed on the top surface. A diffusion layer was formed below it.

Beneath the diffusion layer, the core without any evidences of structural change can be observed. The compound and diffusion layers are considered as hardened case after nitriding [6, 7, 12]. The investigations show that the structure of nitrided layer of carbon steel consists of several phases [1, 13]. On the surface it is the phase ε - nitride $Fe_{2-3}N$. Under this phase, the eutectoid – mixture consisting of phase γ' – nitrides Fe_4N and the phase α is located. Deeper, the phase α, i. e. solid solution of nitrogen in Fe_α is found. The compound layer is composed of iron nitrides type γ' (Fe_4N) and type ε ($Fe_{2-3}N$). The thickness of compound layer and microhardness measurements of the samples nitrided at different temperatures are given in Figs. 2

and 3, respectively.

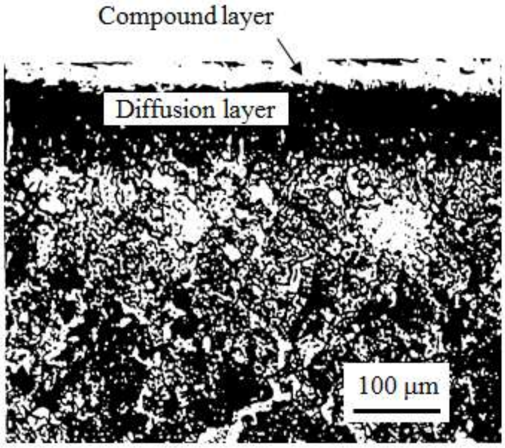


Fig. 1 Microstructure of nitrided layer: The gas nitriding at 630°C, 3 hours

Fig. 2 shows that increasing the treatment temperature from 550°C to 700°C with 3 – hour nitriding time resulted in an increase of compound layer thickness from 11 to 63 μm. Growth of the compound layer is controlled by diffusion of nitrogen through this layer [12-15].

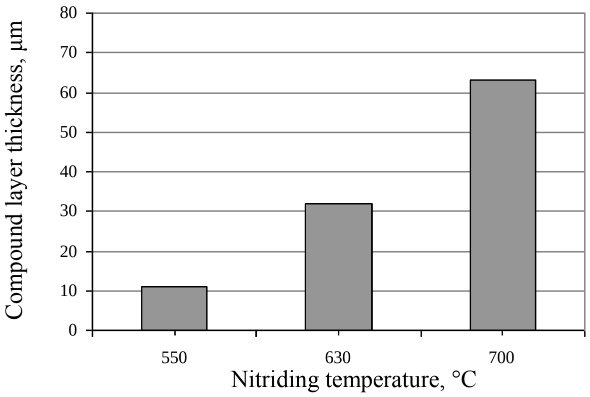


Fig. 2 Compound layer thicknesses after different gas nitriding temperatures; the gas nitriding 3 hours

Increase in treatment temperature results in an increase in the nitrogen diffusivity leading to the formation of a thicker compound layer. The results of measurements of the compound layer microhardness (surface hardness) are shown in Fig. 3.

It can be seen that the increasing in nitriding temperature from 550°C up to 630°C gives higher values of compound layer microhardness and the microhardness increases from 560 H_{100} up to 850 HV_{100}. The highest value of compound layer microhardness (850 HV_{100}) for nitriding temperature of 630°C was achieved. The further increase in nitriding temperature from 630°C to 700°C gives decreased microhardness of compound layer up to 410 HV_{100}.

Fig. 4 depicts the variation of microhardness of nitrided cases. The microhardness profiles indicate that the hardness decreases from the surface to core, since the concentration of metal nitrides decreases towards the core. The microhardness rapidly decreases in the diffusion layer. It is seen (Fig. 4) that the surface microhardness of nitrided samples is in the range of 410 – 850 HV_{100}, while the core hardness remains unchanged – 285 HV_{100}. The highest surface hardness was achieved at the treatment temperature of 630°C. In this case, the peak hardness (850 HV_{100}) is at a short distance from the surface. At the treatment temperatures 550°C and 700°C the lower values of surface microhardness were obtained. Higher treatment temperatures results in coarsening of precipitates (nitrides become larger in size and lower in number) and tempering of the core. These changes of structural characteristics lead to a lower hardness [6, 12, 14].

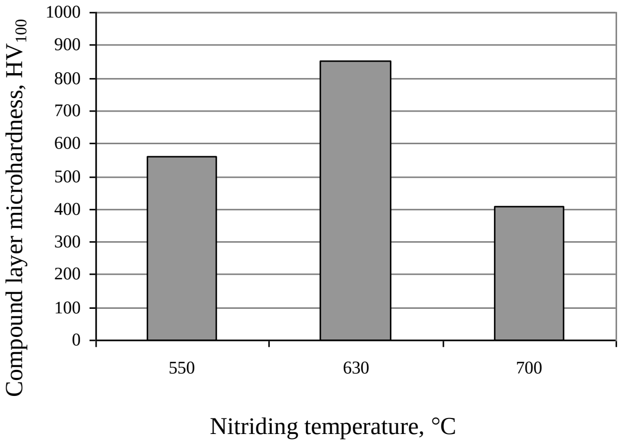


Fig. 3 Compound layer microhardness after different gas nitriding temperatures; the gas nitriding 3 hours

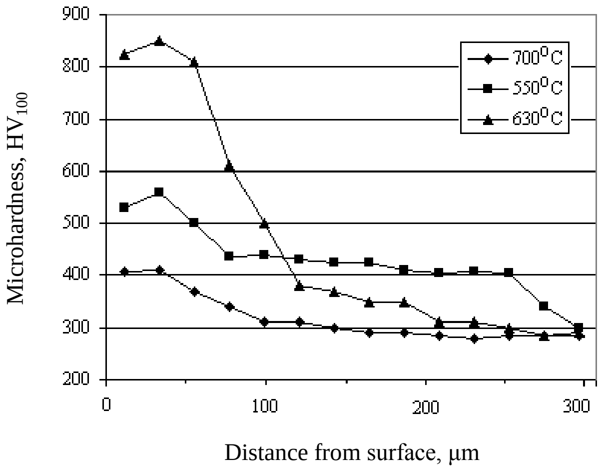


Fig. 4 Microhardness profiles of nitrided samples after different treatment temperatures; the gas nitriding 3 hours

According to the variation of microhardness from treated surface to the core of material, case depths of nitrided samples were determined.

In this study, the case depth was defined as the depth at which the hardness was 10% HV above the core hardness since the hardness value of 10% HV above the core hardness had been also used in the literature to characterize the case depth after nitriding when fatigue characteristics were regarded [7, 12].

It can be seen (Fig. 5) that higher nitriding temperatures do not produce deeper cases. The maximum case depth (280 μm) was obtained at the temperature of 550°C and the minimum case depth (65 μm) was obtained at the temperature of 700°C.

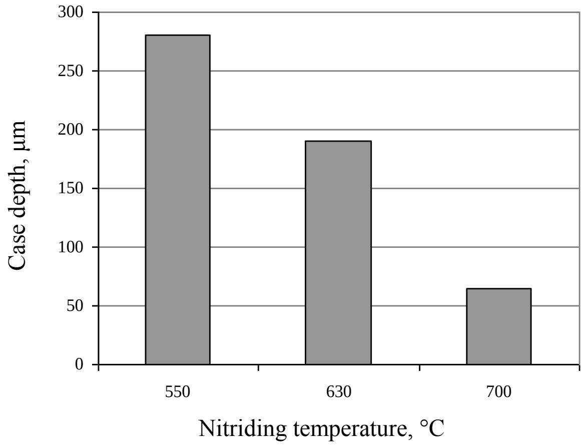


Fig. 5 The case depth after different nitriding temperatures; nitriding time – 3 hours

3.2. Fatigue tests

The S – N curves obtained from rotating bending fatigue tests are given in Fig. 6 for all nitrided and nonnitrided samples. Data of fatigue tests have shown that after nitriding by different temperatures (550°C, 630°C and 700°C) with duration of nitriding time 3 hours, fatigue strength of C45 steel varies widely.

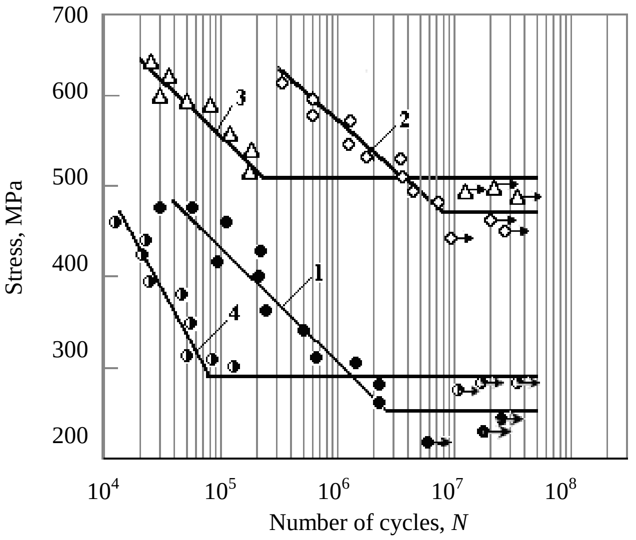


Fig. 6 Influence of nitriding temperature on fatigue strength of steel; nitriding time – 3 hours; *1* – nonnitrided steel, *2* – nitriding at 550°C, *3* – nitriding at 630°C, *4* – nitriding at 700°C

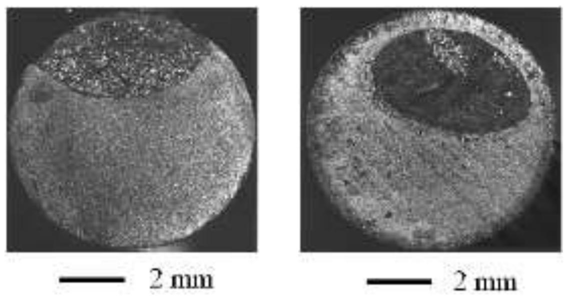
It can be seen that with increasing the nitriding temperature, the fatigue limit (σ_{-1}) of nitrided samples grows at the beginning from 260 MPa (nonnitrided steel) to the maximal value of fatigue limit (510 MPa) achieved at nitriding temperature 630°C (Fig. 6). In other words, the fatigue limit of nitrided steel increased maximum up to 96 % in comparison with the fatigue limit of nonnitrided steel. The further increase in nitriding temperature from 630°C to 700°C does not give any increase in fatigue limit of nitrided steel but on the contrary decrease value of fatigue limit occurs. The decrease of the fatigue limit at nitriding temperature 700°C occurs due to the softening of the core samples, the growth of nitride precipitates, and the reduction of compressive residual stresses in the nitrided layer [16, 17]. However the fatigue limit of nitrided samples remains higher in comparison with the fatigue limit of nonnitrided steel.

It can be seen (Fig. 6) that the maximal meaning of limited durability was achieved at nitriding temperature 550°C. The further increase in nitriding temperature from 550°C to 700°C gives a decreased limited durability of nitrided steel.

3.3. Fractography

The fractured surface of fatigue nitrided and nonnitrided samples was examined with a scanning electron microscope to assess the effect of nitriding on fatigue crack initiation and its propagation.

The fracture surfaces showed that in nonnitrided samples from grade C45 steel, the fatigue crack initiates in usual way on the surface of the metal and further progresses to the core of the sample. In the fatigue fracture, a smooth and glossy segment region was seen, where a fatigue crack initiated moving slowly towards the rough region where a complete fracture occurs (Fig. 7, a).



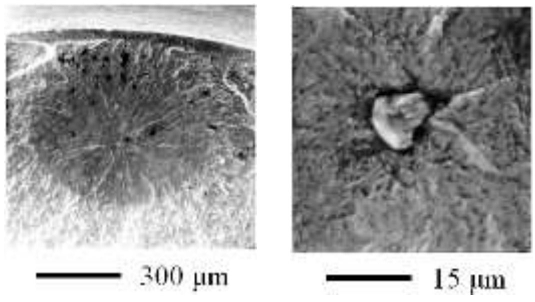
a b

Fig. 7 Fatigue fracture surface of steel samples tested at low levels of cyclic stresses: a – nonnitrided steel, σ = 280 MPa, $N = 2.2 \cdot 10^6$ cycles; b – nitrided steel, σ = 520 MPa, $N = 2.5 \cdot 10^6$ cycles

The fracture surface gloss in the crack propagation region is more expressed at the loads similar to fatigue limit, and it is more emphasized at bigger number of cycles till failure. The fatigue failure of nitrided samples in the small load region begins inside the sample in the boundary, which separates the nitrided (hardened) and unhardened metal (Fig. 7, b).

In this case, fatigue crack propagation region is a glossy ellipse spot, so-called a „fish eye" (Fig. 8, a). The examination of the place of fatigue crack initiation showed that the structural stress concentrators inside the „fish eye" are big non-metallic inclusions (Fig. 8, b). It should be noted that the same fracture mechanism for nitrided specimens has been also reported elsewhere [7, 10, 18-21].

At high levels of stresses the initiation of cracks occurs in the surface nitrided layer. Investigation of fatigue fracture surfaces showed that the samples have higher fatigue resistance when the fatigue crack initiates inside the sample. The results of experimental investigations are summarised in Table 2. It can be seen that different values of fatigue limit after nitriding by different temperatures were obtained. Independently of nitriding temperature, the fatigue limit was higher in nitrided samples in comparison with fatigue limit of nonnitrided samples.



a b

Fig. 8 Fatigue fracture surface of nitrided steel samples tested at low levels of cyclic stresses: a – „fish eye“; b – non-metallic inclusion in the centre of „fish eye“

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 [3]. The maximum improvement of 96% in fatigue strength was developed for 3 h, 630°C gas nitrided samples, which had highest (850 HV_{100}) compound layer hardness. The increase in fatigue limit after nitriding at different temperatures is related to increase in hardness of compound layer.

It can be seen (Table 2) that the case depth is not always the largest (190 μm) while the maximal value of fatigue limit (510 MPa) is achieved. The results of this study show that growth of fatigue limit of nitrided samples is not directly related to the thickness of compound layer and case depth.

Table 2

Structural and fatigue properties of gas nitrided plain carbon (grade C45) steel

Structural and fatigue properties	Non - treated	Nitriding at 550°C	Nitriding at 630°C	Nitriding at 700°C
Compound layer hardness, HV_{100}	–	560	850	410
Compound layer thickness, μm	–	11	32	63
Case depth, μm	–	280	190	65
Fatigue limit, σ_{-1}, MPa	260	470	510	396
Improvement in fatigue limit, %	–	81	96	52

In some earlier studies [8, 9, 22], it was shown that the presence of a compound layer did not have any significant effect on high cycle fatigue strength of nitrided steel since a fatigue crack initiated under a strengthened nitrided layer. Residual stresses have a major effect on fatigue properties of nitrided steels [8, 9, 11, 22].

The improvements in fatigue strength of steel are produced by introducing compressive residual stresses in surface layer and conversely, reducing in fatigue strength of steel occurs by introducing tensile residual stresses. This is because cracks tend to open and propagate fast under a tensile residual stresses but close and propagate slowly under a compressive residual stresses [22].

In previous works [1, 11, 21] it was shown that after nitriding compressive residual stresses in surface layer of steel arise up to 600 – 800 MPa. Arising of the residual stresses in surface after nitriding is related to the fact that the compound and diffusion layers have larger specific volume than the basic metal. Thus, large compressive residual stresses arise in the diffusion layer and tensile residual stresses occur in the core.

4. Conclusions

1. The results of this study showed that independently of gas nitriding temperatures (nitriding time was constant – 3 hours), fatigue strength of investigated steel (C45) was improved. The fatigue limit smooth samples after nitriding has increased maximum up to 96% in comparison with the fatigue limit of nonnitrided steel.
2. The samples nitrided at 630°C had the highest fatigue limit in comparison with samples nitrided at 550°C and 700°C respectively.
3. The improvement of fatigue strength of investigated steel after gas nitriding was related to considerable increase in hardness of the compound layer (hardness increased up to 850 HV_{100}) as well as formation of high compressive residual stresses in the surface layer.
4. It was determined that a deep case depth was not needed to achieve maximum growth of fatigue limit of nitrided steel.
5. Examination of the fatigue fracture surfaces showed that the samples had a higher fatigue resistance when the fatigue crack began to develop under the nitrided (hardened) sample surface at the centre of “fish eye” from structural stress concentrator – non-metallic inclusion.

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INFLUENCE OF NITRIDING TEMPERATURE ON SURFACE STRUCTURAL CHARACTERISTICS AND FATIGUE STRENGTH OF STEEL

S u m m a r y

In this research the influence of nitriding temperature on surface structural characteristics and fatigue strength of plain carbon steel (C45) was investigated. The samples were gas nitrided at different temperatures at constant nitriding time. The influence of such surface structural characteristics as case depth, hardness of the compound layer and case hardness on fatigue behaviour of nitrided steel was investigated. Also, the fatigue fracture surfaces nitrided and nonnitrided samples were observed by a scanning electron microscope. It was determined that the hard compound layer and high compressive residual stresses in the nitrided layer are the two major factors in improving the fatigue strength of plain carbon steel. However, the deep case depth is not necessary to improve fatigue strength of examined steel. Investigation of fatigue fracture surfaces showed that the samples have higher fatigue resistance when the fatigue crack begins to develop under the nitrided (hardened) sample surface at the central part of „fish eye“ from structural stress concentrator – non-metallic inclusion.

Keywords: fatigue strength, gas nitriding, plain carbon steel, case depth, hardness.

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