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INVESTIGATION OF THE INFLUENCE OF THE PHASE COMPOSITION AND THE NITRIDE ZONE THICKNESS ON THE WEAR RESISTANCE OF NITRIDED STEELS

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ABSTRACT:
The aim of the present work is to investigate the influence of the phase composition and the thickness of the nitride zone over the wear resistance of nitrided BH11 и BH21 steels.
The results reveal the relationship between the depth of penetration of the antibody, the phase composition of the nitride zone on the wear resistance of the nitrided steels.
It has been established that, for both steels - BH11 and BH21, the highest wear resistance is obtained after forming a mono-phase γ' nitride zone in the nitrided layer, which corresponds to the smallest depth of penetration of the antibody.
KEY WORDS:
Nitriding, Wear resistance, Tool Steel

1. INTRODUCTION

One of the basic methods for increasing the wear resistance of the details is the purposeful improvement of their surface layer properties through thermal, chemical-thermal or other types of hardening treatment [10, 11, 12].

The thermal treatment of tool steels intended for work in vacuum at high temperatures (10-10^2 Pa) is usually preferred to the treatment in chamber electrical furnaces and solved salts because of the following: the increased reliability of the tools, which is due to the non-oxidative heating and the lack of decarbonization; the degasification of the heated tools, which causes certain increase in the resilience and improvement of the plasticity of the material; decrease in the deformation of thermally treated products, due to the application of heating and cooling; increase in the wear resistance; maintaining and even improving the roughness of the tool surfaces. It has been proved that with the decrease in the coefficient of linear expansion and the increase in the Young's modulus of elasticity, the wear resistance of the materials goes up [3, 9].

In order to increase the exploitation durability of the instruments, designed to work at high temperatures, nitriding in vacuum can be used (10-10^3 Pa), while the saturation with nitrogen is being carried out in a smouldering discharge (ion nitriding). This method is a relatively new method of chemical and thermal treatment, which has been widely applied due to the advantages it offers in comparison to the classical gas and liquid nitriding, namely: speeding up the process from two to four times in dependence on the chemical structure and the composition of the steels under treatment; the highly alloyed chromine, austenite and other steels are nitrided without any substantial preliminary depassivating; the energy consumption is lower;

the maximum hardness is the same as in the process of gas nitriding, but the formation is much faster and the distribution of hardness in depth is smoother. The ion nitrided layers have higher resilience and stronger connection to the basic metal; there are no toxic substances precipitated and the modern ecological requirements have been observed [8].

The ways of providing high quality in machine-building production is directly related to the increase in their exploitation durability, which in its turn is defined to a large extent by the wear resistance of the materials.

It is known that the main factors, influencing the wear resistance, are divided into technological, constructive and exploitation ones [9, 10]. Structure, chemical, physical and mechanical properties all fall into the group of the constructive factors, while the contact scheme, macro- and micro-geometry of the fricative surfaces, and the lubrication options belong to the constructive factors. The exploitation factors include the relative velocity of sliding, the relative load, the temperature mode and the lubrication. The surface layer, formed during the friction process is characterized by an increased level of free energy, physical and chemical activity, as well as by certain mechanical properties. This is the layer, on which the mechanism of contact interaction and the level of destruction in the process of friction depend.

It is established that the level of adhesion and the coefficient of friction depend on the type of the crystal lattice. Materials with a hexagonal lattice have a reduced level of adhesion, a low coefficient of friction, and a higher level of wear resistance in comparison to the metals with BCC or FCC lattice [6, 7, 8].

Tools and details with surface hardening treatment work very frequently under the conditions of abrasive wearing out. On the other hand, the abrasive wearing out makes speeding up of the process of testing possible.

The aim of the present work is to investigate the influence of the phase composition and the thickness of the nitride zone in the nitride layer over the wear resistance of tool steels intended for work at high temperatures.

2. METHODOLOGY OF INVESTIGATION

2.1. Materials for Investigation and Modes of Thermal Treatment

Two types of tool steel intended to work at high temperatures - BH11 and BH21 – BS4659 (4Cr5MoVSi and 3Cr2W8V- GOST) have been investigated, and their chemical composition is given in Table 1.

Table 1. Chemical composition of the steels in weight percentage

Material	C	Mn	Si	Cr	V	Mo	W	S
BH 11 (4Cr5MoVSi)	0,38	0,22	0,98	4,5	0,47	1,2	-	0,006
BH 21 (3Cr2W8V)	0,30	0,26	0,18	2,7	0,29	-	8,01	0,015

Samples sizing 15x15x10 mm have been produced from the steels, and they have been thermally treated in a vacuum furnace in accordance with the modes, given in Table 2.

Table 2. Modes of thermal treatment of the steels in vacuum (P =50Pa)

Material	$t_{temp.}$ [°C]	$t_{hard.}$ [°C]	$t_{temp.}$ [°C]
BH11	780	1030	700
BH21	780	1110	700

*Footnote: *The steels have been hardened by argon gas at the pressure of 6 barr. The process of their tempering has been conducted in vacuum.*

Thus treated, the samples have been grinded by Ra - 0,32 μm and after that nitrided, following the modes, given in Table 3.

Table 3. Modes of ion nitriding

Mode №	Parameters			
	$t_{nitr.}$[°C]	P_{NH3} [Pa]	τ, [h]	$t_{temp.}$ [°C]
1	510	150	4	700
2	550	150	4	700
3	510	450	4	700
4	550	450	4	700
5	510	150	10	700
6	550	150	10	700
7	510	450	10	700
8	550	450	10	700
9	530	300	7	700

2.2. METALLOGRAPHIC INVESTIGATIONS

In order to clear out the morphological peculiarities of the nitrided layers, a methallographic analysis has been carried out.

For defining both the structure and the thickness of the obtained layers a microscope – Axioskop – has been used and metallographic pictures have been taken with the help of it. The radiometallographic structural analysis of the samples has been done by means of a Roentgen diffraction meter in cobalt radiation.

The thickness of the nitrided layer is defined by the depth, at which hardness, equal to the core plus 500 Mpa, is obtained.

Measuring the micro hardness of the obtained layers is accomplished by means of a micro hardness meter – Leitz – with a load of 100 g, by the Vickers' method. The grinded samples for investigation have been developed by a 3% solution of nitric acid in ethyl alcohol.

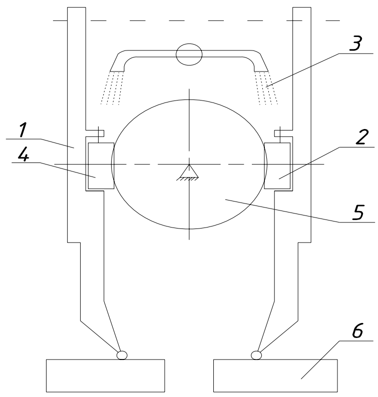


Figure 1. Sample bodies testing scheme: 1-standard sample; 2-testing sample; 3-liquid; 4-loading shoulders; 5- counter-body (roll); 6-holders

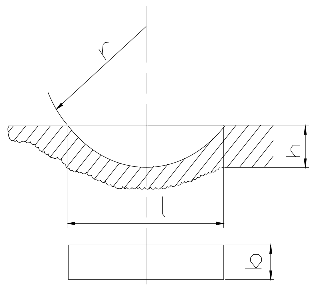


Figure 2. Scheme of the hollow obtained after testing the nitrided samples for wearing

In order to define the relative wear resistance of the sample bodies a speeded test of their abrasive wear resistance has been carried out, following the scheme: a hard roll (counter-body) – a plane. On the cylindrical surface of the roll diamond coating has been laid down, which is slided both on the plane surface of the tested sample and on the standard during a continuous pouring of a cooling liquid over – Figure 1.

The assessment of the testing results is carried out with the help of the relative wear resistance $K_V = V_{sample.} / V_{stand}$, defined as a ratio of the worn volumes – from the sample material (V_{sample}) and the standard (V_{stand}). After carrying out the testing in accordance with the scheme of wearing, a hollow (h) appears both on the surface of the nitrided sample and on the standard. Its length (l) is measured by means of a tool microscope to 0.01mm.

The following approximate relationship - $h = \frac{l^2}{8r}$ - exists between the size of the hollow and its chord, and by it the depth of penetration of the counter-body (the roll) into the nitrided layer is defined.

An experiment with the following input factors was conducted: temperature of nitriding (t_{nitr}) –X_1, ammonia presuure in the vacuum chamber (P_{NH3})-X_2, duration of treatment (τ) – X_3 and a target (output) parameter – the depth of penetration of the roll into the nitrided layer – h. The separate factors were changed in the limits, presented in Table 4.

Table 4. Factors and levels of changes

Factors Levels	t_{nitr}[°C] $_1$	P_{NH3}[Pa] X_2	τ [h] X_3
Zero level	530	300	7
Interval of varying	20	150	3
Upper limit	550	150	10
Lower limit	510	300	4

3. EXPERIMENTAL RESULTS AND ANALYSIS

3.1. Preliminary Thermal Treatment and Ion Nitrided Samples

The results from hardness measuring after the thermal treatment in the vacuum furnace are given in Table 5.

Table 5. Results from the preliminary thermal treatment

Steel	Hardness [HRC]	
	Hardening	Tempering
BH21	50	32
BH11	54	29

Through the micro hardness of the ion nitrided samples measured in depth (Figures 3 and 4) the maximum surface hardness - $HV_{0,1}$, the total thickness of the nitrided layer - δ_{total} and with the help of the metallographic microscope the thickness of the combined zone δ_{cz} were defined. The results are given in Table 6.

3.2. Wear Resistance of the Ion Nitrided Steels

The results, obtained for the abrasive wear resistance (Kv - relative wear resistance of the testing samples, h - penetration of the counter-body into the nitride zone of the nitrided layer) of the nitrided steels are given in Table 6.

Table 6. Results from the metallographic analysis and the relative wear resistance of the nitrided steels

Mode №	Steel									
	BH11					BH21				
	$HV_{0.1}$ [MPa]	δcz [μm]	h [μm]	Phase composition	Kv	$HV_{0.1}$ [MPa]	δcz [μm]	h [μm]	Phase composition	Kv
1	9980	0	8.6	α	0.58	10480	2	10	γ'	0.68
2	10480	3	6.3	γ'	0.40	10170	4	9.4	γ'	0.53
3	10170	0	10.5	α	0.75	10640	5	12	γ'+ε	0.80
4	11310	6	7.5	γ'	0.43	11000	8	9.8	γ'+ε	0.64
5	10200	1	8.5	α	0.56	10000	2	9.9	γ'+ε	0.65
6	10970	9	7.5	γ'	0.43	10970	6	7.4	γ'+ε	0.38
7	10170	4	6.4	γ'	0.40	10120	2	7.2	γ'	0.37
8	10500	7.5	5.6	γ'	0.33	10480	7	6.9	γ'	0.35
9	11000	5	7.4	γ'	0.42	10970	4	7.8	γ'+ε	0.45

The results presented in Table 6 show the positive influence of the process of ion nitriding over the wear resistance of the steels under investigation. In dependence on the chemical composition of the steels and the modes of nitrididng, the relative wear resistance changes in different limits.

The positive influence of the process of ion nitriding over the wear resistance of the investigated steels is related to the availability of alloying elements (V, Mo, Cr, W) in the hard solution and its high stability after saturation with nitrogen, as well as to the slow coagulation of the nitrided particles. This leads to increasing the resistance of the nitrided layer towards plastic deformation.

In the nitrided layer a slower process of coagulation of the carbides is observed, which is in dependence on the in-depth nitrogen concentration [3, 6].

Therefore the improved wear resistance of the surface nitrided layer is not to be considered apart from and out of the context of the complex of exploitation characteristics of the nitrided details.

On the basis of the obtained results, presented in Table 6, for the two types of steel the following importants adequate regressive equations, concerning the size of the hollow – h, obtained as a result of the penetration of the counter-body into the nitride zone of the nitrided layer, were worked out:

$$h = 8.93 - 0.7X_1 - 1.23X_3, \text{ for BH21 steel;}$$

$$h = 7.59 - 0.88X_1 - 0.90X_2X_3, \text{ for BH11 steel.}$$

The obtained mathematical model for both types of steel are with a high coefficient of correlation (R = 0.83-0.85) and adequate at a level of importance equal to 0.05.

The negative values in front of X_1, X_2, and X_3 show that with the increase in the temperature of nitriding and in ammonia pressure in the vacuum chamber, as well as with the increase in the time of treatment, the size of the hollow in the nitrided layer reduces and the relative wear resistance – Kv – of the obtained nitrided layers goes up. This is explained by the fact that this particular change in the factors related to the process of ion nitriding leads to obtaining layers with a bigger thickness of the nitrided zone, which is a two-phase one (γ'+ε).

Table 6 shows that after nitriding BH21 steel, layers with monophase γ' and two-phase (γ'+ε) combined zone are formed.

From Table 6 it can be seen that BH21 steel possesses the highest relative wear resistance Kv=0,35 after nitriding at: temperature of 550^{0}C, duration of treatment 10h and ammonia pressure in the chamber 450Pa (mode 8, Table 2). The obtained nitrided layer is with micro hardness $HV_{0,1}$ = 10480MPa and thickness of the combined zone, which is with a phase of mixed composition (γ'+ε) – Figure 3 b, c – equal to 7μm. During the testing of the samples the counter-body has penetrated at a depth of 7 μm into the nitrided zone, which is equal to its thickness. This illustrates the strong resistance against wearing, which points at the two-phase (γ'+ε) nitride zone, where the ε –phase is in a bigger amount than the γ'- phase. The clearly formed brittle two-phase (γ'+ε) zone in the sorbid structure (lower thickness - 32HRC) of the basic material, influences mainly on the relative wear resistance of BH21 steel. The even distribution of the micro hardness in-depth the nitrided layer, which is a result of this particular mode of nitriding – Figure 3a, plays an important role as well.

From Table 6 it can be seen that BH11 steel after nitriding at: temperature 550 ^{0}C, duration of treatment 10h and ammonia pressure in the chamber 450Pa (mode 16, Table 2) has the highest relative wear resistance Kv= 0,33.

The nitrided layer has micro hardness $HV_{0,1}$ = 10500MPa and the thickness of its combined zone whose phase composition is (γ'+ε) – Figure 4 b, c. During the process of testing the samples, the counter-body (the roll with diamond coating) has penetrated into the nitride zone at a depth of 5, 6 μm, which is much lower than its thickness.

We can conclude from Table 6 that new layers are formed – without combined zone or only with monophase γ' combined zone. The relative wear resistance in the nitrided layers with combined zone γ' (K_v=0.33-0.42) is much higher than in layers without nitride zone (K_v=0.56-0.75). In the nitrided layer where no new combined zone is formed the relative wear resistance (K_v=0.53-0.70) is lower. The reasons for that we can find in the morphology of forming the α-phase (diffusion zone), which has higher plasticity and lower surface thickness $HV_{0,1}$ = (9800 - 10200MPa), as well as in the type of the crystal lattice. The even distribution of the micro hardness in-depth the nitrided layer, obtained at this particular mode of treatment - Figure 4a, also plays an important role.

The penetration of the counter body h for BH11 steel is 76% of total thickness of the nitrided layer's zone, while for BH21 steel it is 100%. That shows the higher wear resistance, on which influences the nitrided (combined) zone of the nitrided layer for BH11 steel, than for BH21. This is explained with the higher brittleness of the two-phase (γ'+ε) which is in the formed nitrided layer of BH21 steel.

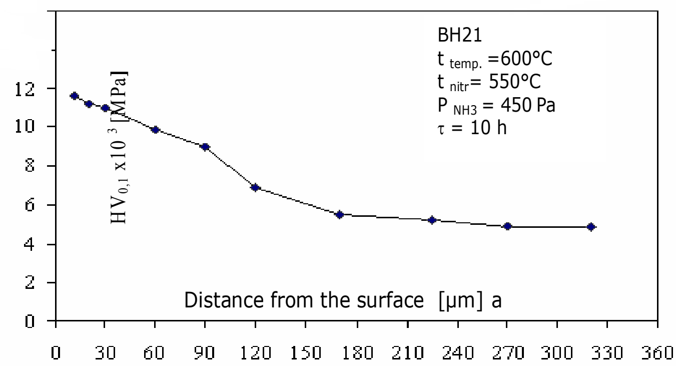
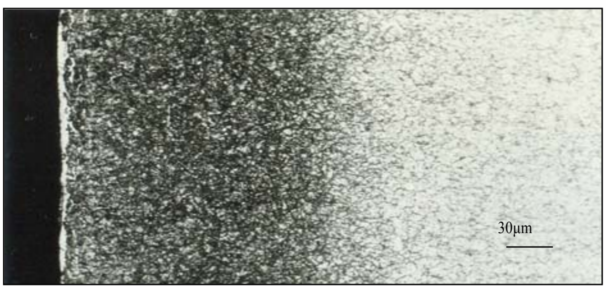
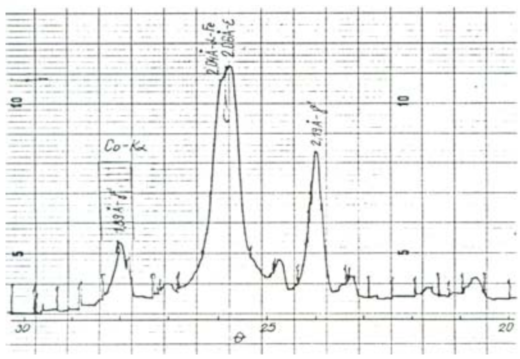
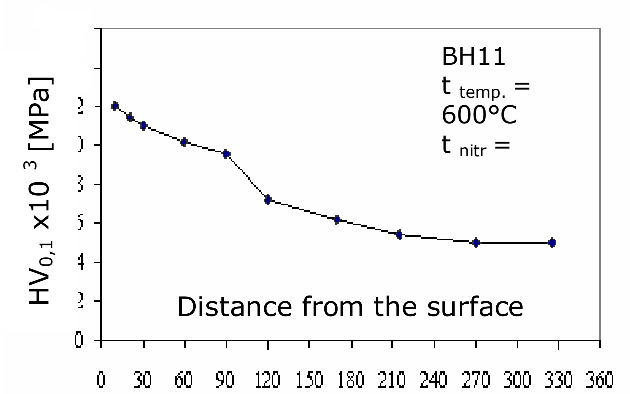


Figure 3. Distribution of the micro hardness in depth the nitrided layer - a;
b – microstructure of a nitrided layer;
c – roentgen graph of the nitrided layer of BH21 steel, nitrided at: t_{nitr} = 550°C; P_{NH3} = 450 Pa; τ = 10 h







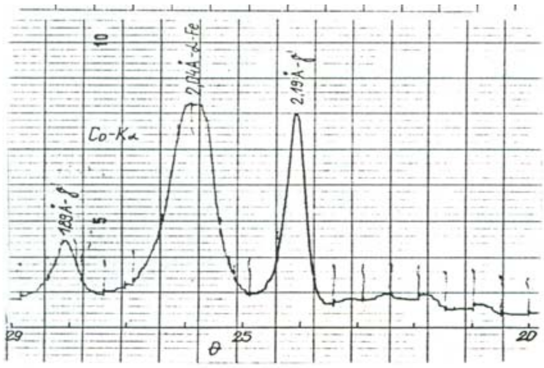


Figure 4 – Micro hardness distribution in depth the nitrided layer - a;
b – microstructure of the nitrided layer;
c – roentgen graph of a nitrided layer from BH11 steel nitrided at: t_{nitr} = 550°C; P_{NH3} = 450 Pa; τ = 10 h

4. CONCLUSIONS

- It has been proved that after ion nitriding the relative wear resistance of the steels under investigation increases, which depends on the phase composition of the surface layer. The higher wear resistance is obtained when there is a mono - γ'-phase. The relative wear resistance of the diffusion zone of steel BH11 is the lowest. At BH21 steel only combined zone (γ' or γ'+ε) is formed in the nitrided layer.
- It has been established that after ion nitriding at temperature 550°C, duration of treatment 10h and ammonia pressure in the chamber 450Pa, the relative wear resistance of BH21 and BH11 steels is the highest and approximately the same (Kv=0.35 - 0.33). The thickness of the monophase nitride zone in the nitrided layer for BH11 steel is 7.5µm, while for BH21 steel it is 7µm, and the penetration of the counter-body (the roll) spreads all over 76 % of the thickness of the nitride zone in the nitrided layer for BH11 steel, and for BH21 steel it is 100%.
- It has been proved that the relative wear resistance is directly related to the phase composition of the combined zone, formed in a troostite-martensite structure, obtained after tempering the investigated steels at 700 ^{0}C.
- A mathematical model for both steels has been drawn, which reflects the dependence between the control parameters (temperature of nitriding and ammonia pressure in the chamber, duration of treatment) of the process of nitriding and the degree of penetration of the counter-body into the nitride zone of the nitrided layer.

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