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ABOUT THE INFLUENCE OF THE PROCESS OF ION NITRIDING OVER THE CRITICAL CRACK OPENING IN BH21 STEEL

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ABSTRACT: This paper considers the influence of the process of ion nitriding over the resistance against fragile destruction of BH21 BS4659 (3Cr2W8F-GOST) steel, tempered at 700°C. The resistance of the nitride steel against fragile destruction is defined at static bending by means of the critical crack opening - δ_{ic} . The paper discusses the influence of the parameters of the process of ion nitriding (ammonia pressure in the chamber, temperature of nitriding, time of treatment) over the critical crack opening. A conclusion is drawn that after ion nitriding the resistance against fragile destruction of BH21 goes up, which is expressed by means of the parameter δ_{ic} .
KEYWORDS: ion nitriding, ammonia, resistance against fragile destruction, crack opening - δ_{ic}

INTRODUCTION

Tools for material treatment, such as prints and press forms, working under very heavy conditions, are very demanding. The requirements towards them can be satisfied by the use of steels with high content of carbide-forming elements in them δ_{ic} [1,2,3]. Most often the heat treatment of hot work tool steels is carried out in salt baths or in vacuum.

The reliability and exploitation durability of the hot work tools depend mainly on the following characteristics: destruction stability K_{ic} and critical crack opening δ_{ic} [1,2,14]. At present the criterion K_{ic} is most frequently used for comparative assessment of materials with respect to their resistance against fragile destruction [1,6,7,8]. However, its application with steels with limit of flow $R_{p0.2} < 1100\text{MPa}$ is restricted, as the plastic zone at the summit of the crack becomes quite big. In order to avoid these additional preconditions, it is necessary to use some of the criteria of the non-linear mechanics of destruction – e.g., δ_{ic} . The critical crack opening δ_{ic} is an integral characteristic of the plastic deformation and the plastic constant of the material, which explain its susceptibility to fragile destruction.

The process of nitriding is a method of increasing the hardness, wear resistance, heat resistance, resistance to tiredness and durability of the details [5,8]. The nitrided details can be regarded as multilayer materials, comprising a matrix phase and one or more phases of the thin layer, which differ in their chemical composition, in the parameters of their crystal lattice, as well as in their physical and mechanical properties. These differences lead to a state of stress, arising at the border between the matrix and the nitrided layer. In addition to the natural residual stresses of the materials, caused by compressive stresses, in the process of work also occur applied stresses, resulting from the conditions of loading. If the total stresses in an arbitrary cross-section exceed the values of the critical stresses for a given multilayer system, then the fragile destruction will start in the boundary diffusion area or in the thin layer [8]. The aim of the present work is to investigate the influence of the process of ion nitriding over the resistance against fragile destruction in BH21 steel by defining the crack opening at static bending- δ_{ic} .

MATERIALS FOR INVESTIGATING AND MODES OF HEAT TREATMENT

Hot work tool steel BH21 BS4659 (3X2B8Φ-ГОСТ) with increased heat resistance has been chosen for the investigation. The chemical composition of the above mentioned steel has been defined by equipment for automatic analysis „Spectrotest”, as shown in Table 1.

Table 1. Chemical composition of the steel

Material	Weight percentages						
	C	Mn	Si	Cr	V	W	S
BH21	0,30	0,26	0,18	2,70	0,29	8,0	0,015

Table 2. Modes of heat treatment in vacuum furnace

Material	t_{temp} [°C]	t_{hard} [°C]	HRC	t_{temp} [°C]	HRC
BH21	760	1110	49	700	32

In order to achieve the desired mechanical parameters, the samples have been subjected to heat treatment. The preliminary heat treatment has been carried out to obtain the necessary mechanical parameters and structure, what ensures both the uniform penetration of the nitrogen in depth and the favorable running of the diffusion [5].

Standard test tubes for three-point bending have been made of the steel and used for defining δ_{lc} [10,11] - fig.1. All samples have been subjected to vacuum heat treatment, following the modes given in Table 2.

The summit of the cut section on the sample has been done by means of a thread-erosive machine with a radius of approximation $R = 0,090$ mm and length 5 mm. All samples have been grinded at $R_a = 0,32$ μ m. [6,8].

Treated this way, the samples have been then ion nitrided in the installation "ION – 20", following the modes given in Table 3.

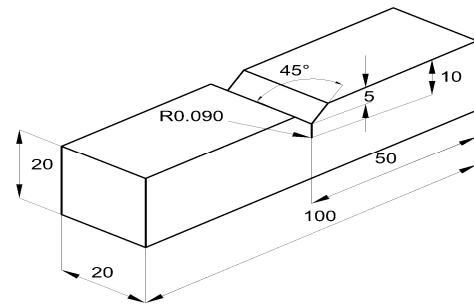


Fig.1. Shape and dimensions of the samples

METHODOLOGY OF DEFINING δ_{lc}

The samples have been tested by means of the testing machine "Instron-1343" under the following conditions: traverse velocity - 0,5 mm/min; console tensile resistant preceptor for opening with responsiveness of 2,5 μ V/mm, 10mm base and 2mm run. The balance check of both the testing machine and the preceptor for crack opening is done before testing each sample. The testing is carried out at room temperature and recording of the diagram "loading – replacement of the edges of the cut section" is made.

The calculation of δ_{lc} has been done on the basis of a standard methodology [9,10,11].

The testing has been conducted by application of the load with concentration on the face of the cut section with a crack on the opposite side. The distance between the supporting rollers is $L=4W$.

The speed of the crack opening during the testing is within 0,05 to 0,3mm/min. The diagram loading (F) – replacement (V) is automatically recorded during the tests – fig. 2.

The aim of the testing is to define the critical value of the crack opening δ_{lc} at its root at the moment of its propagation. It is defined by the record of the curve „F-V” for the moment of sudden destruction or for the beginning of crack increase, i.e., by the value of V_c – fig.2. V_c u V_{Fc} are replacements of the edges of the crack, corresponding to characteristic points from the diagram „F-V”- fig.2. The following formula is used for defining δ_{lc}

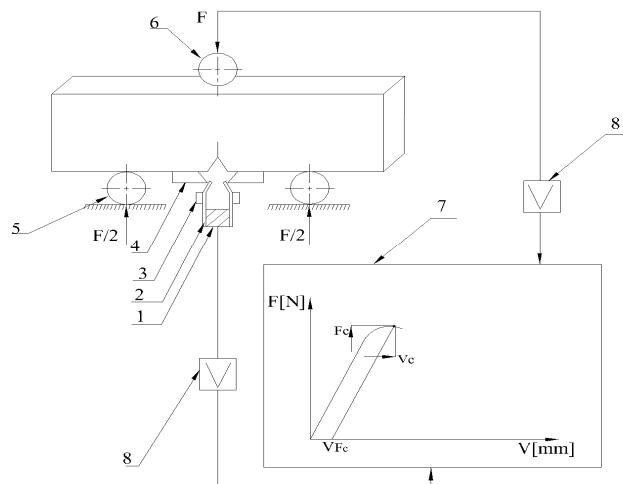


Fig.2. Scheme of the experimental setting:

1. Remote control block; 2. Elastic element; 3. Preceptor;
4. Fixing element; 5. Supporting roller; 6. Bending arbor;
7. Recording unit; 8. Amplifier

V_c u V_{Fc} are replacements of the edges of the crack, corresponding to characteristic points from the diagram „F-V”- fig.2. The following formula is used for defining δ_{lc}

$$\delta_{lc} = \frac{K^2 (1 - \mu^2)}{2 \cdot R_{0,2} \cdot E} + \frac{0,4 (W - B)}{0,4 \cdot W + 0,6 + z} \cdot V_{Fc}, [\text{mm}] \quad (1)$$

$$K = \frac{FL}{B \sqrt{W}} Y \left(\frac{B}{W} \right), Y = -1,555 (1 - 5,456 \frac{B}{W}); \quad (2)$$

where: δ_{lc} – critical opening of the edges of the crack at maximum loading; K–coefficient of intensity of the stresses on the summit of the crack for a sample with thickness at maximum loading at the summit of the crack for $\mu= 0,3$ – Poisson coefficient; $R_{0,2}$ - conditional limit of flow; E - elasticity module; $W=20$ mm - height; $B=20$ mm – width of the sample; $a = 10$ mm- mechanical cut section and crack; $Z = 1,7$ mm – thickness of the plates for fixing the sensor.

METALLOGRAPHIC INVESTIGATIONS

In order to clarify the morphological peculiarities of the nitrided layers and their phase composition, metallographic analysis has been carried out. Metallographic microscope – Axioskop – has been used for carrying out the metallographic investigation of the samples, which has taken metallographic pictures of the nitrided layers. Micro-hardness is measured by means of the micro-hardness meter „Shimadzu” with a load of 100g by the Vickers’ method. The fractographic analysis has been performed by means of a scanning microscope “Philips-Sem 515”.

EXPERIMENTAL RESULTS AND ANALYSIS

By measuring the micro-hardness of the nitrided sample in depth the maximum surface hardness $HV_{0,1}$ is defined. The total thickness δ_{tot} of the nitrided layer is measured by the depth at which hardness, equal to the core hardness is achieved; by means of the metallographic microscope the thickness of the combined zone δ_{cz} is defined. The obtained results are given in Table 3.

Table 3. Modes and results from the ion nitriding of the samples

Стoмaнa BH21							
N ^o of the experiment	t, X_1 °C	P_{NH_3}, X_2 Pa	τ, X_3 h	δ_{ic} mm	δ_{cz} μm	δ_{tot} μm	$HV_{0,1}$ -
1	510	150	4	0,016	2	130	1048
2	550	150	4	0,008	4	180	1017
3	510	450	4	0,017	5	120	1064
4	550	450	4	0,005	8	200	1100
5	510	150	10	0,014	2	190	1000
6	550	150	10	0,004	6	260	1097
7	510	450	10	0,009	2	200	932
8	550	450	10	0,003	4	220	1048
9	530	300	7	0,006	5	280	1141

Note: After heat treatment $\delta_{ic}=0,00834\text{mm}$ [14]

On the basis of the obtained results, given in Table 3, for the investigated steel the following significant adequate regressive equation is obtained for opening the edges of the crack δ_{ic} at static bending:

$$\delta_{ic} = 0.00922 - 0.0045 X_1 - 0.001 X_2 - 0.002 X_3 \quad (3)$$

The mathematical model obtained for steel has a high coefficient of multiple correlation $R = 0.9592$ and is adequate at a level of significance 0.05. $F_{0.05, 3, 5} = 19.17 > F_{таб.} (0.05, 3, 5) = 5.4095$.

The negative values in front of X_1 , X_2 and X_3 in the derived equation show that with increasing the temperature of nitriding - X_1 , the ammonia pressure in the chamber - X_2 , as well as increasing the duration of treatment - X_3 , the value of the crack edges opening δ_{ic} in the multilayer system decreases. As a result, the resistance against fragile destruction in the nitrided samples decreases. This is explained by the fact that for this change of the input factors (X_1, X_2, X_3) of the process of nitriding, nitrided layers with higher thickness are obtained. From the derived equation 1 it can be noted that the most considerable influence over the crack edges opening δ_{ic} has the temperature of nitriding; the duration of nitriding comes next and, the least considerable influence has the ammonia pressure in the vacuum chamber.

From Table 3 it can be seen that after ion nitriding of BH21 steel at temperature $t = 510^\circ\text{C}$, ammonia pressure in the vacuum chamber $P_{NH_3} = 150 \text{ Pa}$, time of treatment $\tau = 4 \text{ h}$ a layer with combined zone thickness $\delta_{cz} = 2 \mu\text{m}$, total thickness $\delta_{tot} = 130 \mu\text{m}$ and maximum micro-hardness 1048 $HV_{0,1}$ is formed. At these characteristics of the layer the critical crack edges opening is $\delta_{ic} = 0,016 \text{ mm}$ – fig.3.

With increasing the time of nitriding from 4h to 10h (mode 5, table.3) a layer is obtained, having higher total thickness $\delta_{tot} = 190 \mu\text{m}$, and combined thickness of $\delta_{cz} = 2 \mu\text{m}$. The critical crack edges opening of $\delta_{ic} = 0,014$ is a bit smaller than at the short-time mode of ion nitriding. This is probably due to the small thickness of the fragile combined zone ($\delta_{cz} = 2 \mu\text{m}$), formed at these two modes of nitriding – fig. 4. With increasing the temperature from 510°C to 550°C the biggest crack edges opening $\delta_{ic} = 0,008 \text{ mm}$ is observed at the second mode of treatment from Table 3. This value is obtained at a layer with combined zone thickness $\delta_{cz} = 4 \mu\text{m}$, total thickness of $\delta_{tot} = 180 \mu\text{m}$ and maximum micro-hardness 1017 $HV_{0,1}$. It is important to say that for all modes of nitriding at temperature 550°C the critical crack edges opening δ_{ic} is smaller than it is after the processes of hardening and tempering – table 3.

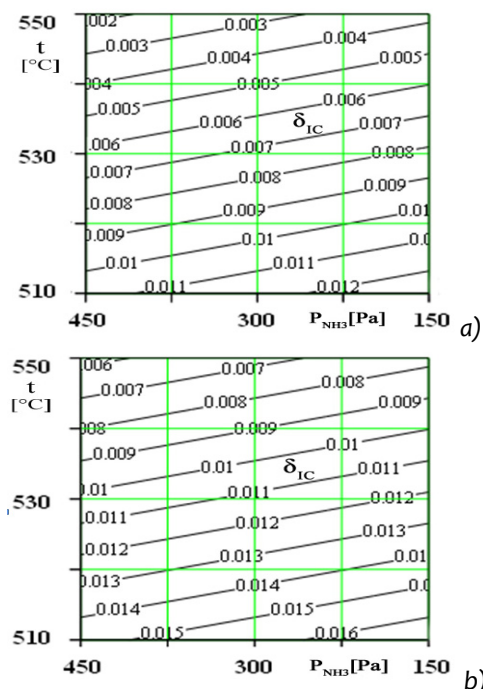


Fig.3. Graphic dependencies between the temperature of nitriding, ammonia pressure and the critical crack opening at times of treatment: a) $\tau = 4 \text{ h}$; b) $\tau = 10 \text{ h}$

It can be noted that the formed nitrated layers with total thickness of ($\delta_{\text{tot}} = 120 - 130 \mu\text{m}$) and small combined zone ($\delta_{\text{cz}} = 2-5 \mu\text{m}$), as well as the available compressive stresses [15] near the summit of the crack, lead to increasing the resistance against fragile destruction.

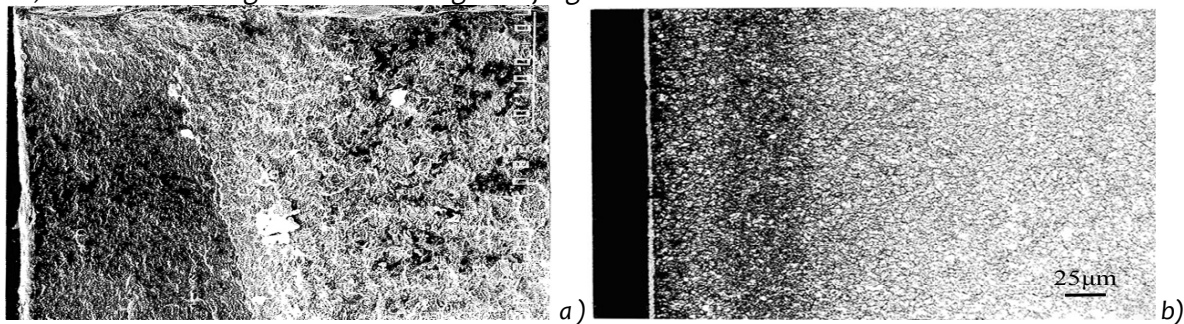


Fig.4. Ion nitrated BH21 steel at: $t = 510^\circ\text{C}$, $P_{\text{NH}_3} = 150 \text{ Pa}$, $\tau = 10 \text{ h}$

a) Fractograph of the nitrated steel - $\delta_{\text{ic}} = 0,014 \text{ mm}$; b) Microstructure of the nitrated steel

When loading the ion nitrated samples near the summit of the cut section, where there is a nitrated layer, processes of decreasing the residual compressive stresses occur, on the one hand, and, on the other hand, plastic deformation of the layer happens, together with its premature cracking because of its high module of elasticity. Micro-cracks are first formed in the nitrated zone and then in the diffusion zone, which is small-grained in comparison to the basic material – fig. 4a.

The ammonia pressure in the chamber does not influence considerably the critical crack edges opening – equation 1.

On the basis of the obtained results it can be noted that, in comparison to hardened and tempered at 700°C samples ($\delta_{\text{ic}} = 0,00834 \text{ mm}$), all modes of ion nitriding, conducted at 510°C , increase the resistance against fragile destruction in BH21 steel – fig. 3.

CONCLUSIONS

It has been established that at all modes of ion nitriding conducted at temperature of 510°C , the critical crack edges opening (δ_{ic}) is bigger than it is at hardening and tempering, while at temperature of 550°C it (δ_{ic}) is smaller.

It has been proved that the highest value of crack edges opening $\delta_{\text{ic}} = 0,017 \text{ mm}$ is obtained at $t = 510^\circ\text{C}$, $P_{\text{NH}_3} = 450 \text{ Pa}$, $\tau = 4 \text{ h}$.

A regressive equation is derived, covering the relationship between the technological factors of the process (temperature of nitriding, ammonia pressure in the chamber, duration of treatment) and the aimed parameter (the critical crack edges opening) for BH21 steel, ion nitrated after hardening and tempering at 700°C .

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