Results of steel 20Kh13 samples with combined hardened-structured surface erosion tests

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Abstract. The work presents the results of erosion tests and metallographic studies of samples from blade steel 20Kh13 with diffusion coating obtained by ion nitriding and samples from nitrided steel 20Kh13 with a relief previously formed on the surface. Studies of the samples resistance to high-speed water droplet erosion were carried out using the unique hydraulic impact rig «Erosion-M» of the NRU «MPEI». It was established that the use of nitriding and nitriding with preliminary surface structuring allows increasing the relative erosion resistance in the area with a maximum wear rate of at least 2 times, and in the area with a steady wear rate of at least 3 times. It was also found that structuring the surface layer before the nitriding process allows reducing the wear rate in the area with a maximum erosion rate compared to the nitrided surface by at least 10%.

1. Introduction
Numerous methods are currently known for controlling water droplet erosion, which are based on the use of separation devices (active methods) located on the nozzle blades periphery and removing part of the moisture from the two-phase flow, as well as the formation of various types of coatings or modifications of functional surfaces (passive methods), which allow to increase the erosion resistance of blade devices that perceive the impact of drops not caught by separation devices [1].

To solve the current problem of water droplet erosion, it is necessary to understand not only the physics of fast-flowing wear processes, but also the effect on their intensity of surface relief geometry, as well as its properties and characteristics. In this regard, research is currently underway in the world to study and create a "smart" surface [2] that can withstand or reduce the effects of high-speed impact of liquid droplets under changing operational loads. One way to solve this problem can be to conduct studies on the selection of optimal characteristics of the structured relief [3] and the development of a method for strengthening such a surface, which together can withstand destructive loads. The development of a qualitatively new method of protection will allow to achieve significant scientific results on the creation of a "smart" surface resistant to drop impact, applicable also in the applied field, taking into account the development of the energy industry and new technological "challenges."

One method of strengthening may be the process of modifying the surface to be protected by its diffusion single- and multicomponent saturation with metals and non-metals using ion-plasma techniques. As an effective protection against corrosion, erosion and prevention of reduction of fatigue strength of working surfaces of elements of power turbine plants, nitriding process is of great interest [4-8]. Nitrogen atoms embedded in the matrix of the base material effectively compress the structure, thereby increasing hardness (protection against drop-impact erosion) and "healing" the existing defects (corrosion protection, increasing the endurance limit [9]).
As a result of studies conducted earlier at the NRU «MPEI», it was revealed that surface modification by ion-plasma nitriding allows at least 3 times to increase the erosion resistance of blade steels with ultra-high-speed drop impact (studies were carried out for steels 12Kh13 and EI961 as part of the implementation of Agreement No. 14.577.21.0180 of 27.10.2015 with the Ministry of Education and Science of the Russian Federation, ID No. RFMEIF7715X018).

Creating an ordered surface pattern using laser structuring is also one of the most promising methods in connection with the potentially high speeds of creating such surfaces, as well as the ability to obtain given geometric parameters of the pattern with high accuracy. Creating a relief with specified geometric parameters can potentially increase the ability of the blade material to withstand various types of damage [10, 11], including high-speed drop impact.

The combination of surface modification by structuring it and subsequent nitriding may prove to be a promising new method for protecting vanes operating under extreme conditions of high-speed vapor drop flow.

2. Description of involved equipment, methods of conducting studies
To form an ordered relief using pulse laser processing and carry out surface hardening processes by diffusion saturation with nitrogen atoms, experimental samples were made of blade steel 20Kh13. Figure 1 shows the schematic diagrams of the considered surface treatment options.

![Diffusion saturation](image1)

Figure 1. Diagrams for the creation of hardened (a) and a combined hardened-textured surface (b)

The structured relief was created using the FMark NS-FB-20 laser complex (TsLT LLC, Russia) [12] based on an infrared ytterbium fiber laser. Selection of relief formation parameters was based on results of preliminary performed parametric tests [13, 14].

The process of ion nitriding of steel samples 20Kh13 without and with a structured surface was carried out in a vacuum plant "Gefest +" [9, 14].

Experimental studies of the resistance of blade steel samples 20Kh13 to water droplet erosion were carried out at the unique erosion rig "Erosion-M" of NRU "MPEI" [15] according to GOST 23.219-84 [16].

As a result, kinetic erosion curves were obtained, drawn in the coordinates of mass loss from the sample ($\Delta m$, g) and test time ($t$, min). The weight loss of the sample was calculated by the formula:

$$\Delta m_i = m_0 - m_i,$$  \hspace{1cm} (1)

where $m_0$ is the initial mass of the sample, g; $m_i$ is the mass of the sample after the experiment, g; $i$ is the experiment number.

3. Results and discussion
The surface nitriding process was carried out for two types of samples - without relief and with a previously formed structured relief (see Figure 2).
On a Tescan Mira3 LMU raster electron microscope, images of metallographic slips of samples with various surface treatment options were obtained. After obtaining the slip images, the surface nitriding depth was measured, and the dimensions of the obtained relief, such as the width of the protrusions and depressions, as well as their depth, were taken. Geometric parameters of the obtained relief: depth of the depression (height of the protrusion) - 15 μm; the width of the projection is 50 μm; the width of the depression is 50 μm. The direction of the grooves is longitudinal. The nitriding depth of the surface of the samples was 40-60 μm. The nitriding process on the relief samples (see Figure 3) was carried out to the height of the relief protrusions, so that the structured surface was completely modified, as well as deep into the base material with a smooth gradient of microhardness in depth.

Microhardness of samples without covering was 250±30 HV0.1 of steel 20Kh13; for samples with nitriding 1200±20 HV0.1. The surface roughness of the samples without relief after the nitriding process was Ra = 0.5 μm.

Tests of samples from steel 20Kh13 without and with various options of modification of a surface on resistance to a water droplet erosion were carried out at parameters of influence close to service conditions and resulting in the greatest possible wear, namely: speed of impact of a drop with the samples \( C_{\text{coll}} = 300 \text{ m/s} \), diameter of liquid drops \( d_{\text{d}} = 800 \text{ μm} \). Figure 4 shows the obtained kinetic curves of erosion wear of steel samples 20Kh13 without modification, with nitriding and with a structured surface with subsequent nitriding.
Based on the obtained dependencies, it can be concluded that ionic nitriding and nitriding with preliminary surface structuring does not increase the incubation period of the process of water droplet erosion of steel 20Kh13, but allows to increase the relative erosion resistance in the area with maximum wear rate not less than 2 times, and in the area with steady wear rate not less than 3 times. As a result of erosion tests, the effect of the initial formation of the relief before nitriding was revealed. It consists in the fact that the use of surface structuring before the nitriding process allows to reduce the wear rate in the area with the maximum erosion rate compared to the nitrided surface by at least 10% (see Figure 5, in which the incubation period area and the area with the maximum erosion rate are separately separated).

To consider the effect identified above, after erosion tests, metallographic slips of the studied samples were prepared. Since the obtained mass entrainment values were carried out by increasing the exposure time of the same samples, the slips were made for samples whose surface destruction corresponded to a step with a steady-state wear rate.
The slips were made so that the cut plane was in longitudinal and cross section, the so-called erosion "trace" of the studied sample - a conglomerate of grooves and protrusions of the damaged surface at the place of water droplet impact. In addition to imaging the surface fractograms at the drop point, the change in the microhardness of the damaged hardened layer in depth was measured. The results of these studies are shown in Figure 6.

![Graph showing microhardness measurement](image)

Figure 6. Measurement of steel sample 20Kh13 with nitriding surface layer microhardness in the area of erosion trace after drop impact at the stage with a steady rate of erosion:

a - the width of the trace; b - zone A (measurements from the "bottom of the trace" deep into the sample); c - zone B (measurements from surface by depth of nitrated layer)

For the conditions of the studies, the process of diffusion saturation of the surface to a certain depth mainly depends on the nitriding time. Nitrided near-surface layer has microhardness more than 4 times that of substrate material. The transition from the increased microhardness of the nitrated layer to the microhardness of the substrate is smooth and consistent with the identified depth of modification. In the vicinity of the place where the drop load is applied (point 5 in Figure 6, a), the obtained microhardness values indicate an increase in microhardness from the values 500 HV0,1 to 1200 HV0,1 not exposed to nitrided layer. The gradual change of the microhardness values from the impact axis (point 5 in Figure 6, a) of a 800 μm drop in diameter over a distance of approximately 400 μm indicates a decreasing failure intensity from the center of the load application. Value of microhardness in 500 HV0,1 in the center of the impact indicates the wear of the nitrided layer due to the fact that the nitrided layer at the point 5 in Figure 6, and the corresponding point in Figure 6, b still has a depth (20 μm) equal to 1/3÷1/4 of the nitrided layer total depth.
Damage to the reinforced near-surface layer at the steady stage of erosion occurs due to the probable occurrence of longitudinal fatigue cracks in the brittle and, at the same time, very hard nitrided layer. Possibly, with high-speed spreading over a surface previously having it’s relief due to multiple droplets, the liquid flow finds it’s way out in penetration along the grain boundaries of the nitrided layer, gradually tightening and expanding the resulting longitudinal cracks. Further, droplet shocks result in chipping of layers formed by longitudinal cracks.

The nitriding process on the relief samples was carried out to the height of the relief protrusions, thus the structured surface was modified completely to the entire depth of the formed relief. The increased microhardness is maintained to the full depth of the modified relief projection layer, then smoothly reduced to the microhardness values of the base material. This circumstance is possible due to the fact that diffusion saturation with nitrogen occurs locally, for each individual groove (on the transverse slip - ledge), in contrast to the untreated surface. Therefore, the processing process is more saturated due to the higher specific gravity of the energy interacting with the surface.

Figure 7 shows the fractograms and the results of measurements of microhardness of the surface layer of a 20Kh13 steel sample with a structured surface and nitriding in the area of the erosion trace after and outside the droplet impact, as well as the results of microhardness measurements in the width of the trace, from the “bottom of the trace” deep into the sample and from the surface by the nitrided layer depth.

![Fractogram](image1)

![Measurement Graph](image2)

Figure 7 - Measurement of the surface layer of the steel sample 20Kh13 with structured surface and nitriding microhardness in the area of erosion trace after the drop impact at the steady state erosion rate: a - the width of the trace; b - zone A (measurements from the "bottom of the trace" deep into the sample); c - zone B (measurements from surface by depth of nitrided layer
The formation of the pattern on the surface of the sample creates areas which, when burned by a laser beam, are filled with a mixture of molten base substrate material. Uniform nitriding of the resulting peaks and depressions causes additional surface hardening, removing potentially hazardous areas that can serve as catalysts during subsequent erosion tests. The microhardness of the nitrided relief is maintained throughout the depth of the peaks and depressions, further deep into the hardened nitrided layer and smoothly reduced to the microhardness of the base material (see Figure 7, c). Wear patterns (see Figure 7 (a)) the passage of the reinforced layer and the destruction of the base substrate material (see Figure 7, b).

Formation of relief with subsequent nitriding increases resistance to the drop impact at a site with a maximum erosion rate of a relatively separately nitrated surface due to possible other spillage physics due to the already created surface relief, which is explained by the following.

The period with the maximum erosion rate is fast flowing and, to some extent, preparatory for the subsequent period with a steady rate of erosion. Namely, preparatory in the sense of creating a certain relief, after the end of the incubation period, initially adapting to the impact drop, which subsequently, after the appearance of cavities and troughs, leads to a constant wear rate of the material - the occurrence of a period with a steady rate of erosion.

The creation of a structured relief with given characteristics anticipated the creation of such a relief, as a result of which the rate of erosion decreased and a smoother transition characteristic to the period with a steady rate of erosion was formed. Complete destruction, created using laser relief, subsequently leads to gradual destruction deep into the nitrided layer. Further, the physics of the wear process becomes close to the process occurring with the sample only with nitrinding and the subsequent coincidence of curves 2 and 3 in Figure 4.

4. Conclusion

1. As a result of the work, several versions of blade steel 20Kh13 samples with a strengthened surface were created by laser structuring of the surface and its modification by ion nitrinding.
2. A set of erosion tests and metallographic studies of the obtained samples was carried out. Erosion resistance of the described hardening methods and influence of high-speed water droplet impact on microstructure of the near-surface layer and change of microhardness in depth in the drop impact zone are revealed.
3. The use of nitriding and nitriding with preliminary surface structuring does not increase the incubation period of the process of water droplet erosion of steel 20Kh13, but allows increasing the relative erosion resistance in the area with maximum wear rate by at least 2 times, and in the area with steady wear rate by at least 3 times.
4. The effect of the initial creation of the relief before nitriding is established, which consists in the fact that the use of surface structuring before the nitriding process allows reducing the wear rate in the area with a maximum erosion rate compared to the nitrided surface by at least 10%.
5. It is assumed that the formed relief increases the resistance of the nitrided layer in the area with the maximum erosion rate due to the surface already pre-prepared for droplets impact, which perceives this effect characteristic of conditions close to the period with a steady-state erosion rate.
6. The surface hardening options discussed show their potential suitability for increasing the erosion resistance of steel 20Kh13. To increase the durability of the nitrided layer over the period with maximum erosion rate, it is necessary to preliminary laser structure the surface with certain values of depth and distance between the created grooves, depending on the droplets impact parameters - the size of the particles and their collision rate.

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