Fatigue tests results of blade steels with modified surface

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Abstract
The paper presents the results of metallographic studies and fatigue tests of blade steel 12kH13 and EI961 samples with modified near-surface layer. Fatigue tests and studies of the samples with the modified layer were carried out using the research equipment URI (unique research installation) "Hydroshock rig Erosion-M" of NRU "MPEI". The surface modification is found to increase the fatigue strength of blade steel up to 50%. Sample surface after modifications features a cell structure with the characteristic cell size ranging from 1÷2 μm to 4÷8 μm; total thickness of the modified layer for steel samples 12kH13 and EI961 was about 40 μm.

1. Introduction
The process of nitriding (surface saturation with nitrogen ions N⁺) is of particular interest as one of efficient protection methods against corrosion, erosion and avoiding the decrease of fatigue resistance for elements of power turbine units. Nitrogen ions integrated into the base material matrix provide efficient sealing of the structure increasing its hardness (protection against water droplet erosion) and healing any present defects (protection against corrosion, fatigue strength increasing).

Increasing erosion resistance of wet steam turbine rotating blades phases by means of certain strengthening process must not compromise the durability limit of blade steel. Therefore, evaluation of fatigue performance of hardened blade steel is one of the necessary regulated tests at the development and implementation stage of hardening processes of rotating blades [1].

The task of this study was to carry out the research and measure the fatigue strength of blade steels 12kH13 and EI961 with modified surface.

2 Research methodology
Surface modification of blade steel samples 12kH13 and EI961 was carried out by means of the vacuum apparatus “Gefest-HIPIMS” (see Figure 1). Modification was performed via surface saturation of samples in the nitrogen environment (nitriding), which lasted 1 hour, 2.5 hours and 5 hours.

The surface modification process included the following stages:
- preliminary preparation of sample surfaces (polishing, removal of surface contaminations and degreasing);
- loading of samples into the vacuum chamber;
- pumping out the vacuum chamber to reach the high vacuum condition after preheating it to 150°C;
- ionic cleaning of the surface;
- modification of sample surface in the nitrogen environment during 2.5 hours.

Operating parameters of the surface modification process main phases in the vacuum apparatus “Gefest-HIPIMS” are shown below (see Tables 1÷3).
Figure 1. Photograph of the vacuum apparatus “Gefest-HIPIMS” for magnetron sputtering

Table 1. Main operating parameters of the pumping out and preheating phase of the surface modification process

| Phase duration, min | Base layer voltage, V | Base layer temperature, °C | Vacuum chamber pressure, Pa |
|---------------------|-----------------------|-----------------------------|-----------------------------|
| 45                  | -                     | 100-150                     | maximum 8·10⁻³               |

Table 2. Main operating parameters of the ionic cleaning phase of the surface modification process

| Phase duration, min | Base layer voltage, V | Base layer temperature, °C | Vacuum chamber pressure, Pa |
|---------------------|-----------------------|-----------------------------|-----------------------------|
| 30                  | 600÷1,000             | maximum 350                 | maximum 0.35                |

Table 3. Main operating parameters of the surface modification phase

| Phase duration, min | Base layer voltage, V | Nitrogen consumption, nl/hr | Base layer temperature, °C | Vacuum chamber pressure, Pa |
|---------------------|-----------------------|-----------------------------|-----------------------------|-----------------------------|
| 150                 | 600÷1,000             | 18.0                        | maximum 380                 | 2.1                         |

Characteristics of samples with modified surface were measured using the research equipment URI (unique research installation) “Hydroshock rig Erosion-M” of NRU “MPEI” capable of investigating the composition, structure, thickness, microhardness and erosion resistance of modified layers.

Composition, structure and thickness of modified layers were studied using the scanning electronic microscope TESCAN MIRA 3 LMU equipped with the EMF spectrometer X Max 50 (Oxford Instruments). In addition to the general energy dispersion microanalysis of the modified surface composition, it was analyzed layer by layer by means of the glow discharge optical emission spectrometer GD Profiler-2 (Horiba Jobin Yvon) to obtain element distribution profiles in the layers.

Metallographic sections were made using a set of sample preparation equipment including: abrasive cut-off wheel with movable linear cutting system POWERMET 3000 (Buehler GmbH), electrohydraulic press SIMPLIMET 1000 (Buehler GmbH) and section polishing machine BETA/1 (Buehler GmbH).

Microhardness of modified layers, as well as samples without surface modifications was measured by means of the hardness meter DuraScan 20 (Emco-Test). Measurements were carried out according to the Vickers method, under load of 0.05 kgf (0.491 N).
Standard fatigue tests [2] by pure bending and rotation of blade steel 12kH13 and EI961 samples without hardening and with modified surface, were carried out by means of the experimental apparatus CU-1. During the fatigue studies, the main criterion for evaluation of the durability limit was either complete destruction of the test sample, or appearance of macrocracks. In order to build a fatigue curve and to measure the durability level $\sigma$, as well as to perform a comparative analysis of fatigue testing results (in terms of durability level $\sigma$), a batch consisting of minimum 15 identical test samples was studied. Within the stress interval of 0.95÷1.05 of the durability level, at least three samples were studied, of which at least half were not supposed to collapse prior to the research reference point. The research reference point for durability limit evaluation was taken as $10^7$ cycles at the test frequency of 50 Hz.

3. Results and discussion

As a result of surface processing of samples made of blade steels 12kH13 and EI961, 20 to 60 μm thick modified layers were obtained. Fatigue tests have been performed for all surface modification options; below please find the results of studies of the samples featuring the best fatigue resistance (the surface modification process lasted 2.5 hours).

Figures 2 and 3 show images of cross sections and characteristic surface appearance of blade steel 12kH13 and EI961 with modified surface samples.

![Figure 2](image2.png)

**Figure 2.** Image of modified surface and microstructure of the blade steel EI961 sample nearsurface layer

![Figure 3](image3.png)

**Figure 3.** Image of modified surface and microstructure of the blade steel 12kH13 sample nearsurface layer

It has been established that the grain size in the modified layers does not change; there are virtually no inclusions (chromium nitride) along grain boundaries in the modified layers; the width of the transition zone, where the nitrogen content decreases to the base material level, is 5÷10 μm. The modified surface of the test samples under consideration features a cell structure; characteristic cell sizes range from 1÷2 μm to 4÷8 μm. Thickness of the nearsurface layer, in which the nitrogen content increases sharply (the nitride layer) is within 2 μm. The average thicknesses of the modified layers after processing for 2.5 hours are shown in Table 4. The results of energy dispersion microanalysis of the elemental composition including the average contents of the elements in the modified layers are shown in Table 5.
Table 4. Modification depth after 2.5 hours long processing

| Item No. | Sample description                        | h, μm    |
|----------|-------------------------------------------|----------|
| 1        | Steel EI961 after 2.5 hours of nitriding  | 37.1±1.6 |
| 2        | Steel 12kH13 after 2.5 hours of nitriding | 38.1±1.8 |

Table 5. Results of elemental composition analysis of modified layers

| Sample         | Average elemental contents in the modified layer, % by weight |
|----------------|-------------------------------------------------------------|
|                | N  | Si   | Cr   | Mn  | Fe   | Ni   | W   |
| Steel EI961    | 4.0±0.2 | 0.40±0.02 | 11.5±0.1 | -  | 79.5±0.3 | 1.85±0.13 | 2.12±0.05 |
| Steel 12kH13   | 3.3±0.9 | 0.48±0.03 | 13.1±0.4 | 0.23±0.04 | 82.5±1.1 | 0.43±0.05 | -   |

Figure 4 shows post-modification nitrogen distribution profiles along the surface layer depth obtained by means of the glow discharge optical emission spectrometer. The average nitrogen content in the modified layers is within the level of 2.4÷4.2%. The nitrogen concentration varies slightly within the nitrided layer (the farther from the surface, the lower is the concentration), and it increases sharply near the surface.

![Figure 4](image)

Figure 4. Nitrogen distribution in the nearsurface layer of blade steel samples, grades EI961 (a) and 12kH13 (b), after 2.5 hours long processing

Table 6 shows the average surface microhardness values of blade steel samples EI961 and 12kH13 with the modified layer; data analysis of Table 6 demonstrates that the above modification increases surface microhardness 3.5÷5 times compared to the original level.

Table 6. Results of mechanical properties studies of steel grade samples 12kH13 and EI961 without modifications and with modified surface

| Item No. | Sample description                        | Microhardness acc.to Vickers H0.2, HV |
|----------|-------------------------------------------|---------------------------------------|
| 1        | Steel 12kH13 without modifications        | 269±4                                 |
| 2        | Steel EI961 without modifications         | 273±6                                 |
| 3        | Steel 12kH13 after modifications          | 1,330±40                              |
| 4        | Steel EI961 after modifications           | 1,055±20                              |

Results of the above fatigue tests are shown in Figures 5 and 6. Analysis of the resulting correlation between the stress amplitude $\sigma_a$ and the number of cycles to failure $N_p$ by pure bending and rotation (symmetrical load cycle) shows that the above 2.5 hours long surface modification increases the durability limit of 12kH13 and EI961 steels at least by 50%.
4. Conclusions
The surface treatment process under study features good implementation prospects for development of passive protection methods against various wear types and for increasing the equipment life cycle exposed to high operating loads. It is the authors’ opinion that the risk of power generation equipment responsible elements failure (for instance, rotating blades of the wet steam turbine stages) due to combined impact of fatigue damage and surface destruction development due to progressing erosion and corrosion processes can be reduced if the production process combines surface modification and subsequent formation of wear resistant ion-plasma coatings.

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