Metallographic studies results of 20kH13 steel samples with textured relief, modified surface and protective coating

Alexey Mednikov¹, Aleksandr Tkhabisimov¹*, Marat Dasaev¹, Andrey Burmistrov¹ and Olga Zilova¹

¹National Research University “Moscow Power Engineering Institute”, Moscow, Russian Federation

Abstract. One of the possible ways to improve passive methods of the steam turbine blade material protection from water droplet erosion is to create "smart"-surface that combines the properties and characteristics that could not be implemented simultaneously when using one or another proven method of protection. To solve the problem of developing such surface, this article presents the metallographic research results of the blade steel 20K13 samples with various already used and promising passive methods of protection, consisting in the use of ion-plasma and polyurethane coatings, surface modification by means of diffusion saturation with nitrogen ions, creation of the structured surface relief by laser ablation. The results of the carried out researches have allowed to reveal both morphology of a possible "smart"-surface, and influence on a microstructure and characteristics of a near-surface layer of samples after treatments considered.

1 Introduction

Nowadays, there are different methods to combat water droplet erosion, which are based on the use of various separation devices (active methods) located on the periphery of the nozzle blades and removing part of the moisture from the two-phase flow, as well as the formation of different types of coatings or modifications of functional surfaces (passive methods), which allow to increase the erosion resistance of the blade material, which perceives the impact of drops not captured by separation devices [1-5]. One of the directions of protection passive methods improvement is the creation of "smart"-surface that combines the properties and characteristics that could not be simultaneously realized when using one or another proven method of protection [6-9].

Processes with the use of ion-plasma technologies of protective coatings formation in vacuum on the basis of nitrides, carbides, carbo-nitrides of refractory chemical elements or modification of the protected surface due to its diffusion of single- and multi-component saturation with metals and nonmetals are very promising methods. To date, effective and proven for various applications are coatings based on chromium Cr and it’s carbide CrC, superhard diamond-like DLC coatings, as well as processes of surface saturation with nitrogen ions (nitriding) [10-14].

The use of laser structuring for the creation of different-scale ordered surface relief and changes in it’s properties is also one of the most promising methods due to the potentially high rates of creation of such surfaces, as well as the possibility of obtaining given geometric parameters of the topography with high accuracy. The application of the laser surface structuring method may be relevant for the improvement of wear-resistant properties of such highly loaded and operated under severe conditions objects as steam turbine blades. Creation of relief with known and specific required geometric parameters can potentially increase the ability of the blade material to withstand various types of destructions [15, 16].

Formation of ion-plasma coatings in combination with surface modification by means of it’s structuring or nitriding may turn out to be a new promising way to protect turbine blades operated under extreme conditions of the impact of high-speed steam-droplet flow.

Also, one of the potentially promising compositions of coatings is wear-resistant rubber-like coatings on polyurethane basis, which allow to dampen the impact of droplets and reduce the rate of wear, as well as strong adhesion by applying to the previously formed relief [17, 18].

Various combinations of the considered methods will allow to endow the surface with both hydrophilic and hydrophobic properties [12-16] and to improve it’s strength characteristics, which in aggregate should

* Corresponding author: abt-bkt@mail.ru
contribute to increase the resistance of the material to droplet load.

2 Methods

Experimental samples from steel 20kH13 applied to manufacture blades of steam turbines low pressure cylinders last stages have been made for the purpose of the ordered various scale relief formation with use of pulse-laser processing, carrying out of processes of surface modification and protective coatings formation. Fig. 1 shows the basic schemes of all considered variants of surface treatment.

![Figure 1: Proposed schemes of steel 20kH13 experimental samples surface treatment: a) surface nitriding; b) formation of ordered relief; c) formation of relief with subsequent nitriding; d) formation of relief with subsequent application of protective polyurethane coating; e) formation of relief with subsequent formation of protective ion-plasma coating; f) formation of protective ion-plasma coating with subsequent formation of ordered relief.](image)

2.1 Relief structuring on sample surfaces

Relief texturing of the surface was carried out on specially made experimental samples of steel 20kH13 and was carried out by using the FMark NS-FB-20 laser complex (OOO «TsLT», Russia), based on an infrared ytterbium fiber laser having the following technical characteristics: wavelength - 1064 nm, pulse duration - from 4 to 200 ns, pulse frequency - from 20 to 100 kHz, radiation power at the output of the focusing system - from 1 to 20 W. A lens with a focal length of 100 mm was used. At the same time, the laser beam is focused on the treated surface using the biaxial deflection system MS-II-10 (RAYLASE AG, Germany).

Samples were processed by laser pulses with frequency $f = 50$ kHz, at the speed of linear movement of the beam $V = 500$ mm/s and at the source power $N = 20$ W (see Fig. 2). The choice of relief formation parameters was based on the results of preliminary parametric tests [21].

![Figure 2: Process of ordered structured relief formation on the steel 20kH13 sample surface](image)

Fig. 2. Process of ordered structured relief formation on the steel 20kH13 sample surface
2.2 Modification of sample surface

Modification was carried out by means of surface diffusion saturation of samples in nitrogen medium (nitriding) at "Gefest +" unit of NRU «MPEI» [22]. Preliminary preparation of sample surfaces was carried out in the electrolyte-plasma polishing unit EPP-10. Before the process of surface modification was carried out, the chamber was pumped to a high vacuum with preheating to intensify the process of degassing the chamber and samples. After pumping the plasma-forming gas (argon) was supplied to the vacuum chamber and ion cleaning of samples was performed to remove the oxide film and activate their surface. Negative voltage was applied to the product. Then the process of blade steel 20Kh13 samples nitriding surface was carried out.

2.3 Formation of protective ion-plasma coatings on the samples surface before and after the structured relief formation

Two types of protective coatings were formed on the 20Kh13 steel samples: CrC-based and diamond-like DLC coating. Both of these coatings have proven to be highly effective in erosion testing, so together with the relief structuring they are potentially resistant to the process of water droplet erosion.

Coating on the basis of DLC was carried out on the "Gefest HPIMS" unit of NRU «MPEI» [23, 24] after the formation of a structured relief. Experimental samples and parts were installed in a vacuum chamber. The special design of the tooling provided planetary rotation of the samples inside the unit. The process of DLC-coating formation took place in the following sample. Pre-prepared samples were placed in the vacuum chamber of the unit, after which the chamber was pumped out and the samples were heated. After the pressure in the vacuum chamber was reached, preheating of the samples was switched off, the vacuum gate was throttled halfway and the plasma-forming gas argon was injected. Negative voltages (bias voltages) were applied to samples moving inside the vacuum chamber, glow discharges were ignited and ion cleaning was performed. After ionic cleaning, a pure titanium (Ti) carbide-forming metal adhesive layer was applied to the samples. After the adhesion layer was formed, an intermediate layer of titanium carbide (TiC) was applied. A final diamond-like coating was then applied.

Cr-CrC-based coating was formed on the «Gefest» unit of NRU «MPEI» in a similar way. The only difference was that the structured relief was formed on the samples of steel 20Kh13 after the coating on the basis of Cr-CrC.

2.4 Application of protective polyurethane paint

Polyurethane coatings are a product of mixing thermosetting and thermoplastic powder compositions. Polyurethanes, which are the products of interaction between glycols and polyisocyanates, serve in this case as film formers. They have high adhesion, which provides excellent adhesion to the treated surface, as well as a combination of flexibility and hardness. This quality gives significant wear resistance and durability. Application of such type of coatings creates a reliable and durable protective layer, which is also resistant to corrosion, abrasion and scratches [17-19].

Within the framework of this work a polyurethane coating was used, which is a complex two-component mixture with good adhesion. Application of this type of coating was carried out with the help of a special spray gun on the samples after the formation of a structured relief.

2.5 Manufacture of samples metallographic grinding

The following machine tools were used to prepare the samples for research: the abrasive cut-off machine with linearly moving cutting system PowerMet 3000, automatic pressuring press Simplimet 1000, installations for cold casting of samples Cast n'Vac 1000 and grinding and polishing machine Beta with automatic nozzle Vector and modular liquid distribution system PriMet 3000.

3 Results and Discussion

As a result of the studies identified:

- for structured relief:
  - The geometric parameters of the obtained structured relief: the depth of the depression (height of the protrusion) 70 μm; the width of the protrusion is 70 μm; the width of the depression is 25 μm. The direction of the grooves is transverse. At the moment when the laser pulse "hits" the surface, in the place of the contact spot, the energy of the pulse is absorbed and local material is rapidly heated, which leads to its melting and ablation (evaporation). In this case, most of the molten metal moves from the center of the contact spot to its periphery, forming a depression (crater) on the surface. As a result of these processes, the surface cools at a very high speed and quickly freezes. Some of the nanoparticles removed from the surface as a result of the ablation process return to the surface and contribute to the creation of multimodal roughness. For a selected structured surface relief of transverse grooves, a geometry consisting of protrusions and depressions is characteristic, and the molten metal when exposed to a laser beam leaves characteristic solidified formations in the form of drops at the edges of the protrusions.

- for structured terrain with polyurethane coating:
  - When forming, the polyurethane coating lays in an even layer, uniformly filling all pores, cavities, and also the sections between the protrusions of the structured relief.

The appearance of the sample from steel 20Kh13 with the formed polyurethane coating and the resulting surface profile are shown in Figure 3. The thickness of
The polyurethane coating was 330-350 μm. The roughness of the polyurethane coating Ra = 34.6 μm.

Fig. 3. Appearance of the sample from steel 20kH13 with a polyurethane coating (a), surface profile after the formation of the polyurethane coating (b)

- for ion plasma coating:
The formed DLC coating has a thickness of 10-12 μm, the microhardness of the substrate material is 250 ± 30 HV0.2, and the microhardness of the coating is 750 ± 30 HV0.2. Formed based coating Cr-CrC has a thickness of 6-7 μm, the microhardness of the substrate material is 250 ± 30 HV0.2, the microhardness of the coating is 550 ± 20 HV0.2.

- for ion-plasma surface modification (nitriding) without relief and with structured relief:
The surface modification process was carried out for two types of samples - without relief and with a preformed structured relief (see Figure 4). The depth of nitriding of the surface of the samples without relief was 50-80 μm. The selected nitriding depth is confirmed by the results of previously obtained positive tests for comparative erosion resistance. As a first approximation, the nitriding process on the relief samples was carried out to the height of the relief ledges, thus, the textured surface was completely modified to the entire depth of the relief formation. The microhardness of the samples from steel 20kH13 without coating was 250 ± 30 HV0.2; and for samples with nitriding 1050 ± 20 HV0.2. The surface roughness of the samples without relief after the nitriding process was Ra = 0.6 μm

Fig. 4. Experimental samples of steel 20kH13 after nitriding (a), after a preformed structured relief and subsequent nitriding process (b), surface profile after nitriding (c)

For the conditions of the studies, the process of diffusion saturation of the surface to a certain depth mainly depends on the nitriding time. The modified nitrided near-surface layer has a microhardness that is 4 times higher than the microhardness of the substrate material. The transition from increased microhardness of the nitried layer to the microhardness of the substrate is smooth and consistent with the revealed modification depth.

The nitriding process on the relief samples was carried out to the height of the relief ledges, thus the textured surface was completely modified to the entire depth of the relief formation. The increased microhardness is preserved to the entire depth of the modified layer of the relief protrusion, then gradually decreases to the microhardness of the base material. This circumstance is possibly due to the fact that the diffusion saturation with nitrogen occurs locally, with respect to each individual groove (on the transverse section — the protrusion), in contrast to the untreated surface. Therefore, the processing process is more intense due to
the higher specific energy density interacting with the surface.

- for ion-plasma coating followed by the formation of a structured relief:

Structuring the relief on the surface of a sample with a pre-formed ion-plasma coating creates regions that, when burned with a laser beam, are filled with a mixture of molten base material and coating material. Uniform filling of the troughs leads to additional hardening of the surface, removing potentially hazardous areas that can serve as catalysts during subsequent kaleudarny erosion processes. The microhardness of this mixture is higher than that of the base material, but less than that of the coating, it remains throughout the depth of the trench and gradually decreases to the microhardness of the base material.

- for a structured surface topography followed by the formation of an ion-plasma coating:

After creating a structured relief in the process of coating formation, the frozen molten droplets on the surface make changes to the layered structure of the coating, reducing its adhesion and creating surface areas with low coating adhesion, which could potentially affect the erosion resistance of the coating.

By using a Tescan Mira3 LMU scanning electron microscope, images of metallographic sections of samples with various surface treatment options were obtained (see table 1).

| Processing type | Scale: 200 µm | Scale: 50 µm | Characteristics |
|-----------------|---------------|--------------|-----------------|
| Structured surface relief | ![Image](image1.png) | ![Image](image2.png) | Relief: depression depth - 70 µm; width of the ledge - 70 µm; width of the depression - 25 µm. Direction of the grooves: transverse. Substrate: steel 20kH13. |
| Ion-plasma surface modification (nitriding) | ![Image](image3.png) | ![Image](image4.png) | Depth of modification 50-80 µm, microhardness of unmodified substrate material 250±30 HV0.2, modified layer 1050±20 HV0.2. Roughness Ra = 0.6 µm. Substrate: steel 20kH13. |
| Structured surface relief with ion-plasma modification (nitriding) | ![Image](image5.png) | ![Image](image6.png) | Modification depth 65-70 µm, microhardness of unmodified substrate material 250±30 HV0.2, 1000±20 HV0.2. Relief: depression depth - 70 µm; width of the ledge - 70 µm; width of the depression - 25 µm. Direction of the grooves: crosswise. Substrate: steel 20kH13. |
| Structured polyurethane-coated relief | ![Image](image7.png) | ![Image](image8.png) | Thickness of polyurethane coating is 330-350 microns. Roughness Ra = 33.4 . Relief: depression depth - 70 µm; width of the ledge - 70 µm; width of the depression - 25 µm. Direction of the grooves: crosswise. Substrate: steel 20kH13. |
4 Conclusions

1. As a result of the work, several variants of samples with a hardened surface were obtained by using various methods, such as relief structuring, surface modification, polyurethane coating formation and the formation of an ion-plasma coating on the surface before and after creating a relief on it.

2. A complex of studies of the properties of the obtained samples was carried out, including the surface morphology and the effect on the microstructure and characteristics of the surface layer of various conglomerates of the considered methods.

3. The considered options for surface hardening show their potential suitability for increasing the erosion resistance of the base material of the substrate (steel 20kH13), which requires further studies with varying characteristics of the modified layers and erosion resistance tests.

The results of the work were obtained with the financial support of the Ministry of Education and Science of the Russian Federation in the framework of the grant of the Russian Federation President for state support of young Russian scientists No. MK-3377.2019.8. (Agreement № 075-15-2019-334 from 11.06.2019).

References

1. M. Ahmad, M. Schatz, M.V. Casey An empirical approach to predict droplet impact erosion in low-pressure stages of steam turbines Wear, 402–403, 57–63 (2018)

2. W. Su, T. Zhou, P. Zhang, H. Zhou, H. Li, Q. Sui Effect of the orientation of laser stripes on the abrasion resistance of biomimetic laser textured surfaces. Optics and Laser Technology, 107, 380-388 (2018)

3. H. Nomoto Solid particle erosion analysis and protection design for steam turbines. Advances in Steam Turbines for Modern Power Plants, 219-239 (2016)

4. A.M. Emelyanenko, F.M. Shagieva, A.G. Domantovsky, L.B. Boinovich Nanosecond laser micro- and nanotexturing for the design of a superhydrophobic coating robust against long-term contact with water, cavitation, and abrasion. Applied Surface Science, 332, 513–517 (2015)

5. P. Bizi-bandoki, S. Valette, E. Audouard, S. Benayoun Time dependency of the hydrophilicity and hydrophobicity of metallic alloys subjected to femtosecond laser irradiations. Applied surface science, 273, 399–407 (2013)

6. M.-K. Tang, X.-J. Huang, J.-G. Yu, X.-W. Li, Q.-X. Zhang Simple fabrication of large-area corrosion resistant superhydrophobic surface with high mechanical strength property on TiAl-based composite. Journal of Materials Processing Technology, 239, 178–186 (2017)

7. G. Ilieva Mechanisms of water droplets deposition on turbine blade surfaces and erosion wear effects. Journal of Applied Fluid Mechanics, 10(2), 551-567 (2017)

8. P. Wieciński, J. Smolik, H. Garbacz, K.J. Kurzydlowski, Erosion resistance of the nanostructured Cr/CrN multilayer coatings on Ti6Al4V alloy. Vacuum, 107, 277–283 (2014)

9. P. A. Mouche, C. Ang, T. Koyanagi, P. Doyle, Y. Katoh Characterization of PVD Cr, CrN, and TiN coatings on SiC. Journal of Nuclear Materials, 527, Article 151781 (2019)

10. M.A. Khan, M. Duraiselvam, S.S. Panwar, T. Jena, S.R. Dhineshkumar Thermo-mechanical characterization of laser textured LaMgAl11O19/YSZ functionally
11. I. Gulyaev, V. Kuzmin, E. Kornienko, S. Vashchenko, D. Sergachev Microstructure Formation Properties of ZrO2 Coating by Powder, Suspension and Liquid Precursor Plasma Spraying. Materials Today: Proceedings, 11, 430-435 (2019)

12. F. Ghadami, A.S.R. Aghdam Improvement of high velocity oxy-fuel spray coatings by thermal post-treatments: A critical review. Thin Solid Films, 678, 42-52 (2019)

13. G.T.P. Azar, C. Yelkarasi, M. Ürgen The role of droplets on the cavitation erosion damage of TiN coatings produced with cathodic arc physical vapor deposition. Surface and Coatings Technology, 322, 211-217 (2017)

14. B. S. Mann, Vivek Arya HVOF coating and surface treatment for enhancing droplet erosion resistance of steam turbine blades. Wear, 254 (7-8), 652-667 (2003)

15. L. Cao, C. Tu, P. Hu, S. Liu Influence of solid particle erosion (SPE) on safety and economy of steam turbines. Applied Thermal Engineering, 150, 552-563 (2019)

16. H. Bagheri Esfe, M. J. Kermani, M. Saffar Avval Effects of surface roughness on deviation angle and performance losses in wet steam turbines. Applied Thermal Engineering, 90, 158-173 (2015)

17. Y. Zhu, J. Xiong, Y. Tang, Y. Zuo EIS study on failure process of two polyurethane composite coatings. Progress in Organic Coatings, 69 (1), 7-11 (2010)

18. S. Bhargava, M. Kubota, R.D. Lewis, S.G. Advani, A.K. Prasad, J.M. Deitzel Ultraviolet, water, and thermal aging studies of a waterborne polyurethane elastomer-based high reflectivity coating. Progress in Organic Coatings (79), 75-82 (2015)

19. Š. Cakić, G. Nikolić, Č. Lačnjeva, M. Gligorić, M.B. Rajković The thermal degradation of aqueous polyurethanes with catalysts of different selectivity. Progress in Organic Coatings, 60 (2), 112-116 (2007)

20. A.B. Tkhabisimov, A.F. Mednikov, M.R. Dasaev Laser emitting parameters influence during the formation of a regulated micro - nanoscale relief to change the steel surface properties. Natural and Technical Sciences, 2, 274 – 282 (2018)

21. A.B. Tkhabisimov, A.F. Mednikov, M.R. Dasaev, On influence of laser radiation parameters in the formation of ordered micro-nanoscale relief on the change of steel damage properties. Natural and technical sciences, 12, 274-283 (2018)

22. G. V. Kachalin, A. F. Mednikov, A. B. Tkhabisimov, E. A. Zhukova, State-of-the art, problems and methods to improve erosion resistance of materials used for manufacturing of turbines. Research Journal of Pharmaceutical, Biological and Chemical Sciences, 7(5), 955-63 (2016)

23. Kachalin G V, Mednikov A F, Tkhabisimov A. B. Possibilities of the application of ion-plasma technologies to improve the wear resistance of the functional surfaces of power equipment elements. WIT Transactions on Ecology and the Environment, 186, 729-37 (2014)