

# Influence of ion-beam treatment on structure and deformation resistance of 12Cr1MoV steel under static, cyclic and dynamic loading

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**Abstract.** Features of modification of structure and properties of 12Cr1MoV steel subjected to ion-beam irradiation by zirconium ion beam have been investigated with the use of optical and electron microscopy, and microhardness measurement. It was shown that after the treatment the modification occurs across the entire cross-section of specimens with the thickness of 1 mm. Changes in mechanical properties of these specimens under static, cyclic and impact loading were interpreted in terms of identified structure modifications.

## 1. Introduction

Heat resistant 12Cr1MoV steel is used for the manufacturing of energetic equipment parts operating at temperatures of 570 ... 585 °C under action of (thermal)elastic-plastic loads. One of the main reasons of such elements failure is fatigue fracture due to the thermo-mechanical effect [1, 2]. Ion treatment for a long time has been used for surface layer modification to obtain new structural-phase states there [3]. A new method of treatment by irradiation of the beam of metal ions, providing more deeper structure modification in contrast with traditional ion implantation has been developed in the ISPMS RAS. However, the impact of Zr ions beam is accompanied by heating the surface layer of the steel and formation of new phases of iron with zirconium.

The purpose of the study is to investigate the influence of vacuum-arc ion beam treatment on the structure and mechanical properties of heat resistant 12Cr1MoV steel. For a comprehensive assessment of the effect specimens were tested under static and cyclic tension and impact bending on the machine equipped with a instrumented striker.

## 2. Materials and research methods

12Cr1MoV steel specimens for testing were cut out of a tube fragment by the wire electroerosion machine, and then subjected to a standard heat treatment: normalization at 960 ... 980°C and subsequent high tempering at 740 ... 760°C [4]. Further, these specimens are referred to as initial state ones.



Tensile tests were carried out for dog-bone shaped specimens with the dimensions of gauge length of  $20 \times 5 \times 1$  mm. Specimens with dimensions of  $55 \times 10 \times 8$  mm were prepared in accordance with Russian Federation standard GOST 9454 to determine the impact toughness. The tests were carried out for 3 temperatures: ambient –  $20^\circ\text{C}$ , increased –  $375^\circ\text{C}$  and high –  $600^\circ\text{C}$  (that is similar to study conducted by the authors earlier [5]).

Flat specimens with the size of  $70 \times 10 \times 1$  mm were used for the cyclic tensile testing. There is a stress concentrator in the form of a central hole with the diameter of 2 mm. Specimens of the same shape were used for the cyclic alternating bending tests. The following are the basic parameters of the tests: cyclic tension (maximum load – 320 MPa, minimum – 115 MPa, frequency – 15 Hz); cyclic alternating cantilever bending (range – 6 mm, shoulder – 51 mm, frequency – 9.5 Hz). Ion-beam treatment of specimens was performed with the use of high-current vacuum-arc source of metal ions UVN-0.2 "Quantum" [6]. According to data of pyrometric monitoring during the treatment the surface layer of a specimen experiences a short-term heating up to the temperatures of 600 ... 900  $^\circ\text{C}$ .

However, due to the rotation relative to the ion source, the specimens were only for a short time exposure to the effect of the beam that made it possible to avoid continuous heating (taking place at the moment of interaction of specimens with the ion beam) and the consequent loss of strength (tempering). To a certain extent one can say that in addition to the exposure, the specimens were undergoing cyclic thermal impact which would lead to structural changes far beyond the thin surface layer where zirconium ions can penetrate.

Tests on static tension were performed with the help of electromechanical testing machine Instron 5582. Impact tests were conducted on motorized pendulum impact machine Instron 450 MPX equipped with a instrumented striker. Cyclic tension tests were carried out on a servo-hydraulic testing machine Biss UTM 150. Microhardness measurement was performed with the use of PMT-3 microhardness meter with a load onto Vickers pyramid of 0.49 N (50 g).

### 3. Study of the modified layer microstructure

The microstructure of the specimen without the treatment represents a mixture of ferrite-pearlite grains (figure 2, b). After the ion beam treatment not only a thin surface layer was modified, whose formation is associated with the possible penetration of zirconium ions, but also with noticeable changes in the structure of the material core. The modified surface layer to a depth of 130  $\mu\text{m}$  when etching has a distinct white color and is characterized by grains of substantially large size (up to several tens of microns, figure 2, c). Its interface with the underlying substrate layers (core) is not clearly defined.

The reason for the grain size growth from  $29 \pm 3 \mu\text{m}$  in the as-supplied state specimens to  $48 \pm 6 \mu\text{m}$  in the surface layer of this steel can be assumed due to the heating to high temperatures (up to  $900^\circ\text{C}$ ). On the other hand, keeping the original grain structure of the core can be explained by periodic cooling of the specimens due to their rotation relative to the ion beam (source). Moreover, thermal cycling during the treatment came to decrease in the grain size from  $29 \pm 3 \mu\text{m}$  to  $d \leq 20 \mu\text{m}$ . Analysis of microimages showed that this structure corresponds to the ferrite-sorbite mixture.

It is shown that the microhardness of specimens without the treatment is equal to  $1.67 \pm 0.1$  GPa while the value after the treatment varies considerably at different distances from the surface (figure 1, a). In the measurement of the microhardness through the cross section (on the lateral face) there was revealed its reduction to a depth of 70 ... 100  $\mu\text{m}$  from the irradiated surface and is equal to  $1.45 \pm 0.04$  GPa. At the same time, at a depth more than 100  $\mu\text{m}$  microhardness has increased to about  $2.16 \pm 0.1$  GPa and remains at approximately constant level that exceeds one of specimens in as supplied state ( $H_\mu \sim 1.7$  GPa). Thus, there is a softening of the surface layer to a depth of about 100 ... 150  $\mu\text{m}$  in the specimen after the treatment, while it was deeper microhardness contrary increased by  $\sim 22\%$ .

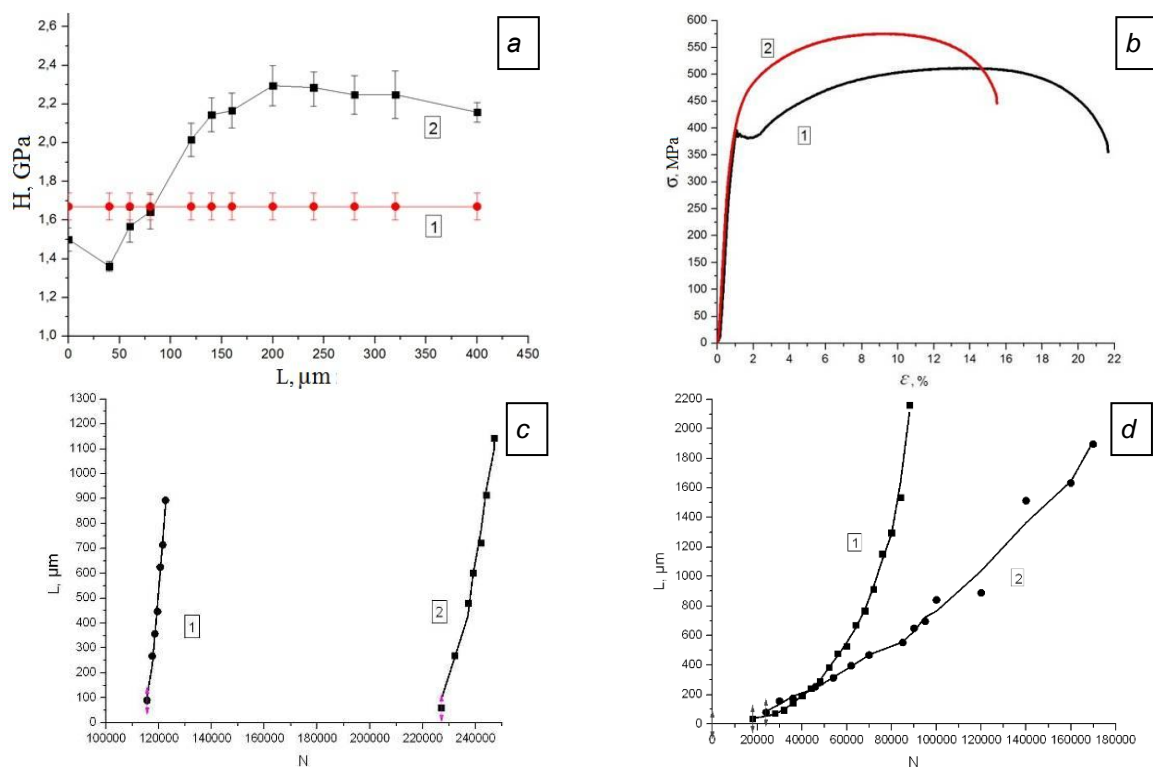
### 4. Results of tests

*4.1. Tests on static tensile.* The presence of yield tooth on the loading diagram is characteristic for material without the treatment that is typical for deformation of low carbon steels, figure 1, b. Yield point

for such specimens makes  $\sigma_{0.2} = 387 \pm 23$  MPa, tensile strength  $\sigma_U = 495 \pm 35$  MPa, and value of elongation at failure  $\varepsilon = 20 \pm 3$  % which is close to one for the standard data of the steel [5]. After the irradiation the tensile strength value  $\sigma_U = 570 \pm 17$  MPa and value of elongation at failure  $\varepsilon = 16 \pm 0.7$  %; in doing so there is no formation of the yield plateau in the specimens after the treatment. Thus, after the irradiation the tensile strength was increased by  $\Delta\sigma = 76$  MPa (15 %), and values of relative elongation decreased by  $\Delta\varepsilon = 4$  % (19 %).

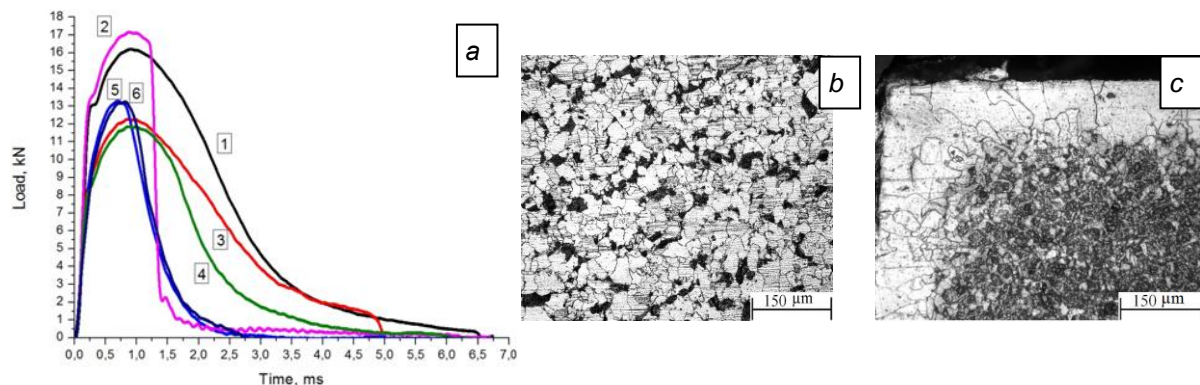
**4.2. Cyclic testing.** Data of the cyclic tensile tests showed that the number of cycles prior to failure of specimens after the  $Zr^+$  ion beam treatment was increased twice, as well as the time before the crack nucleation (also,  $\sim 2$  times), figure 1, c. For specimens after the treatment the characteristic feature is formation of small microcracks on a surface, while the strain relief is formed in a much less degree. Formation of obvious sliding traces within individual grains is evident in specimens without the treatment.

According to the test results, the fatigue life of specimens under the cyclic alternating bending increased twice due to the treatment by  $Zr^+$  ion beam, figure 1, d. The strain induced relief of these specimen looks smoother. Fatigue failure is accompanied by a significantly lower crack opening in comparison with the non-treated specimens.



**Figure 1.** a) Changes in microhardness as a function of the distance from irradiated surface; b) stress-strain curve for tensile testing of 12Cr1MoV steel specimens of dog-bone shape; dependence of crack length vs the number of cycles; c) cyclic tension; d) alternating bending; 1) without the treatment; 2) after the irradiation.

**4.3. The impact test.** Investigation of the microstructure for all types of specimens was carried out. It was found that with increasing the temperature the grain size of the specimen in initial state does not change. At the same time, after the treatment the grain size in the specimen core is not substantially changed, while it is increased in the surface layer. The depth of the modified surface layer makes 120  $\mu m$ . The average grain size of the modified surface layer is  $27 \pm 3$   $\mu m$ .



**Figure 2.** a) diagram of impact loading; 1), 3), 5) specimen without the treatment, 2), 4), 6) after the treatment; Testing temperature – 20°C (1, 2); 375°C (3, 4); 600°C (5, 6); optical micrographs of 12Cr1MoV steel specimen cross sections in as supplied state (a) and after the ion-arc treatment by the Zr<sup>+</sup> beam (b).

Microhardness measurement on the irradiated surface and through the cross section of the treated specimens was performed. For all specimens it was revealed reduction in microhardness values in surface layer at a depth of about 200 μm which agrees well with metallography data. However, at the depth of 200 ... 1000 μm strain hardening due to cyclic thermal influence during the treatment is registered. Impact test of specimens were carried out at the temperatures of 20°C, 375°C and 600°C. It was shown that the ion-beam treatment results in a substantial decrease in toughness mostly at room temperature.

Investigations of impact toughness of the material in an initial state and after treatment were fulfilled. Recorded diagrams of specimen impact fracture at different temperatures are presented in figure. 2, a. Impact toughness values (KCV) were recalculated on the base of instrumented striker data. Test results showed that the impact toughness of the specimens after the surface treatment was reduced by 5 ... 42 %. The maximum decrease in toughness was observed at 20°C. This is due to softening of surface and subsurface layers, and hence a decrease in energy intensity of crack origin under dynamic loading [7]. Simultaneously, ductility of the specimen core is reduced, that is proved by the microhardness measurement data.

## 5. Conclusion

It was revealed the treatment by the zirconium ion beam irradiation of 12Cr1MoV steel specimens with the thickness of 1 mm the structure modification occurs through the entire cross section. There is decrease in microhardness values in the surface layer at the depth up to 100 μm, while in the core it is by 23 % higher than that in the non-treated specimens. Hardening of the core influence the results of the static tension tests: an increase in tensile strength by 15 % and reduction of elongation by 19% takes place. Under fatigue tests by the cyclic tension scheme it was revealed that modification of the structure during the treatment gives rise to delay in the development of localized plastic deformation prior to the origin and growth of the main fatigue crack that is manifested primarily in reducing of the mean value of shear strain intensity. As a result, the increase of the fatigue life time of the modified 12Cr1MoV steel specimen can make 2 times.

Under impact loading of the ion-beam treated specimens, the impact toughness at ambient temperature is decreased by 42 %. Heating up to 375 °C and 600 °C give rise to some structural changes that is accompanied nearly equal value of KCV for non-treated and irradiated specimens.

Offered method of the treatment cannot be characterized as a way of surface modification since locally arising high temperatures lead to a change in the structure through the entire cross section. The observed effects of the mechanical properties changing under static and cyclic loading should be interpreted with consideration of a loaded specimen as a multilevel system: surface layer alloyed with zirconium (a few μm) – thermally "weakened" layer (up to 100 ... 130 μm) - hardened one due to cyclical

short-term thermal effect of substrate material. In so doing this multi-level system becomes the most effective to resist crack nucleation and growth under cyclic loading.

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