Structural Levels of Deformation and Failure of Heat-Resistant 12Cr1MoV Steel Modified by Vacuum Arc Treatment by Zr^{+} Ion Beam

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Abstract. Study of structural changes occurring in the surface layer modified by ion-beam irradiation was carried out by means of optical, scanning and transmission electron microscopy. It was shown that irradiation induces the structure modification not only in the surface layer, but along the entire cross section of 1 mm thick specimens. It was elucidated that the complex pattern of structural changes is responsible for the pronounced variation of mechanical properties taking place under static tension and cyclic alternating bending.

Keywords: fatigue durability, vacuum arc treatment, ion beam, deformation, fracture

INTRODUCTION

Surface modification is the effective method for preserving and improving physical and mechanical properties of structural materials. At the same time, under mechanical loading, the difference in elastic moduli of the modified surface layer and substrate gives rise to the generation of stress concentrators that after relaxation may be followed by the development of shear bands (localized plastic deformation bands) or formation of (micro) cracks [1]. Under cyclic loading, such dissimilarity of properties, as a rule, gives rise to microcracking of the surface hardened layer when the microcracks tends to act as structural micronotches [2, 3]. Hence, it is important to reveal the modes and parameters of the surface layer modification that will provide for a certain compromise between strength, ductility, thickness, cracking resistance, etc.

Some modes of surface treatment by vacuum-arc irradiation implementing metal ions have been developed in ISPMS SB RAS. More specifically, Zr-ions were employed for the treatment that comprised overheating accompanied by the formation of new Zr-containing chemical compounds. The modified surface layer was characterized by a certain decrease of microhardness, however the fatigue life of specimens significantly increased [4]. The aim of this study is to analyze the structure, mechanical properties and deformation behavior of 12Cr1MoV steel specimens after the treatment.

EXPERIMENTAL

The material under study was represented by a heat resistant 12Cr1MoV steel designed for operation at high temperatures (570°C–585°C) [5] since authors try to minimize the thermal influence (local overheating) occurring under vacuum arc ion-beam treatment. Flat specimens with the gauge length of 70×10×1 mm were cut from a pipe fragment by the electro-spark method.

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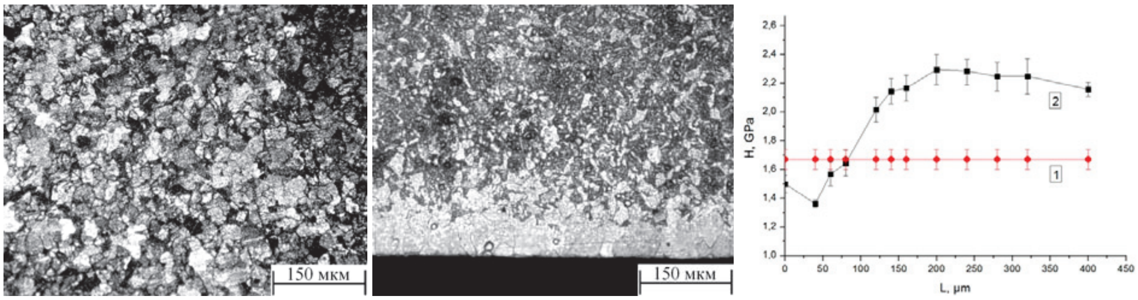

(a) (b) (c)

FIGURE 1. Optical micrographs of specimens: (a) in the initial state and (b) after the ion beam treatment, (c) is dependence of microhardness on the distance from the specimen surface: without the treatment (*1*) and after the ion-beam irradiation (*2*)

Irradiation of specimens by Zr^+ ions was carried out with the help of high current vacuum-arc source of metal ions using "Kvant" installation [6]. According to verified data the surface layer of the specimens experienced intermittent heating up to the temperatures of 800°C–900°C during the treatment. However, due to the set-up capability of mechanical rotation of specimens in relation to the ion source the samples were periodically exposed to the beam. Since the heating takes place only when the ion-beam affects the surface, this allowed to avoid continuous heating (i.e. the time of specimens interaction with the ion beam was limited) and the resulting softening (high tempering).

It must be stressed that besides the ion beam irradiation during the treatment, the specimens were subjected to the thermal cycling, which should have induced the structural changes not only in a thin surface layer where the zirconium ions were able to penetrate. Fractography study was carried out with the use of JSM-7500FA and Quanta 200 3D scanning electron microscopes. The microhardness measurement was performed using PMT-3 micro-hardness meter. Mechanical tests for static tension were carried by Instron-8852 electro-mechanical testing machine.

STUDYING THE STRUCTURE OF A MODIFIED LAYER

Figure 1(a) shows the optical micrograph of the nontreated specimen microstructure that can be characterized as a combination of ferrite and pearlite grains. In order to estimate structural changes that took place under the irradiation, the transverse cross section was polished and etched. This allowed estimating the thickness (depth) of the modified layer (Fig. 1(b)). As it was expected, the structure modification during the treatment happens not only in the layer with a several micron thickness were Zr ions can penetrate. One can observe the formation of a modified (non-etched - white) layer that is characterized by quite coarse grains (up to several tens microns). Visually, it can be estimated to be from 90 to 130 μm thick, whereas a pronounced interface with the underlying layers of the substrate (core) is hard to detect. The reason for such a substantial growth of the grain size in the surface layer for this particular heat resistant steel might be explained by a (cyclic) heating to high temperatures (at least 900°C). On the other hand, the thermal cycling took place in the specimen core during the treatment, which resulted in the structure fragmentation and reduction of the grain size from 30–50 μm (Fig. 1(a), non-irradiated specimen) down to ≤20 μm (Fig. 1(b)). The optical micrograph analysis testifies this structure to be mostly comprised of sorbite (or troostite).

The structural changes that tool place after the irradiation resulted in the increased microhardness as compared with the nontreated specimens. As it is seen form Fig. 1(c), before the treatment the microhardness H_μ = 1.67 ± 0.1 GPa, while after the treatment it changes substantially, the change magnitude depending on the distance from the surface. On the flat surface that served as the site for ion-beam impact the microhardness H_μ amounted 1.5 ± 0.1 GPa. This testifies the temperature during the treatment to be significantly higher in contrast with operating one for this particular type of steel (not higher than 600°C). Thus, the softening took place. During the measurement done at the lateral face at the distance of 100 μm from the surface, the decreased value of microhardness H_μ was determined to be 1.45 ± 0.04 GPa. These results agree well with the observations of transverse cross section (Fig. 1(b)). At the same time, at the depth of more than 100 μm, one can observe the increase of H_μ up to 2.16 ± 0.1 GPa. Then it remains nearly constant, which is higher in comparison to the initial specimen microhardness (H_μ = ~1.7 GPa). Thereby, a softened surface layer that was formed during the treatment occupies the depth of 100–150 μm, while the microhardness in the core layers is contrary increased by approximately 22%.

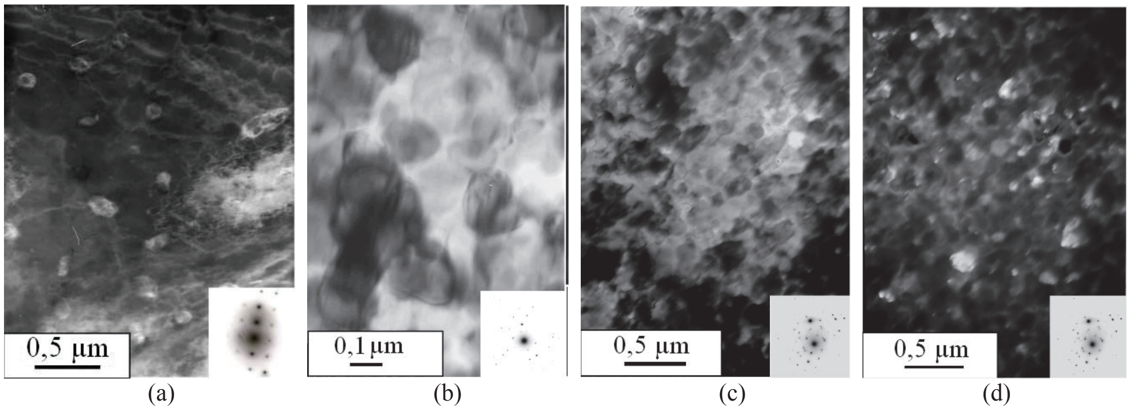

FIGURE 2. TEM micrographs illustrating the structure of the surface layer of 12Cr1MoV steel: (a, d) are dark field images; (b, c) are bright field images; (a, b) are samples before the treatment; (c, d) are samples after the treatment

We presume that the latter result is obviously related to the thermal cycling effect conditioned by the irradiation and the grain refinement.

Fine structure of surface layer was examined by means of TEM. As it can be seen from TEM-micrographs, the structure of the initial steel is represented by large ferrite grains with the size of more than 1 μm and with inclusions of cementite (Fe_3C) having the average size of 120 nm (Fig. 2(a)). After irradiation by the Zr^+ ion beam, the surface layer structure is represented by $FeZr_3$ and $FeZr_2$ phases, as well as ferrite grains. The average size of the grains reaches 100–150 nm (Figs. 2(b–d)).

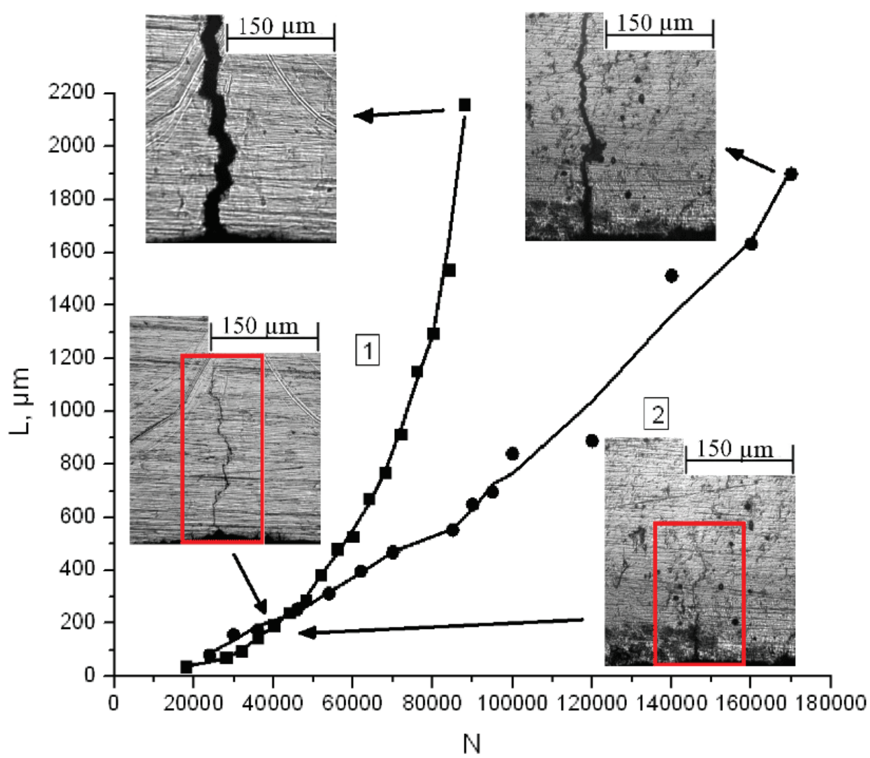

FIGURE 3. Fatigue crack length as the function of cyclic loading in specimens without the treatment (*1*) and after the ion-beam treatment (*2*)

The static tension testing was carried out with the use dog-bone shaped specimens. It was shown that the yield strength of nontreated specimens reaches $\sigma_{0.2}$ = 387 ± 23 MPa, the ultimate strength σ_u = 494 ± 36 MPa and the elongation at failure ε = 20 ± 3%. These data are close to reference ones for this type of steel [7]. After the irradiation, the ultimate strength σ_U is equal to 570 ± 17 MPa, while elongation at failure ε = 16 ± 0.7%. It also should be noted that specimens after the treatment do not exhibit yield plateau in the loading diagram. Thus, after the treatment the tensile strength increased by 76 MPa (15%), while the elongation at failure decreased by 4 (19%).

The cyclic alternating bending tests were carried out. Figure 3 demonstrates the optical micrographs of specimen surface captured during the mechanical testing and plots representing the dependence of crack length on the number of loading cycles (Fig. 3). One can see that in specimens of both type the crack occurs after almost the same number of cycles, while the rate of fatigue crack growth in the specimen without the treatment is much faster. Moreover, the optical images characterizing the specimen surface in the vicinity of a stress concentrator (notch) zone are given for the initial stage of loading (shortly after the crack occurrence) and prior to the fracture. Comparison of the optical micrographs allows to suggest that the rapid development of deformation on cyclic bending is hindered because of the irradiation (and most probably due to the formation of the modified surface layer of several microns thick), which is responsible for the increased fatigue life. This correlates well with the surface roughness measurement data. The analysis of the surface roughness in the vicinity of fracture zone was carried out by New View 6200 optical interferential profilometer. According to the data obtained, the surface roughness *Ra* of the non-treated specimen is 1.5 times higher (*Ra* = 0.710 μm) as compared to that of the irradiated sample (*Ra* = 0.490 μm).

CONCLUSION

It has been elucidated that during the vacuum-arc ion-beam treatment by the zirconium beam, the structure modification of the 12Cr1MoV steel specimens takes place and varies along the cross-section. At the distance of 100–120 μm, the microhardness decreases, which is related to local overheating. However, in the specimen core it increases, which may come from cyclic pattern of thermal influence. The technique used cannot be characterized as the method for surface modification, since the reached high temperatures give rise to local structure changes along the entire cross section (1 mm for the specimens used in this study).

Thus, the observed effects of mechanical properties changing under static and cyclic loading can be interpreted from the point of view of loaded specimens to be multilevel (layered) systems, i.e. subsurface layer alloyed by the zirconium (a few μm thick), thermally "softened" (tempered) layer (100–120 μm), substrate (core) strengthened layer due to short-term thermal cycling. Such multilevel system exhibits satisfactory resistance to mechanical loading and alternating bending, since the crack propagation is hindered, while the flow stress is enhanced.

ACKNOWLEDGEMENTS

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