Zr^+ ION BEAM SURFACE IRRADIATION FOR IMPROVING FATIGUE DURABILITY OF 12Cr1MoV AND 30CrMnSiNi2 STRUCTURAL STEELS

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Abstract
A cyclic tension and alternating bending tests of 12Cr1MoV and 30CrMnSiNi2 steels specimens in as–supplied state and after surface nanostructuring by Zr+ ion beam have been carried out. Distinctions in formation of strain induced relief, as well as the cracking pattern of modified surface layer are illustrated by methods of optical microscopy and interferential profilometry. Changes to occur in subsurface layer are characterized by means of nanoindentation and fractography (scanning electron microscopy) of fracture surfaces. The description of differences of deformation behavior is carried out with use of the multilevel approach.

Keywords: surface modification, nanostructuring, multiple cracking, deformation, fracture

1 Introduction

The majority of machine parts and structure elements during their exploitation experience the impact of variable loads, which can give rise to their fatigue fracture. In doing so regardless the long history of study the problem of fatigue fracture and approaches to increase fatigue durability are of substantial importance [1]. Surface modification is an effective way both to protect and to improve the mechanical properties of structural materials. On the other hand, under mechanical loading the distinction of elastic modules of a modified surface layer and adjacent bulk material causes the stress concentrators to occur, whose relaxation may give rise to localized development of plastic deformation or fracture [2-4]. Under cyclic loading such distinction of the properties brings to microcracking within strengthened surface layer, to act as structural micronotches. In doing so, development of techniqus and modes for modifying surface layer should be a compromise between strength/ductility of this layer and its thickness (this as well depends on several other factors, including adhesion etc.) [5].

Ion implantation technique for a long time is widely used for industrial applications and is an extensively studied process [6-8]. Recently, with the use of vacuum arc ion sources the modes of surface layer nanostructuring which allowed modifying surface layers with a thickness of some microns that is several times higher in contrast with ion implantation at conventionally used regimes were realized. Such nanostructured surface layers are not coatings yet, but no longer can be called as ion-implanted ones. Study of influence of such layers on fatigue life-time of structural steels is of particular interest due to the possibility of simultaneously improve the fatigue durability and wear resistance [9-11]. To study the influence of surface layer

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nanostructuring of 12Cr1MoV and 30CrMnSiNi2 steels by Zr+ ions on the fatigue durability increase is the purpose of this paper.

2 Material and experimental methods

Two types of structural steels were used in the current research for Zr+ ion beam surface modification: heat resistant ductile 12Cr1MoV steel intended for operation at high temperatures as well as 30CrMnSiNi2 high strength steel used for manufacturing of heavy-loaded machine parts [12]. The choice of 12Cr1MoVsteel for research was caused by the fact that the steel experiences no structural changes at the temperature at which the process of surface layer nanostructuring by ion beam is performed. Besides, the steel is quite ductile, so the study of processes of localized deformation and fracture under cyclic load provides much more evidence at lower rate of deformation processes.

High strength 30CrMnSiNi2 steel is used for manufacturing of responsible and highly loaded parts that as well experience action of alternate loadings. Increasing of fatigue durability of this steel is a complex process because of its low heat resistance and high level of alloying. In contrast with 12Cr1MoV steel treatment new regimes for the surface nanostructuring were determined that make possible to slightly decrease mechanical properties as well as to substantially enhance fatigue durability.

Flat specimens with gauge length of 70×10×1 mm for 12Cr1MoV steel were prepared from a pipe fragment by electro-spark cutting. The specimen of 30CrMnSiNi2 steel measure of 70×8×1 mm was cut from a rod ingot. For running fatigue tests the holes of 2 mm in diameter were mechanically drilled as a stress concentrator in the specimens being located at a distance of 50 mm from one of its edges. For static tension tests dog-bone shapped specimens with gauge length of 20×5×1 mm were also employed, as well as ones with the stress concentrators (similar to the specimens for fatigue tests). The specimens were mechanically polished and divided into 2 groups: a) in as-supplied state (without treatment) and b) specimens irradiated with Zr^+ ion beam (after ion beam treatment). In the as-supplied state 12Cr1MoV steel possesses the ferrite-pearlite structure with a characteristic grain size of 30 ÷ 50 μm.

Ion nanostructuring of surface layer of the steel specimens was carried out with a help of high current vacuum arc source of metal ions UVN-0.2 "Quant". Images of specimen surface were obtained by means of optical microscopes Carl Zeiss Axiovert 25 CA and EPIQUANT, as well as scanning electron microscope Carl Zeiss EVO 50. Surface profilometry were performed with the help of Optical Interferometer of white light NewView 6200. X-ray phase analysis was conducted by X-ray difractometer DRON-7.

Tests on static tension were carried out with the use of electromechanical testing machine Instron 5582 while for the cyclic tension servo hydraulic testing machine Biss UTM 150 were employed. Surface micrographs of specimen were captured by Canon D550 digital photo camera during the process of fatigue tests. Nanohardness of specimens was measured by Nanotest (Micromaterials Ltd., UK).

3 Study of the modified surface layer of 12Cr1MoV steel

The structure of the subsurface layer of the steel in the initial state is represented by large ferrite grains >1 μm with inclusions of cementite (Fe_3C), whose average size makes 120 nm (**Fig. 1, a**). subsurface layer structure after nanostructuring by the beam of Zr+ ions is represented by phases of $FeZr_3$, $FeZr_2$, as well as ferrite grains. The average size of the grains in the surface layer makes 100-150 nm (**Fig. 1, b**).

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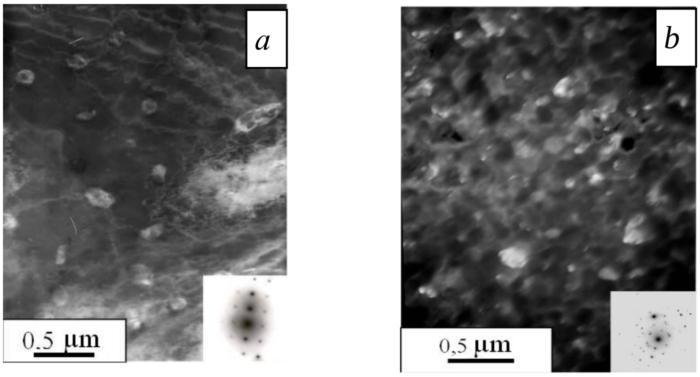

Fig.1 SEM micrographs of the subsurface layer structure of 12Cr1MoV steel: a) initial state; b) after nanostructuring; (dark field)

Microhardness of 12Cr1MoV steel specimens was measured with the load onto the indentor of 5 N. Specimen after ion-beam irradiation were additionally indented onto lateral face being preliminary polished in order to estimate the change of the parameter through the thickness (along the cross section) (**Fig. 3, a**). The data of measurement clearly indicate that that the treated specimen experience some softening of the subsurface layer at the thickness of 100 μm, while in the core the microhardeness possesses increase up to 22% (**Table 1**).

Table 1 Values of hardness

Type of specimen	Hardness, [**GPa**]	Изменения, [**%**]
Initail state	1,67±0,07	-
After irradiation (surface)	1,5±0,06	↓ 10
After irradiation (cross section, core)	2,16±0,05	↑ 22

3.1 Study of the modified surface layer of 30CrMnSiNi2

It was found that during the ion-beam treatment highly dispersed particles with the size of ~100 nm (**Fig 2, a**) were formed on the surface of the irradiated specimens. With the help of the X-ray spectral microanalysis the phase composition of nonirradiated specimens was determined (**Fig. 2,b**) and after the modification (**Fig. 2,c**). One can see that besides the elements being characteristic for the initial chemical composition, some additional peaks occur which testify for the presence of zirconium in the specimen after the treatment. At the depth of 6-8 μm the chemical composition does not differ from one of the untreated specimens.

In **Fig. 2,d** the fine structure of the irradiated specimen over its cross section is presented. It is evident that near the surface the ferrite-cemented structure has formed as a result of heating that is characteristic for sorbite (at the depth of not more than 100 μm). In the underlayers of the specimen the initial martensite structure is remained. With the help of inclined X-ray beam technique that makes it possible to study thin subsurface layers, the presence of the a-Fe phase is revealed in the irradiated specimen, with the lattice parameter a=2.8687 Ǻ and the size of coherent scattering regions of (CSR)=58 nm.

The specimens not subjected to the irradiation are characterized by the presence of the a-Fe phase with the lattice parameter a = 2.8722 Ǻ and the size of CSR = 26 nm. Additionally, intermetallic compound phases such as Zr_3Fe, $FeZr_2$, and so ZrC are observed.

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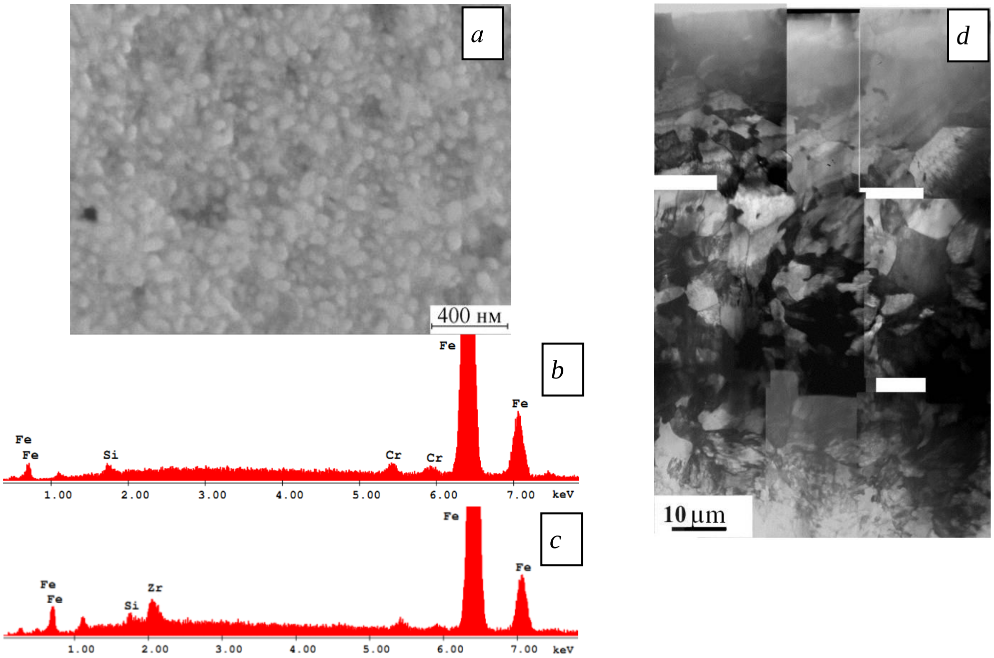

Fig.2 a) SEM -micrograph of the specimen surface after the treatment; data of X-ray spectral microanalysis of the specimen surface b) as-supplied state; c) after the treatment; d) TEM image of the subsurface layer cross section of the specimen after the irradiation

Microhardness of 30CrMnSiNi2 steel specimens was measured with the use of PMT-3 microhardness meter at load applied onto Vickers pyramid of 1 N. The measurements were conducted on the flat surface for all types of specimens. The lateral face (cross section) was additionally analyzed for the treated specimen (see **Table 2**).

Table 2 Values of hardness

Type of specimens	Hardness, [**GPa**]	Changing, [**%**]
Initial state	4.6±0.08	-
After the treatment (flat surface)	2.2±0.06	↓ 52
After the treatment (cross section, core)	5±0.2	↑ 8

As is seen from **Fig. 3** microhardness is changed after the irradiation by zirconium ion beam as the function of the distance from the specimen surface. All the points besides the first, the third and the last ones, were calculated being averaged over 3 indentations, while the first, the third and the last points were based on averaging over 20 measurements. Microhardness in the first point was measured onto the flat surface of the specimen. Mimimum hardness over the cross section was registered close to the surface (at the depth of ~18 um). Microhardness began to grow at the depth of 150-180 µm while after it was lowered and remained constant.

Most probably the carbon during the surface layer recrystallization and the formation of ferrite grains could migrate towards deeper layers that also could give rise to local strengthening at the depth of 150-180 µm. The softened zone extended from the surface to the depth not exceeding 100µm.

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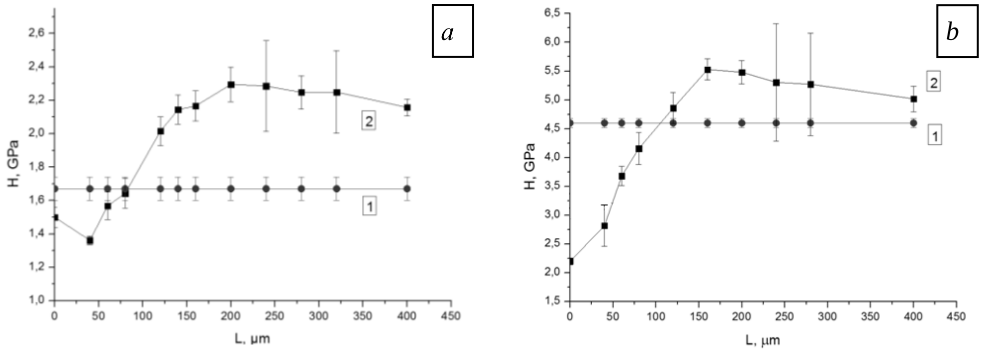

Fig.3 Graph on microharnes through the specimen cross section as function of the distance from the surface of 12Cr1MoV (a) and 30CrMnSiNi2 (b) steel; 1) the specimen without the treatment; 2) after treatment

3.2 Static tension tests of 12Cr1MoV steel

During tensile tests the loading diagram of 12Cr1MoV steel for dog-bone shape specimens was registered (**Fig**. **4**). It was found that for untreated specimens the presence of sharp yield point (yield tooth) is evident like it takes place for low carbon steels. Yield point of such specimens makes σ_{ys} = 270 ± 25 MPa, ultimate strength – σ_{us}= 494 ± 36 MPa and elongation – ε = 20 ± 3 % which is close by values to the reference book data for this steel [14]. After the surface nanostructuring the value of ultimate strength is increased up to σ_{us} = 570 ± 17 MPa while elongation becomes lower ε = 16 ± 0.7 %. In doing so, there is no formation of the yield plateau at the diagram of the processed specimens.

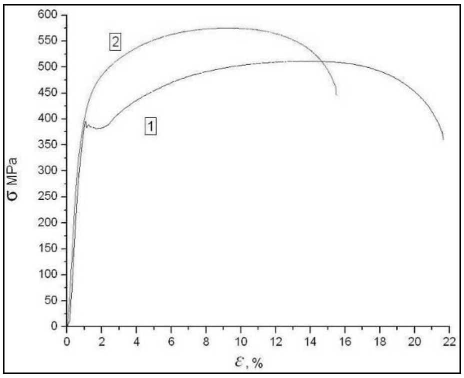

Fig.4 Loading diagram of 12Cr1MoV steel for dog-bone shape specimens: 1) specimen without the treatement, 2) nanostructured one

Microscopic investigations of specimens surface near the area of the main crack at a certain distance from the fracture edge were carried out, **Fig. 5**. It is found that the specimen without surface treatment has the pronounced grain-patterened strain induced relief, **Fig. 5, a**.

At the same time under more detailed observation it is seen that the surface of the nanostructured layer has small microcracks characterized by variation in their orientation in the modified layer (**Fig. 5, b**). To our mind the presence of the nanostructured layer on the surface of 12Cr1MoV steel hinders the formation of the grain relief that resulted in lower surface roughness of such

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specimens. The fracture surface of specimens after treatment shows signs of ductile fracture except for surface layer where the fracture was brittle. It is obvious that the thickness of the layer is not strictly constant which is manifested in the form of varying width of subsurface layer characterized by brittle fracture.

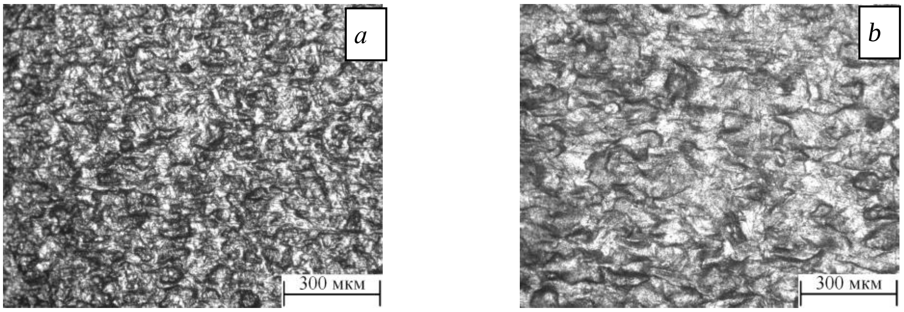

Fig.5 Surface micrographs of failured specimens at static tension tests: a) specimen without treatment; b) after nanostructuring surface; axis of loading is oriented horizontally

3.3 Static tension of 30CrMnSiNi2

The tests on the static tension of the specimens with the central holes were carried out to have the same shape as ones used under cyclic tension (**Fig. 6**). It was shown that the specimens without the treatment had the ultimate strength of σ_{us} = 1628 MPa, relative elongation ε=6%; the specimens subjected to the ion modification had the ultimate strength σ_{us} = 1270 MPa, relative elongation ε = 8%; the specimens after annealing at 700 °C had the ultimate strength σ_{us} = 937 MPa, and the relative elongation ε = 7%. Thus, as a result of the ion treatment of the 30CrMnSiNi2 steel the ultimate strength was lowered by 22 %, relative elongation increased by 25 %; while in the specimens after annealing at 700 °C ultimate strength was lowered by 42 %, relative elongation increased by 17 %.

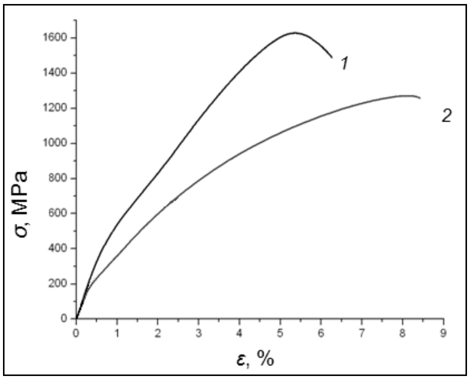

Fig.6 Loading diagrams of 30CrMnSiNi2 steel at specimen tension with central hole; 1) the specimen without the treatment; 2) after treatment

3.4 Cyclic tension tests of 12Cr1MoV

Low-cycle fatigue (LCF). Results of cyclic tension tests have shown that under LCF the number of cycles prior to the fracture of the specimens with nanostructured surface layer is increased by

3 times. In doing so, the time before main crack initiation is increased approximately by3 times. The graphs of dependence of the main crack length versus the number of cycles were plotted. It is clear that for the treated specimen later origination and slower development of the main crack in the specimen is characteristic feature as compared with ones without the treatment. For the latter the rate of crack growth makes 0.085 μm/cycle while for one with nanostructured surface layer it makes 0.023 μm/cycle which is lower by more than 3.5 times. Thus, a modified surface layer effectively hinders the fatigue crack origination. In doing so the number of cycles before the main crack appearance is enlarged by 3 times and reduces the rate of its growth, **Fig. 7**.

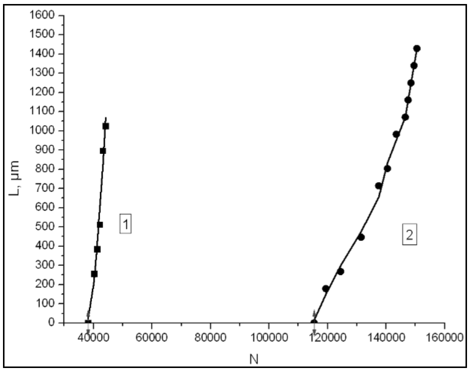

Fig.7 The crack growth diagram as the function of the number of cycles; 1) without treatment; 2) with nanostructured surface layer

High-cycle fatigue (HCF). The results of HCF tensile tests showed that the number of cycles prior to the fracture of specimens with nanostructured surface layer is increased by 2 times. The number of cycles before the crack nucleation is increased approximately by 2 times. Based on analysis of optical micrographs the dependence the crack length on the normalized and the absolute number of tension cycles were calculated and plotted. Much like to the case of the LCF at comparison of the graphs of specimens without treatment and after the surface nanostructuring the difference in time of the main crack origination, as well as the rate of its development becomes visible. Crack growth rate of the specimens without treatment made 0.03 μm/cycle, while for one with the nanostructured surface layer the rate is reduced by 2 times that can be estimated as 0.015 μm /cycle.

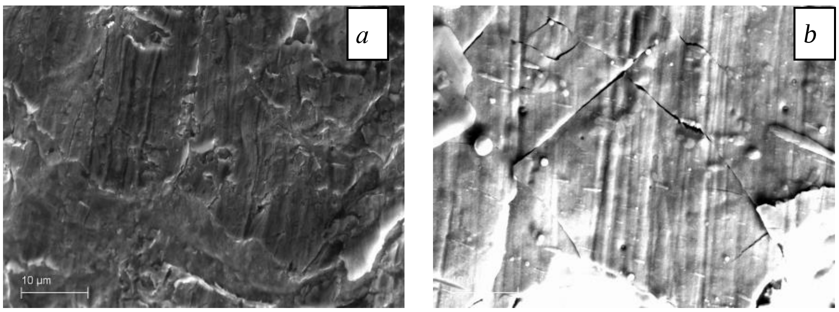

Fig.8 SEM micrographs for the area of fatigue crack growth found at the flat face failured at cyclic tension tests; (a) specimens without treatment; b) with nanostructured surface layer; axis of loading oriented vertically

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Fractography study of specimens fractured under cyclic tension. Micrographs of specimens fractured under cyclic tension were obtained using scanning electron microscope at high magnification, **Fig. 8**. Micrographs gained in fatigue crack growth region, **Fig. 8, b** also point to the brittle pattern of the nanostructured surface layer fracture being compared with untreated specimens, **Fig. 8, a**. It is seen that the surface layer of the nanostructured specimen is damaged by multiple cracks that does not bring to noticeable deformation localization. According to the author’s opinion, multiple cracking allows to redistribute efficiently the loading and minimize the effect of the powerful stress macroconcentrator operating in the vicinity of the main fatigue crack tip.

3.5 Cyclic tension tests of 30CrMnSiNi2

During the tests the average number of cycles prior the fracture were determined. For the specimens without the treatment this value N_p made N_p =110000±31000 cycles while for the specimens after the irradiation – 330000 ± 40000. The specimen after tempering failed after 138000 ± 36000 cycles. Thus, the surface modification by the Zr+ ion beam irradiation of 30CrMnSiNi2 steel specimens ensures increasing the fatigue life-time by 3 times.

The dependence graph of the fatigue crack length vs the number of loading cycles was built being based on the surface image analysis captured during the tests, **Fig. 9**. It is evident that the crack starts substantially later and develops slower in the specimen after the ion treatment. For the specimens without the treatment the rate of the crack growth is L = 0.103 μm/cycle while after the ion treatment L = 0.025 μm/cycle. Thus, the surface layer modification of the specimens by the Zr+ ion beam contributes to the delay of the fatigue crack nucleation by ~3 times, as well as increases the time of its propagation by ~4 times.

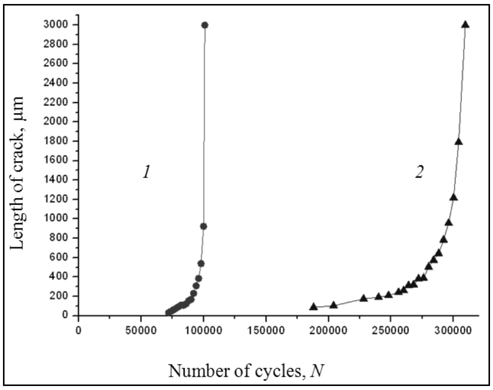

Fig.9 The crack growth diagram as the function of the number of cycles; 1) without treatment; 2) – with nanostructured surface layer

3.6 Test on cyclic alternating bending 12Cr1MoV

According to the testing data the fatigue life-time of specimens under cyclic alternating bending is increased due to nanostructuring of a surface layer by~ 2 times. The series of optical micrographs were used for plotting the graphs to characterize dependence the crack length versus the number of loading cycles. At cyclic bending the main crack originates nearly at the same time for both types of specimens but has significant differences in the propagation rate. Crack growth rate for the untreated specimen was 0.05 μm/cycle while for the specimen with

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modified surface layer it made 0.024 μm/cycle. Thus, it is shown that nanostructuring of surface layer by ion beam to slow the rate of fatiguecrack growth by about 2 times.
Optical observation of strain induced relief. Images of surfaces of both type specimens after the different number of cycles before fracture to cause distinction in the formation of strain induced relief are given in **Fig. 10.** One can see formation of manifested thin folds, **Fig. 10, a** located close to the fracture area (region of maximum curvature) in the specimen without treatment which shows an intensive deformation development in this area. Most likely their presence contributes to a rapid propagation of fatigue crack in this specimen. Reason of formation of these folds is apparently related to the testing scheme that governs periodical application of tensile and compressive stresses [13,14]. Mechanism of material deformation for this case is similar to the corrugation formation which is formed as a result of greater deformation in the subsurface layer. In the nanostructured layer the number of such folds is slightly visible and deformation relief is smoother and changeable more lightly, **Fig. 10, b**.

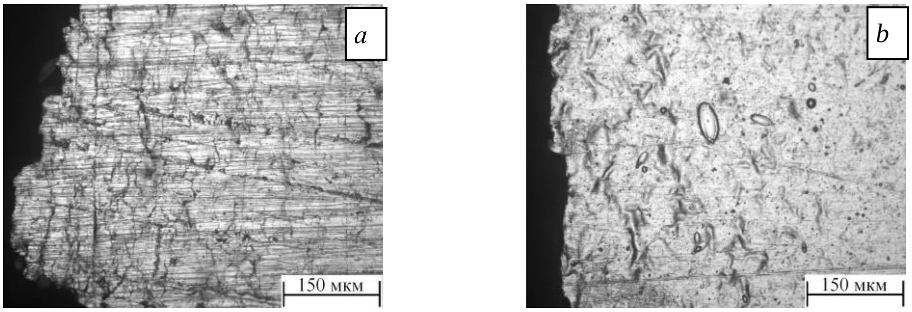

Fig.10 Optical micrographs to illustrate strain induced relief on the surfaceofspecimens: a) without the treatment; b) with nanostructured surface layer

The measurement of surface roughness of failured specimens in the fracture area was carried out with a help of optical interferential profilometer. According to obtained values of roughness parameter Ra the surface roughness of the specimen without treatment is more than 1.5 times higher in comparison with one for the specimen with nanostructured surface layer.
During the test, in the specimen without the treatment clearly pronounced microcracks are formed along the grain boundaries and the main crack is more clearly manifested and propagates just along the microcracks. The specimen after the treatment has the number and size of the microcracks noticeably smaller and the main crack develops to a less extent. More detailed view of the main crack for specimen without treatment testifies for the fact that it is developed intensively not only on the surface, but into the bulk of the specimen as well. The main crack in the nanostructured specimen is less pronounced that could indicate that it develops mainly in the modified surface layer which hinders its spreading into the bulk of the material [15,16]. This may cause a reduction in growth rate under main fatigue crack propagation.

4 Discussion

During the ion-beam tretment of 12Cr1MoV steel a modified surface layer is formed that consists of nanosized zirconium based particles as well as intermetalic phases. In doing so the microhardeness on the surface is decreased by~ 10 %. With further distance from the surface the microhardeness tends to increase to reach the value equal to one in untreated specimen at the

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depth of 100 μm from the top layer. However, due to some structural changes to occur during the treatment the microhardness at the deeper layers exceeds one in the non-irradiated specimen to be 22 % higher at the distance of 130 μm from the surface. It might be summarized like the treatment used gives rise to partial softening of the subsurface layer at the depth of 100 μm and simultaneous strengthenig of the core specimen due to formation of troostite structure with finer grain size. This fact might give the explanation of the data on increasing specimen ultimate strength under tension. In doing so the softened subsyrface layer under static and cyclic tension allows to effectively redistribute the stresses over entire gage length od the specimen to avoid strian localization. At the same time the specimen without the treatment experience the fragmentation due to fast strain induced relief formation under cyclic loading and possess less fatigue durability.

Under cyclic bending the surfece layer in the location to experience the maximum curvature the alternating tensile and compressive stresses acts that give rise to microcracking along grain boundaries, specimen fragmentation, fatigue crack nucleation and failure.

In contrast with above discussed heat resistant steel the high strength 30CrMnSiNi2 steel experince quite different structure changes. In the subsurace layer at the distance of up to 6 μm intermetallic compunds of Fe-Zr system are formed as well as zirconium carbides. Then, at the thicknes up to 35 μm due to action of elivated tempeatures to occur under irradiation the high-temparature tempering takes place that gives rise to ferrite-cementite structure formation with characteristic grain size of 2-5 μm. At the deeper layers for more than 100 μm from the surface the sorbite structure is formed that is characteristic feature for for high temperature tempering to take place at heating above 650 °C. Because of thermal cycling to occur at the ion-beam irradiation the residual austenite transferes into beinite that is responsible for beinite-martinstite structure to be found at the distance more than 100 μm. The latter ensures certain increase of the microhardness in contrast with austenite-martensite structure being characteristic for standard thermal treatment (fig. 6).

The analysis of the results obtained has shown that the ion-beam irradiation gives rise to decrease of ultimate strength due to softening of the subsurface layer that simultaneously was accompanied by increse of elongation at failure. The loading diagrams for specimen with a central hole possess lower degree of the elongation due to the presense of the stress concentrator. At the same time the value of ultimate strength for these specimen coinsides well with one given in reference books.

Much like the case for heat resistant 12Cr1MoV steel the modified subsurface layer provides more uniform distribution of stresses and "heals" dangerous defects on the surface (like scratches, microcracks etc.) whose presence in high-strength steel as a rule ensure large reduction of fatigue durability.

5 Conclusion

The structure investigation of subsrface layer of 12Cr1MoV and 30CrMnSiNi2 steel specimen after the ion-beam arradiation has ben carried out. It is shown that that treatment brings to the formation of the softened subsurface layer with approximate thickness of 100 μm. The core of the specimen also experience the chaging of the hardness that is increased by 22% for 12Cr1MoV steel while for 8% for 30CrMnSiNi2 one.

The tests on cyclic tension have shown that the ion-beam treatment used ensures the increse of fatigue durability by2-3 times in contrast with non-treated specimen. The main reason for the

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revealed changes is softening of the subsurface layer while in contrast the quenched steel specimen are very sensitive to presense and nucleation of microcracks whose evolution usually is completed with fast nucleation and propagation of main fatugue crack.

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