Surface layer modification of 12Cr1MoVand 30CrMnSiNi2 steels by Zr^+ ion beam to improve the fatigue durability

Ilya Vlasov[1,2,*], Sergey Panin[1,2], Viktor Sergeev[2], Alfred Sungatulin[2], Mark Kalashnikov[2], Boris Ovechkin[1], Viktor Panin[1,2]

[1]Department of Material Science in Mechanical Engineering, Institute of High Technology Physics Tomsk Polytechnic University, 634050, Tomsk, Russia

[2] Laboratory of Polymer Composite Materials, Institute of Strength Physics and Materials Sciences SB RAS, 634021, Tomsk, Russia

* Corresponding author: good0@yandex.ru

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 multiple cracking concepts.

Keywords strength, nanostructuring, multiple cracking, deformation, fracture.

1. Introduction

The majority of products and machine parts 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]. Under cyclic loading such distinction of the properties brings to microcracking within strengthened surface layer, to act as structural micronotches. Therefore, the choice of modes and options for modifying the 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.).

Ion implantation technique for a long time is widely used for industrial applications and is an extensively studied process. 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 ion-implanted ones [3]. Study of influence of such layers on fatigue life-time of structural steel specimens is of particular interest due to the possibility of simultaneously improvethe fatigue durability and wear resistance. To study the influence of surface layer nanostructuring of 12Cr1MoV and 30CrMnSiNi2 steels by Zr^+ ions on the fatigue durability increase is the purpose of this work.

2. Materials and research methods

Two types of steel 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 [4]. 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 responsible and highly loaded parts that experience action of alternate loadings. Increasing of fatigue durability of this steel is a complex process since its low heat resistance and high level of alloying. Using the experience of 12Cr1MoV steel treatment new regimes for the surface nanostructuring were determined that make possible to slightly decrease mechanical properties while to enhance substantially fatigue durability.

Flat specimens of size 70×10×1 mm for 12Cr1MoVsteel and 70×8×1 mm for 30CrMnSiNi2 steel were made from a peace of a pipe by electro-spark cutting. For running fatigue tests the holes of 2 mm were drilled as a stress concentrator in the specimens at a distance of 50 mm from one of its edges. For static tension tests specimens in the shape of dog-bone with gauge length of 20×5×1 mm were also used, as well as ones with the stress concentrator (similar to the specimens for fatigue tests). The specimens were mechanically polished and divided into 2 groups: a) in initial state (without treatment) and b) specimens with a nanostructured surface layer by zirconium Zr^+ ion beam (after ion beam treatment). In the initial state steel 12Cr1MoV has 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’s 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 performed using electromechanical testing machine Instron 5582 while for the cyclic tension using servo hydraulic testing machine Biss UTM 150 were employed. Surface micrographs of specimen’s were captured by Canon D550 digital photo camera during the process of fatigue tests. Nanohardness of specimens was measured by Nanotest (Micromaterials Ltd., UK).

2. The results of the experiments

2.1. Study of the modified surface layer of 12Cr1MoV steel

2.1.1. Electron-microscopy study

The structure of the surface 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). Surface 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 is 100-150 nm (fig. 1, b-d).

2.1.2. Specimensnanoindentation

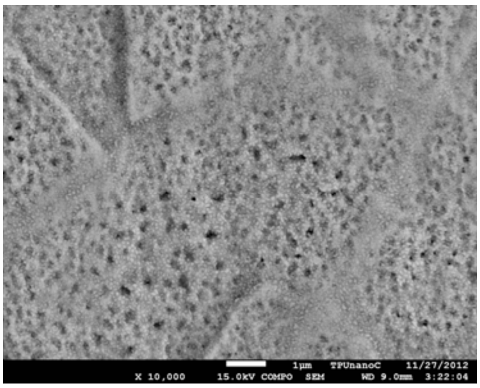
Strength properties of the specimens under study before and after the treatment were evaluated by nanoindentation to penetrate at the depth of 200 nm. It is revealed that due to nanostructuring the hardness of the surface layer increases approximately by 1.5 times. At the same time elastic modulus is reduced almost by 2 times.

2.1.3. X-raydiffraction

For specimens in the initial state structure is primarily represented by the α-Fe iron, whereas after nanostructuring the formation of intermetallic phases of the system Fe-Zr: $FeZr_2$ and $FeZr_3$, as well as the carbides ZrC into the subsurface layer takes place. Structural-phase micro-analysis of the specimen surface with nanostructured surface layer was performed. The data indicate that the overall content of zirconium in the subsurface layer of the specimen makes about 14.2 %.

2.2. Study of the modified surface layer of 30CrMnSiNi2 steel

Investigations of surface layer of 30CrMnSiNi2 steel specimens performed with the use of SEM have allowed to reveal nanosized particles in the modified layer (fig. 1,a). Average size of the particles made 80-10 nm. The particles are non-uniformly distributed over the specimen surface. The thickness of the modified surface layer was also estimated to show the depth of 2 μm (fig. 1,b). X-ray microanalysis has shown that iron in the layer has α-structure, while Zr-containing phases are as well revealed there.



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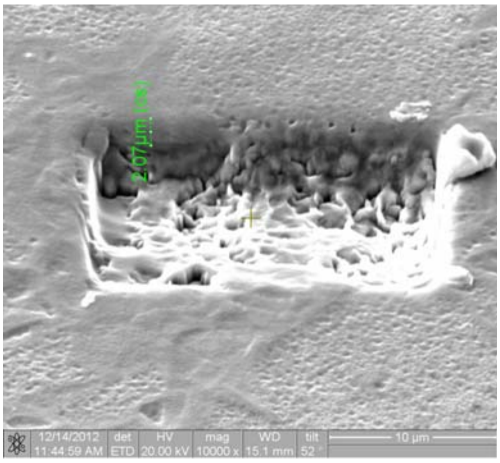


b)

Figure 1. SEM-micrographs of surface nanostructured specimen

2.3. Static tension tests

2.3.1. Mechanical properties

During tensile tests the loading diagram of 12Cr1MoV steel for dog-bone shape specimens was registered. 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 $\sigma_{0.2}$= 270 ± 25 MPa, ultimate strength –σ_u= 494 ± 36 MPa and elongation – ε = 20 ± 3 % which is close by values to the reference book data for this steel [4]. After the surface nanostructuring the value of ultimate strength is increased up to σ_u = 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.

2.3.2. Optical microscopy

Microscopic investigations of specimens surface near the area of the main crack at a certain distance from the fracture edge were performed (fig. 2). It is found that the specimen without surface treatment has the pronounced grain strain induced relief (fig. 2, a). At the same time undermore detailed observationit is seen that the surface of the nanostructured layer has small microcracks characterized by variation in their orientation in the modified layer (fig. 2, b). Whether, presence of the nanostructured layer on the surface of 12Cr1MoV steel hinders the formation of the grain relief that results in lower surface roughness of such 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.

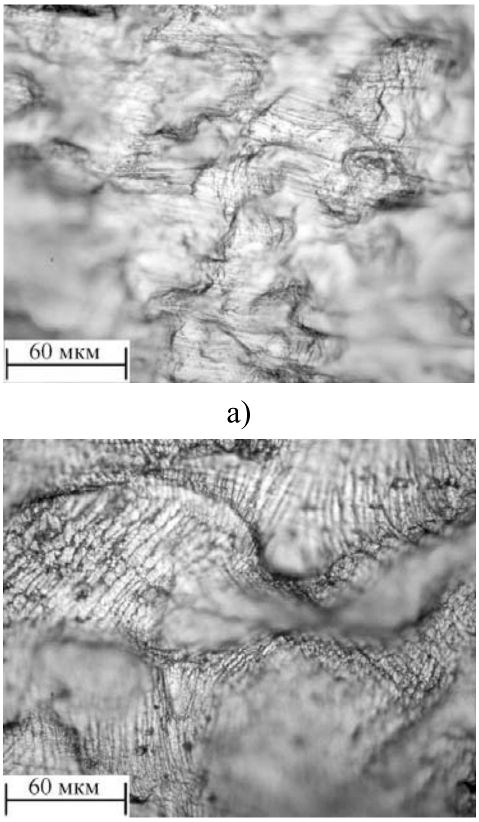


Figure 2. Surface images of failured specimens at static tensile tests: a) specimen without treatment; b) after nanostructuring surface; a), b) optical images, axis of loading is oriented horizontally

2.4. Cycling tension tests

2.4.1. Low-cycle fatigue (LCF) 12Cr1MoV

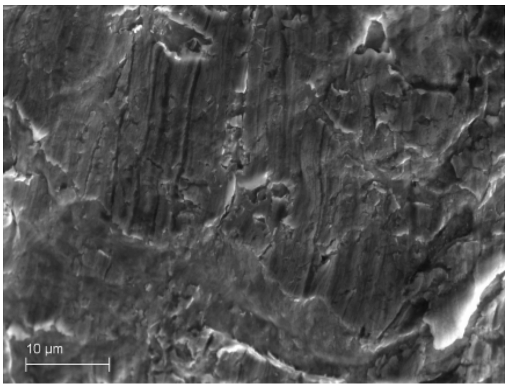
Results of cyclic tension tests have shown that under LCF the number of cycles prior to the fracture of the specimens with nanostructured surface layer is increased by 3 times. In doing so, the time before main crack initiation is increased approximately by3 times. The graphs of dependence of the main crack length versusthe number of cycles were plotted. It is clear that for the treated specimen later origination and more slow development of the main crack in the specimen is characteristic feature as compared with oneswithout 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.

2.4.2. High-cycle fatigue (HCF) 12Cr1MoV

The results of tensile tests under HCF 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 images 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 LCFat comparisonthe graphs of specimens without treatment and after the surface nanostructuring differences 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 made0.03 μm/cycle, while for one with the nanostructurized surface layer its rate is reduced by 2 times that can be estimated as 0.015 μm /cycle.

2.4.3. Fractography study of cyclically fractured specimens 12Cr1MoV

Micrographs of specimens fractured under cyclic tensile were obtained using scanning electron microscope at high magnification (fig. 3). Micrographs gained in fatigue crack growth region (fig. 3, b) also point to the brittle pattern of the nanostructured surface layer fracture being compared with untreated specimens (fig. 3, 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.



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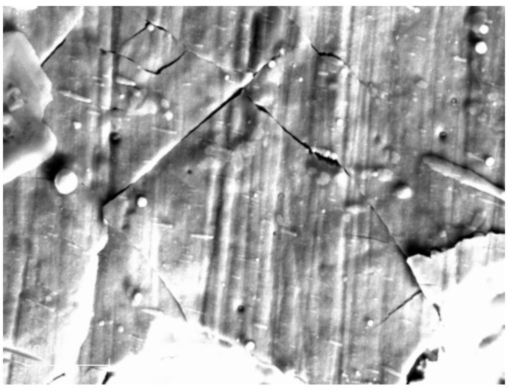


b)

Figure 3. 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

2.4.4. High-cycle fatigue (HCF) 30CrMnSiNi2

The tests on cyclic tension of 30CrMnSiNi2 steel specimens have shown that surface nanostructuring by Zr^+ ions gives rise to double-or-triple time increase of fatigue durability. In doing so nucleation of fatigue crack in specimens with the surface modification happens later, while rate of its growth is noticeably slower in contrast with non-treated specimens.

2.5. Test on cyclic alternating bending

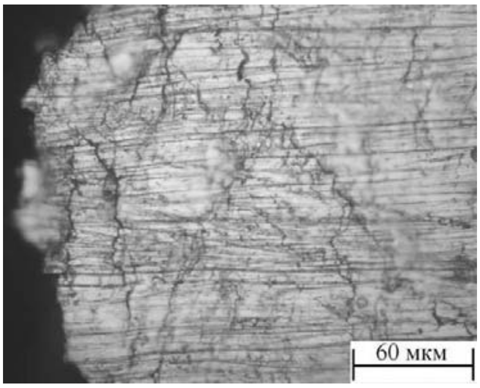
According to the testingdata the fatigue life-time of specimens under cyclic alternating bending is increased due to nanostructuring of a surface layer by~ 2 times. The series of optical images were used for plotting the graphs to characterize dependence the crack length versus the number of loading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

2.5.1. Optical observation of strain induced relief

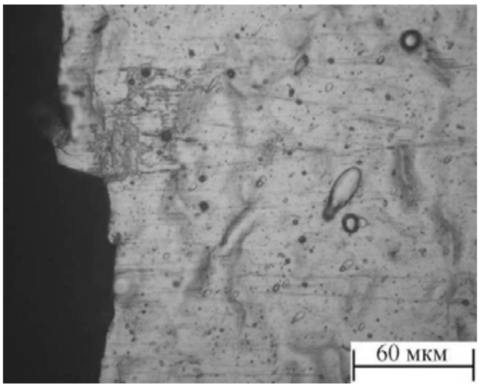
Images of surfaces of both type specimens after the different number of cycles before fracture to cause distinction in the formation of strain induced relief are given in fig. 4. One can see formation of manifested thin folds (fig. 4,a) located close to the fracture area (region of maximum curvature) 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. 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. 4, b).

The analysis 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 R_a 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 crackis 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. This may cause a reduction in growth rate atmain fatigue crack propagation.



a)



b)

Figure 4. Optical micrographs to illustrate strain induced reliefonthe surfaceofspecimens: a) without treatment; b) withnanostructured surface layer

3. Discussion of results

The first effect of the surface layer nanostructuring which is to be stressed is the noticeable increase of flowstress with a ratherlow decrease of ductility. Traditionally, ion beam treatment is accompanied by the formation of a layer with thickness up to 0.1 micron, whereas under the treatment used the thickness of modified layer is estimated as 2 μm (according to the data of optical profilometry of the cracked layer) up to 10 μm (according to data of the scanning electron microscopy at observation of the fracturesurface after fatigue fracture).In doing so, presence of multiple cracksin the modified layerin the vicinity of neck exerts no significant impact on the reduction of plasticity due to the small thickness of the modified layer,as well as realization of multiple cracking. As a result, none of the cracks becomes the main oneand plastic deformation of the composition is determined mostlyby the development of plastic flow in the ductile material of the substrate.

The following key result is associated with notable changein the character of the localized plastic deformation development in the subsurface.It is shown that the layer to have the 1.5 times increased hardness and thickness of several microns can significantly change the pattern of the deformation relief formed. At low strain of the substrate (for example, at static tension out the neck area) deformation relief on the surface almost is not formed while in the region of intensive development of plastic deformation the size of strain induced relief elements is noticeably bigger than that in specimens without the treatment. This result is consistent with data on the cyclic bending tests where during the process of cyclic loading the modified layer behaves much like a thin polymeric film placed on the water surface which is manifested both in dumping the deformation development and remarkable smoothing reliefto form at the specimen's surface. The hindering formation of strain induced relief under tests by another scheme (cyclic tension) when the surface relief to a large extent determines the origin of the main crack brings to increasing by more than twice the time period prior to origin of fatigue crack in the case of the ion beam modification.

Of particular interest in the light of the results obtained is observed difference in the character of the fatigue fractureunder cyclic tension and bending. It should be reminded that in the first case the crack originwas fixed at cyclic deformation not less than 0.75 N_F (the total number of loading cycles). At the same time, under alternating bending tests the main crack is already initiated at cyclic load of 0.15-0.2 N_F which in absolute terms corresponds to the same number of bending cycles of the non-treated specimens and ones with modified surfacelayer. Thus, for the case of cyclic bending the main crack in the specimen without the treatment originated very early but the rate of its growth was rather low in contrast with one under cyclic tension tests. This result, in principle, can be quite simply interpreted, because under the cyclic bending a surface layer is subjected to the periodic effects of maximum tensile and compressive stresses. Therefore, presence of the nanostructured layer on the surface hinders noticeable delay the origination (but not propagation!) of the crack.

However, its subsequent growth is realized significantly slower that was interpreted a result of fewer microcracks arising along the grain boundaryin the specimen without treatment, while the nanostructured layer hinders the formation of microcracks and, consequently, the rate of the crack growth. Another factor that may have a significant impact on the character and the rate of the origination and propagation of the crack is its depth. Despite the fact that in the work direct measurements of the depth of a forming fatigue crack was not carried out but judging by the size of its opening the depth of a crack in a specimen of the initial state should be noticeably higher, which in it turn should bring to its more rapid growth.

In contrast, under cyclic tension test a crack is originated sufficiently later that could be associated with roughly equal distribution of stresses through the cross section. As a result, it is the deformation relief of surface that can be a key factor in terms of origin of the main crack. A visible hindering in its formation has led not only to a multiple increasing of fatigue life-time of nanostructured specimens but also significantly slower rate of the main crack propagation.

It is proved by the notable difference in the rate of its propagation which under LCF (when influence of processes of localized plastic deformation is dominant) amounted up to 3.5 times (nanostructured layer hinders the development of plastic deformation), while under HCF when crack growth is governed by dominant mechanisms of the brittle fracture, the difference in the rate of the crack growth made two times.

In conclusion, one should point the role of multiple cracking processes, especially under conditions of high ductility of the substrate. In our view, the greater contribution to improving fatigue durability is associated with suppression of plastic deformation processes by nanostructured layer of higher strength causing substantial delay of the moment of the fatigue crack initiation. Noticeably smaller contribution to increasing the fatigue life-time is associated with the processes of multiple cracking. At the same time, usingmaterials of higher strength in contrast with substrate, as well as greater thickness of a coating will result in its cracking with their fast coalescence into the main one resulting in a substantial reduction of fatigue durability of "coating-substrate" compositions.The combination oflow thickness of themodified surface layer and the ratio of the elastic characteristics compared with more ductile substrate material with high plastic deformations (for instance, in the area of a neck, or plastic tension under cyclic bending) has led to a formation of the system of multiple crackswhose orientation is not always in line with the normal to the direction of the external load.This is a fact that allows us to discuss the thesis of multiple cracking as one of the ways to improve the fatigue durability of "coating-substrate" compositions.

4. Conclusion

A study of structure and deformation behavior under static and cyclic tension and alternating bending the specimens of heat resistant 12Cr1MoVsteel subjected to surface layer nanostructuring by zirconium ion beam was performed. It is shown that ultra-dispersed particles of zirconates with the size about 100-150 nm were formed as a result of such treatment in a subsurface layer with a thickness up to several microns. Increase of strength properties by 15 % and decrease elongation before fractureby ~ 19 % is fixed due to thistreatmentat the static tensile.

It is found that the formation of the nanostructured surface layer increases the fatigue durability by 2-3 times at cyclic tension tests. The authors associate this with the delay of time the main crack initiationdue tohindering in deformation relief formation which play the key role in propagation of a crack.

At cyclic alternating bending the main crack origination in specimens of both types happens at almost equal numbers of cycles that is associated with the periodic occurrence in the surface layer of tensile and compressive stresses. However, at a subsequent stage the growth rate of the main crack is differing approximatelyin 2 times because of the nanostructured surface layer hinders microcracks origin on the grain boundaries of the substrate. This result is consistent with data of distinction comparison for both fatigue crack growth rate at the cyclic tensile and bending. For the second case, the growth rate is several times less.

The influence of multiple cracking on improving fatigue durability of specimens under study is discussed. It is shown that in the area of the main crack (predominantly under cyclic tensile) the finite length microcracks whose orientation does not always coincide with the direction of normal to the axis of the applied load are formed. This fact may be the manifestation of the effect of channeling plastic shears in the substrate material.

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