

Influence of Zr⁺ Ion Beam Irradiation on Structure and Fatigue Durability of 12Cr1MoV Steel

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Abstract

Cyclic tension and bending tests of heat-resistant 12Cr1MoV steel specimens in as received state as well as after Zr⁺ ion beam irradiation were carried out. The differences in patterns of strain induced relief formation, as well cracking of thin modified surface layer were observed and analyzed by means of optical microscopy and interference profilometry. Changes to take place in subsurface layer of 150 μm thick were characterized by means of microindentation, metallography and scanning electron microscopy. It is shown that mechanical properties of irradiated specimens are mostly influenced due to thermal treatment to take place during irradiation rather than formation of a 2 μm thin surface layer doped with Zr. The differences in deformation behavior are interpreted in terms of physical mesomechanics concepts.

Keywords: Irradiation; metallography

Introduction

Heat resistant 12Cr1MoV steel is used for manufacturing of parts of energetic equipment to operate at high temperatures: 570°C to 585°C. One of the main reasons of failure of structure elements / machine parts made of such kind of ferrite-perlite steel is fatigue fracture due to the cyclic thermo-mechanical effect [1]. Ion implantation for a long time was employed for surface layer modification to form new structural-phase states there [2]. A new technique of surface irradiation by a metal ion beam was developed in ISPMS RAS to provide deeper structure modification in contrast with traditional ion implantation. However, the benefit from deeper penetration of Zr ions is accompanied by extra heating of surface layer and formation of new phases of iron with zirconium there.

The aim 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.

Experimental

Specimens of ductile heat resistant 12Cr1MoV steel that can preserve the constant initial structure at heating up to the temperature of 570°C - 585°C were used in the study. The choice of material for research partly was caused by the fact that the steel does not experience structural changes at the temperature which are developed during the ion beam irradiation.

The specimens were mechanically polished and divided into 2 groups: (a) in the as-received state and (b) ones subjected to zirconium ion beam treatment (irradiated ones). The treatment was performed with the help of high current vacuum arc source of metal ions UVN-0.2 "Quant". The specimens were processed by a zirconium beam with energy of -900 V and current density of 0.1 mA/cm² in a chamber under the vacuum level lower than 7×10⁻³ Pa. The time of the treatment made 19 min. In order to reduce the thermal influence of the ion beam a rotating stage was designed and manufactured. In doing so, heating mostly occurs when ion beam interacted directly with specimen surface. Local strain was estimated by Digital Image Correlation technique (DIC) through calculating the normalized value of shear strain intensity (principle strain) [3].

Experimental Results

The structure of 12Cr1MoV steel in the as-received state is represented by large ferrite grains >> 1 μm with inclusions of cementite (Fe₃C), whose average size makes 120 nm. The structure of surface layer after the Zr⁺ ion beam modification is represented by phases of FeZr₃, FeZr₂, as well as ferrite grains. The average size of newly formed phases in the surface layer makes 100-150 nm.

Micro-hardness was measured by indentation into flat surface. Additionally the lateral face of the treated specimens was examined to characterize the changes to occur through the specimen cross-section (Figure 1a). It is seen that specimens in the as-received state possess the micro-hardness of 1,67 ± 0,07 GPa (curve 1) while after Zr⁺ ion beam irradiation the micro-hardness has changed substantially even in the specimen core (Figures 1a and curve 2). It is seen that in the surface layer of the irradiated specimens the H_u was reduced from 1.67 down to 1,5 ± 0,06 GPa. The depth of the softened subsurface layer can be estimated as equal to 70-100 μm. At the same time quite unexpected result was revealed in the bulk of the irradiated specimens. Regardless of the heat resistant status of the steel under study at the depth more than 100-150 μm micro-hardness increased up to 2,16 ± 0,05 GPa that is higher than one in specimens in the as-received state (Figure 1a).

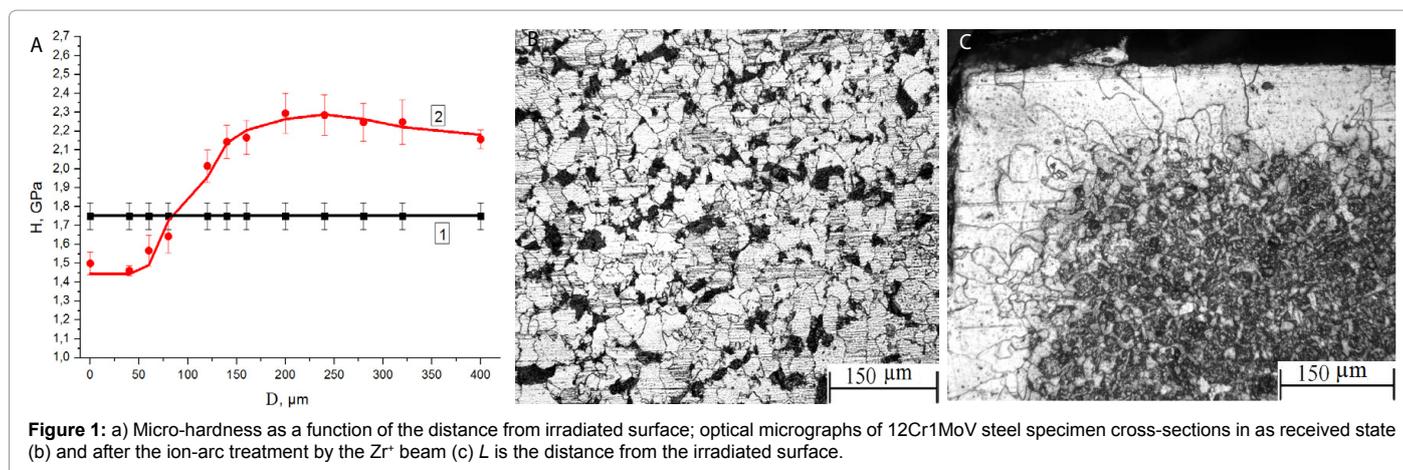
Based on obtained results one can estimate the thickness of the subsurface layer softened during Zr⁺ ion beam irradiation to be equal to 100-150 μm. At the same time, at a depth exceeding 100 μm the micro-hardness is increased by 22% in contrast with specimen in the as-received state. The brief analysis of the obtained results allows us

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to conclude that thermal impact to take place under the irradiation is responsible for three major phenomena. Firstly, deeper penetration depth of Zr⁺ ions into the steel substrate that is nearly one order higher in comparison with conventional technique of ion implantation. Secondly, annealing in the surface layer of several dozen μm thickness (this become clear from microstructure analysis - Figure 1c). Thirdly, certain enhancement of the micro-hardness in the specimen core takes place that will be discussed below.

X-ray diffraction was applied to characterize the chemical composition in the modified surface layer. For specimens in the as-received state structure are primarily represented by the α-iron, whereas after the irradiation in the subsurface layer one can reveal formation of intermetallic phases of the system Fe-Zr:FeZr₂ and FeZr₃, as well as ZrC carbides. Structural-phase micro-analysis of the Zr-doped surface layer (whose thickness does not exceed 1-2 μm) was carried out. The data indicate that the overall content of zirconium in the subsurface layer of the specimen does not exceed 14.2%.

Optical microscopy was employed to observe the structure to form in the surface layer during the irradiation. It is seen from (Figure 1b) that non-treated specimen consists of ferrite-perlite grains. In order to estimate microstructure changes through the thickness the lateral faces of the specimens after treatment were polished (Figure. 1c). One can distinguish the difference in structure within subsurface layer and bulk material (the core). The thickness of the modified layer can be estimated as equal to 90-130 μm while it does not have pronounced boundary with adjacent bulk layers. As far as we can judge from pyrometer data this layer experiences short term heating up to the temperature of 850°C that can give rise to grain growth. At the same time the core of the specimen was subjected to such heating to a lesser degree. The rotation of the specimens in the vacuum chamber during the irradiation (cyclic heating with further cooling when specimen does not contact with the ion beam) combined with possible carbon diffusion from the subsurface layer might give rise to certain decrease of grain size from 27 μm down to 20 μm in the core that becomes visible from (Figure 1c). This might be a possible explanation of microhardness increase in contrast with the non-treated specimens.

During the tensile tests the loading diagram of 12Cr1MoV steel for dog-bone shape specimens was recorded. It was found that for the nontreated specimens the presence of sharp yield point (yield tooth) is evident much like in low carbon steels. Basic mechanical characteristics of these specimens are: $\sigma_{0.2} = 270 \pm 25$ MPa, ultimate strength - $\sigma_u = 494 \pm 36$ MPa and value of elongation at failure - $\epsilon = 20 \% \pm 3 \%$ which are close to reference values. After the irradiation the value of

Samples	Number of cycles before the crack nucleation, × 10	Number of cycles prior to failure, × 10
Low-cyclic fatigue		
As-received state	38 ± 0.5	45 ± 0.45
After the treatment	115 ± 0.8	152 ± 0.7
High-cyclic fatigue		
As-received state	110 ± 0.65	127 ± 0.6
After the treatment	190 ± 72	232 ± 33
Alternating cyclic bending		
As-received state	18 ± 0.1	89 ± 0.2
After the treatment	22 ± 0.2	176 ± 0.4

Table 1: Results of cycling tension tests.

ultimate strength is increased up to $\sigma_u = 570 \pm 17$ MPa while the value of elongation at failure becomes lower $\epsilon = 16 \pm 0.7\%$. In doing so, no yield plateau is seen at the diagram of the treated specimens.

Results of cyclic tension tests have shown that under low-cyclic fatigue (LCF) the number of cycles prior to failure of the non-treated specimens is increased by 3 times (Table 1). In doing so, the time before main crack initiation is increased approximately by 3 times as well. The graphs of dependence of the main crack length versus the number of cycles were plotted. It becomes clear from the plot that in the treated specimen delayed formation and slower growth rate of the main crack is characteristic feature as compared with ones in as-received state. For the latter the rate of crack growth makes 0.085 μm/cycle while for samples with the modified surface layer it makes 0.023 μm/cycle which is lower by more than 3.5 times. Thus, the modified surface layer effectively hinders the fatigue crack origination.

The results of cyclic tension tests under high-cyclic fatigue (HCF) showed that the number of cycles prior to failure of specimens with the modified surface layer is increased by 2 times (Table 1). The number of cycles before the crack nucleation is increased approximately by 2 times as well. Based on the analysis of optical images the dependence of the crack length versus normalized and absolute number of tension cycles was calculated and plotted. As was seen earlier in the case of the LCF tests, crack growth rate in the non-treated specimens was 0.03 μm/cycle, while for irradiated ones it is reduced by 2 times, i.e., 0.015 μm/cycle.

It is shown that fatigue life-time of the treated specimens under cyclic alternating bending is also increased by ~ 2 times (Table 1). A series of optical micrographs were used for plotting the graphs to characterize dependence of crack length versus the number of loading cycles. At cyclic bending, the main crack originates at the nearly same number of

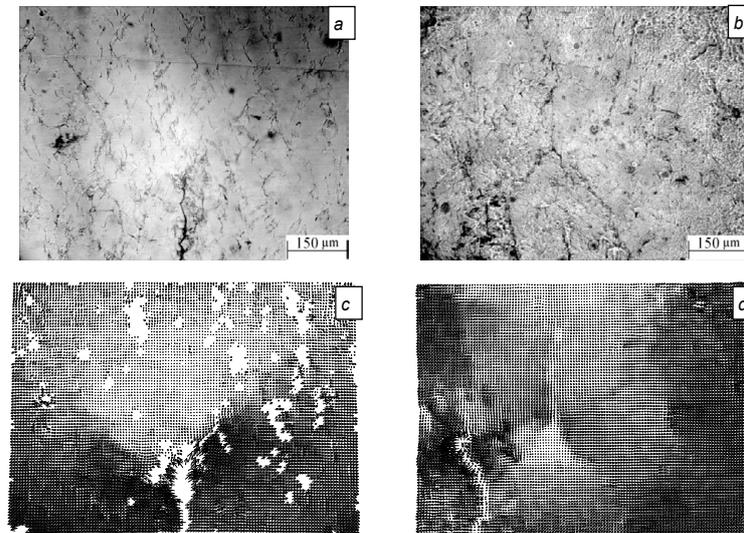


Figure 2: Optical micrographs (a, b) and corresponding displacement vector fields (c, d) in specimens under cyclic bending; a), c) specimens without the treatment; b), d) after the ion beam irradiation; a), c) 3000–4000 cycles; b), d) 3000–4000 cycles.

cycles for both types of specimens but has substantial differences in the propagation rate. The latter for the untreated specimen was $0,05 \mu\text{m}/\text{cycle}$ while for the irradiated specimen it made $0,024 \mu\text{m}/\text{cycle}$. Thus, it is shown that Zr⁺ ion beam irradiation can slow the rate of fatigue crack growth by about 2 times.

The analysis and comparison of optical micrographs and displacement vector fields under cyclic bending tests revealed differences in patterns of strain distribution at fatigue crack opening (Figure 2). During the testing of the untreated specimens, regardless crack branching, it develops with faces opening by normal mode that is accompanied by formation of a couple of mesoscale shear bands to originate at the crack tip (Figures 2a and 2c). Under cyclic bending of the treated specimen it is difficult to reveal exact crack paths (Figure 2b) since presence of the modified (softened) layer disperses stress concentrator in the crack tip and deformation in its vicinity is redistributed over larger area [4]. This becomes evident at observation of the displacement vector field (Figure 2d) some regions with self-consistent displacements are formed that tend to accommodate rotational deformation modes to occur at crack propagation. This might be resulted from difference in strength of modified surface layer and bulk material (the core) since they possess different ductility as well. The similar effect was observed in shot-peened specimens under fatigue tests however modified layer in the latter case experienced compressive stresses [5]. Being based on data of micro-hardness measurement one can postulate that softening of the subsurface layer can play a positive role in increasing the fatigue life time since crack arrest effect can occur in more ductile modified subsurface layer of several tens μm thickness. The idea of stress redistribution under cyclic loading that might explain results obtained in the paper are put forward and studied in [6–8].

Discussion of Results

The obtained results can be interpreted from several points of view. Firstly, an advanced surface modification technique is offered, which gives rise to changing structure and properties of the heat-resistant ductile steel in a very unusual way: i) thickness of the modified subsurface layer to contain zirconium-based phases as well as intermetallic ones (with couple of μm thickness) is much higher as

compared with conventional ion implantation; ii) the softening of the surface layer of $100 \mu\text{m}$ thickness takes place, possibly due to cyclic thermal treatment under the Zr⁺ ion beam irradiation; iii) the micro-hardness in the core of the irradiated specimen of the heat resistant steel with total thickness of 1 mm is increased. Secondly, presence of the softened subsurface layer and hardened core of the substrate gives rise to increasing strength by 15% with decrease in elongation. Thirdly, layered pattern of macrostructure changing ensures the increased durability both at low and high cyclic fatigue. Fourthly, presence of the softened modified subsurface layer along with newly formed phases hinders formation of strain induced relief and by doing so, delays nucleation and propagation of fatigue crack both under cyclic tension and alternating bending. A similar effect was previously observed in medium carbon steel under torsional fatigue [9].

In summary, it is not possible to establish mechanisms leading to increased fatigue life time based only on formation of the modified surface layer or the softening subsurface layer. This requires additional studies and comparison with literary data [10]. In any case, the treatment used looks very promising both for practical applications and study of deformation and fatigue fracture to occur in tests of steels with the modified surface layers. With certain modifications, the offered technique may be extended for structure modification and increasing fatigue life of high strength steels [11].

Since the effect observed to a large extent is attained due to thermal effect on the specimen it is of interest to search for less expensive techniques to achieve the similar effects. This is related to study of effects and acquainting understanding what exactly has changed in the structure that exerts the governing influence on the properties. Since the state of surface layer is one of the most important facts to determine fatigue life time it is possible that delay in deformation development due to formation of modified surface layer (up to $2 \mu\text{m}$ thick) might be of great influence for resistance to cyclic loading. In this regard the technique used in this study might be of interest for industrial applications.

Finally, with regard to structural changes in the core of the irradiated specimens, regardless of the heat-resistant status of the steel,

the annealing took place in the surface layer of 100 μm thickness. This is obviously related to the high temperatures that develop there. However, the refinement of the grain size of the core material may be attributed to thermal cycling. In doing so, some diffusion of carbon towards deeper layers can take place. In this paper this plays a positive role. However, influence of surface hardened (carburized) layers is not so unique [12]. In the latter paper, residual austenite was identified as the factor affecting the structure and mechanical behavior under cyclic loading.

Conclusion

Structure and deformation behavior under static and cyclic tension as well as alternating bending of the heat resistant 12Cr1MoV steel subjected to the zirconium ion beam irradiation was studied. It is shown that as a result of such treatment ultra-fine particles of zirconates with the characteristic size of about $\sim 100 \mu\text{m}$ were formed in the subsurface layer with the thickness of up to several microns. Increasing of ultimate strength by 15% as well as decrease of value of the elongation at failure down to 19% take place that is related to bulk substrate structure modification to occur during the ion beam irradiation.

It is found that formation of the modified surface layer tends to increase the fatigue durability by 2-3 times at cyclic tension tests. This may be partly related to the softening of the subsurface layer of 100-150 μm thickness that ensures decreasing power of stress concentrators to occur under fatigue tests. The certain hardening of bulk heat-resistant steel can be also responsible for increasing mechanical properties.

It is shown that for the case of LCF due to localized plastic flow the difference in growth rate of the main crack in specimens of both types is differed by a factor of 3. For the case of HCF the intensity of plastic deformation processes is less but the difference of the rate of the main crack propagation for specimens in the as-received state and irradiated ones is varied by a factor of two.

At cyclic alternating bending the main crack nucleation in specimens of both types occurs at nearly equal numbers of cycles that is associated with the periodic occurrence of tensile and compressive stresses in the surface layer. However, at the subsequent stage of steady fatigue crack growth the propagation rate is about 2 times lower since the softened modified surface layer ensures effective redistribution of stresses, as well as dispersing stress concentration at the grain boundaries. This lowers stress intensity in the vicinity of main fatigue crack tip as well.

The offered surface treatment technique cannot be characterized

as exactly surface modification since high local temperatures during irradiation lead to structure modification at depths that substantially exceed ones where Zr ions can penetrate. The observed effects of changing mechanical properties under static and cyclic loading might be interpreted from a multi-scale viewpoint: Zr-doped surface layer (units of microns) - thermally softened (annealed) layer (up to 100-120 μm) - hardened due to cyclic short time influence core layer. The most effective behavior of such multilayered systems takes place under cyclic loading.

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