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FATIGUE OF AUSTENITIC STEEL MODIFIED BY OXIDES OF ZIRCONIUM AND YTTRIUM

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The results of fatigue tests on a cantilever bending steel 08Cr18Ni10Ti in the initial and in the modified state (additions of nanosized oxides of Zr and Y) are presented. Significant increase in fatigue strength in the area of low-cycle fatigue and a slight change of the characteristic when a large number of load cycles for the modified steel were shown. The observed changes in fatigue properties are related with the difference in the character of origin and development fatigue damages.

INTRODUCTION

Oxide Dispersion Strengthened (ODS) steels are promising materials for use as critical components of nuclear and nuclear fusion power plants [1]. As known, the high thermal and radiation stability under high irradiation of this type of materials can be achieved by forming system of clusters from stable oxides of the transition metals (Ti, Zr, Y, etc.) enriched with Cr, Mn, Si. Distances between the clusters are commensurate with the length of the free path of radiation point defects (~10-20 nm). Interfaces of such inclusions with the matrix are barriers for dislocations and traps for interstitial atoms and vacancies contributing to their recombination.

Currently, powder technology for producing ODS steels is the most studied (see, e.g., [2]). However, this technology restricts amount of material, is laborious and costly. In this connection study of a possibility for producing such steels by melting is undertaken [3, 4].

The total duration of transitive modes and cyclic loads on the equipment will be enhanced when increasing NPP service life. This would commonly cause to metal fatigue and can induce cracks in the elements of equipment and reactor internals and fuel elements depressurization will be accelerated. Development of new modifications NPP equipment, internals, fuel rods and fuel assemblies requires addressing these problems [5]. Given this, is expedient study of fatigue for prospective structural materials, including ODS steels. In the framework of this work we have studied fatigue of austenitic steel modified with oxides of zirconium and yttrium by the method of vacuum melting.

MATERIALS AND METHODS OF RESEARCH

Modified steel 08Cr18Ni10Ti was obtained by vacuum-arc melting with the addition of nanopowders of ZrO_2-Y_2O_3 (ZrO_2, stabilized with Y_2O_3) and metallic Y [4]. Electrode melting was an electrode from steel 08Cr18Ni10Ti.

Billet in size 93×55.1×9.4 mm (thickness l = 9.4 mm) was cut from the melted ingot. The billet was subjected to thermo-mechanical processing (TMP) with several stages using vacuum rolling plant DUO-170 equipped with furnace:

1 – heating of 1 h at T = 1100 °C, rolling until l = 5 mm, displacement in the furnac

2 – eating of 20 min at T = 1100 °C, rolling until h=3 mm, displacement in the furnace;

3 – cold rolling in several passes until h = 0.5 mm.

Plane-parallel samples in size 0.5 × 3.5 × 40 mm for fatigue tests and samples in the form of paddle with a size of working part 0.5 ×1.5 × 15 mm for uniaxial tensile tests were cut from the obtained sheet. Mass content of impurities in the samples was: Zr ~ (0,08…0,09)% and Y ~ 0,8%

Making steel samples from billet without modifying additives included the same TMP.

We have marked as "modified", the state of steel containing additives of zirconium oxides and yttrium oxides, and without the additives – as "initial".

Tests on alternating cantilever bending with load frequency f = 50 Hz and on uniaxial tensile with the rate 10^{-4} s^{-1} at room temperature were carried out. For microhardness measurements we have used PMT-3. The structure of the samples was studied using optical, transmission (JEM-100SX) and raster (JEOL-840-JM) microscopy.

Discovery of the grain structure of the initial and modified steels was carried out by a combination of electropolishing in a solution of $H_3PO_4 + H_2SO_4 + CrO_3 + H_2O$ (1:1:1:1) and subsequent electrochemical etching in a solution of $HNO_3 + HF + H_2SO_4 + H_2O$ (1:1:1:1) at the current density $j \approx 22$ mA/cm^2 for t= 3…5 s.

RESULTS AND DISCUSSION

Fig. 1 shows the microstructure of the ingot after melting. The open circles are marked oxide nanoparticles. The average distance between them is about 65 nm. Estimates have shown that the density of nanoparticles was 3.6×10^{15} cm^{-3}.

Fig. 2 illustrates grain structure of the initial steel and the modified steel in the annealed state after TMP. We have built distribution histogram of grains (Fig. 3) according to the metallographic data. For the modified steel is characterized more uniform distribution of grain by size, a substantial reduction of the grain size having $d \geq 80$ μm, and displacement of a maximum of histogram from 30 to 20 μm.

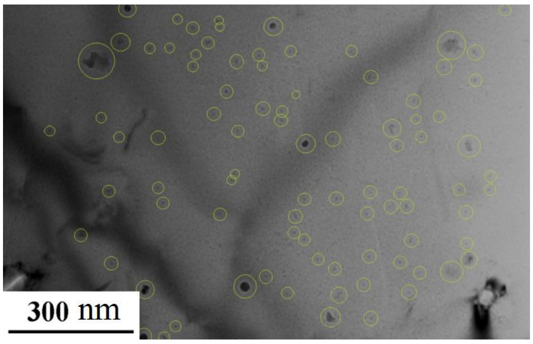


Fig. 1. Microstructure of the modified steel in the cast state

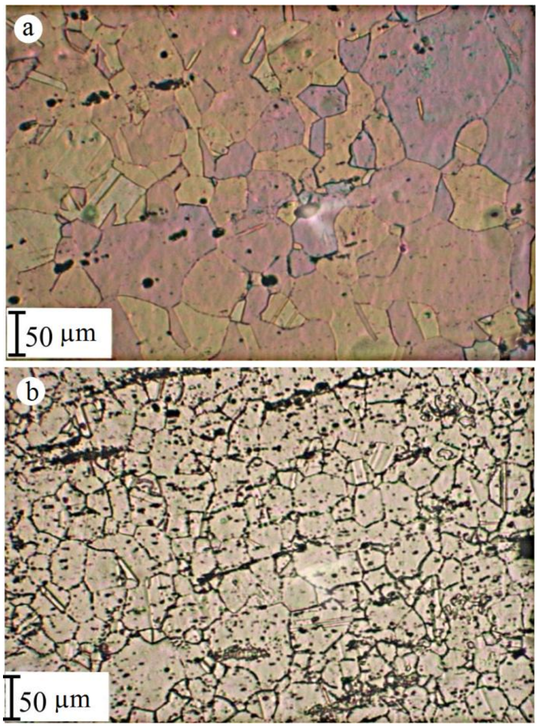


Fig. 2. Grain structure of the initial steel (a) and the modified steel (b)

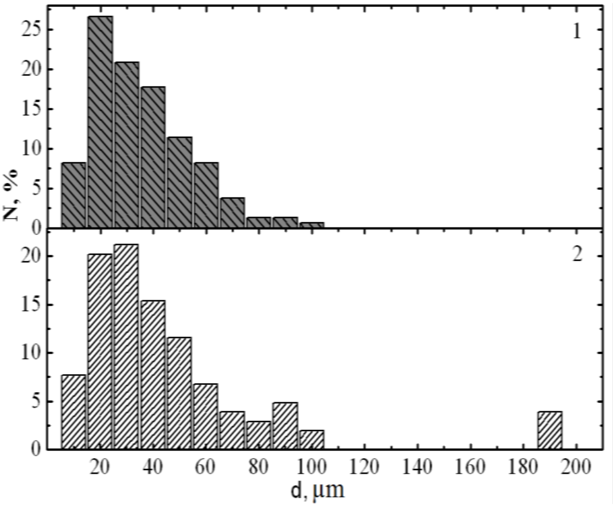


Fig. 3. Distribution of grain size:
1 – modified steel; 2 – initial steel

Table shows the average grain size and mechanical characteristics of the samples. It follows from Table and Fig. 2 that the decrease in average grain size by about 25% in modified steel is accompanied by an increase in the microhardness (3%), yield strength (8%) and a decrease of plasticity (8%).

It follows from tests on alternating cantilever bending that the modified steel possesses higher fatigue strength relative to the initial, especially in low-cycle fatigue (Fig. 4). Thus, fracture stress is increased by ~ 30% for $N = 10^4$ cycles. At the same time growth of the fatigue strength decreases markedly in region of low amplitudes. We can evaluate on the basis of $N = 10^6$ cycles endurance limit value $\sigma_{RM} \sim 420$ MPa and $\sigma_{RI} \sim 375$ MPa in the modified and in the initial steel, respectively.

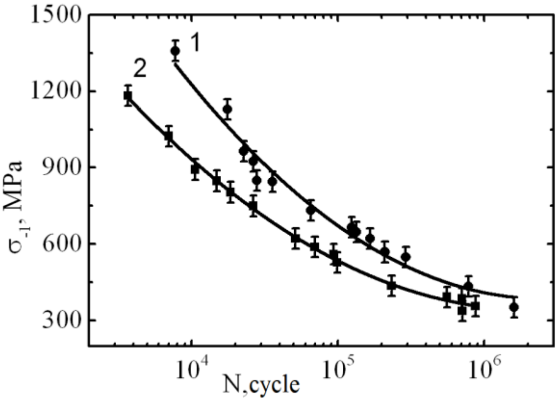


Fig. 4. Wohler curves: 1 – modified steel; 2 – initial steel

Fig. 5 illustrates the change of steel durability due to modification. It is seen in modified steel the increase in durability up to 10^6 cycles near endurance limit and monotonic reducing by two orders of magnitude for stress ~ 800 MPa.

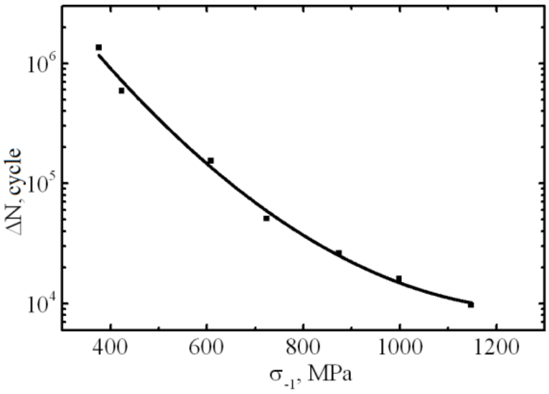


Fig. 5. Change of durability VS fracture stress

In Fig. 6 are presented the photomicrographs of typical sample surface areas near the fracture site depending on load level. Macrocracks total length of the modified and the initial steel is about the same at low loading amplitudes (see Fig. 6,a,b). The length of micro-cracks becomes significantly less in the modified steel relative to the initial steel at large amplitudes (see Fig. 6,d).

We have carried out a fractographic study to identify characteristics of fatigue fractures. Samples of the initial steel and the modified steel at multicyclic fatigue ($N \sim 10^6$, σ_{-1}=338 MPa) and low-cycle fatigue ($N \sim 10^4$, σ_{-1}=1105 MPa) were tested. Fractures were placed in a plane perpendicular to the axis of the specimen at the region of clamp indicating on destruction from maximum of normal stress.

The average grain size (d), Vickers microhardness (H_V), yield strength ($\sigma_{0.2}$), tensile strength (σ_B), uniform (δ_p) and total elongation (δ_o), as well as the relative changes (Δ) of these characteristics for the initial and modified steels

State of steel	d, μm	Δd, %	H_V, MPa	ΔH_V, %	$\sigma_{0.2}$, MPa	$\Delta\sigma_{0.2}$, %	σ_B, MPa	$\Delta\sigma_B$, %	δ_p, %	$\Delta\delta_p$, %	δ_o, %	$\Delta\delta_o$, %
Initial	46	–	1583	–	236	–	645	–	55	Ω	66	–
Modified	35	-24	1628	3	255	8	659	2.3	54	-2	61	-8

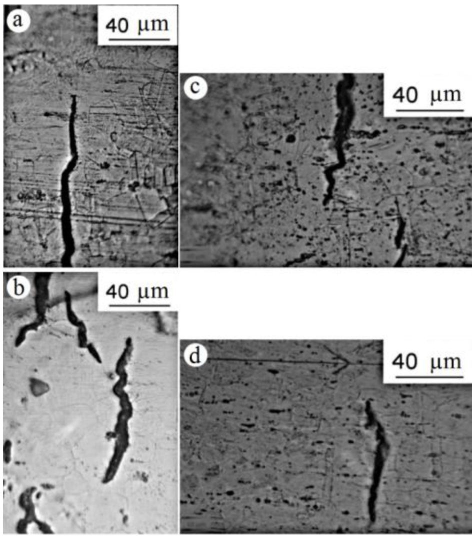


Fig. 6. Type of cracks after fatigue depending on the fracture load in initial steel (a, b) and in modified steel (c, d): a – $N \approx 10^6$ cycle, σ_{-1}=448 MPa; b – $N \approx 10^4$ cycles, σ_{-1}=842 MPa; c – $N \approx 10^6$ cycles, σ_{-1}=402 MPa; d – $N \approx 10^4$ cycles, σ_{-1}=910 MPa

The macrostructure of fracture after multicyclic testing in the initial steel and the modified steel is similar. Cracks in both cases have developed from a few ill-defined seats on one of surfaces of the sample. Almost entire surface of the fracture zones is occupied the areas of fatigue crack growth with underdeveloped structure. Areas of slow and rapid development of cracks are not identified, rupture area is not revealed.

The "blockiness" in the macrostructure of multicyclic fracture as facets of selective shine was revealed. In addition, we have marked substantially lesser size and amount of the facets in the fractures of the modified steel. One can assume that this difference in the macrostructure of fractures relates to the difference in the steels structure.

Low-cycle fractures as the initial and the modified steel, unlike multicyclic fractures, have multiple clearly defined foci of fracture in the form of scars on both surfaces of the samples (Fig. 7,a,b). Cracks have developed from these centers towards each other. The areas of slow and speed-up propagation of the cracks here are not divided, however there is some more developed macrorelief in the fractures of the modified steel. Rupture area at these fractures is located in the central part of the sample cross section due to cracks developing from opposite sides of the sample.

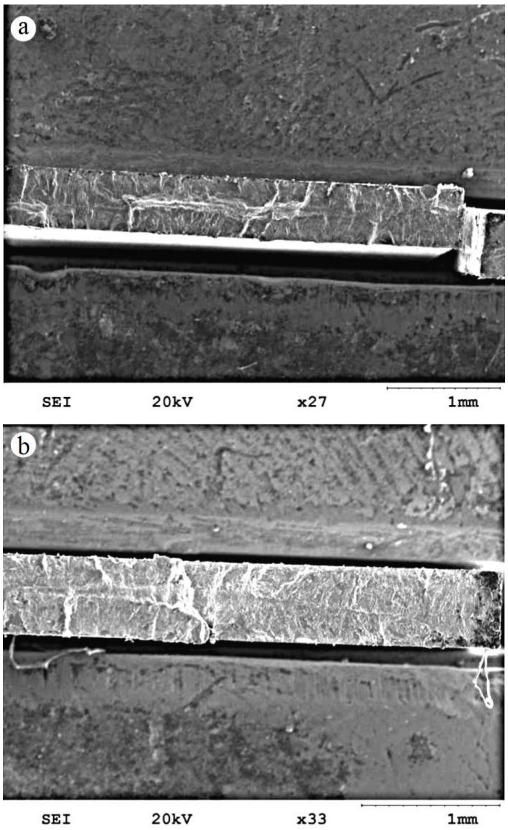


Fig. 7. Macrostructure of low-cycle fatigue fracture: a – initial steel; b – modified steel.

Rupture area at low-cycle fracture in the initial steel takes less than 0.1 mm in width, and has kind of step formed by shear stresses. It looks almost like a meet line fracture of the modified steel (see Fig. 7,b).

Research of micro-relief on the fractures gives possible to establish the following. There is preferably grooved structure of micro-relief for multicyclic fractures as in the initial steel (Fig. 8) and in the modified steel (Fig. 9), but the nature of the grooves is slightly different. In the initial steel there are continuous grooves of scalloped structure that are typical fatigue fracture for the ductile steels of low strength [6].

In the modified steel we observed arranged on the background of less-smoothed quasi-chipped facets indicating somewhat higher fracture energy intensity. However, the rate of increase in the energy intensity of the fracture for multicyclic fatigue of the modified steel is low in comparison with the initial steel, which correspond fairly close values of fatigue strength.

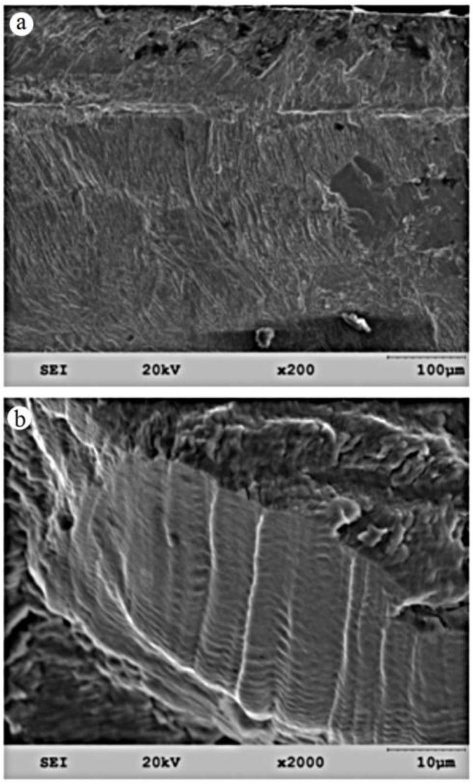
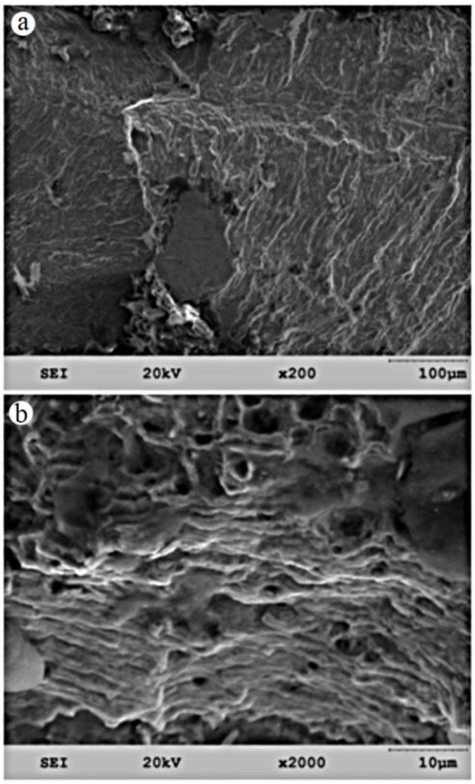


Fig. 8. Microstructure of multicyclic fracture of the initial steel



Puc. 9. Microstructure of multicyclic fracture of the modified steel

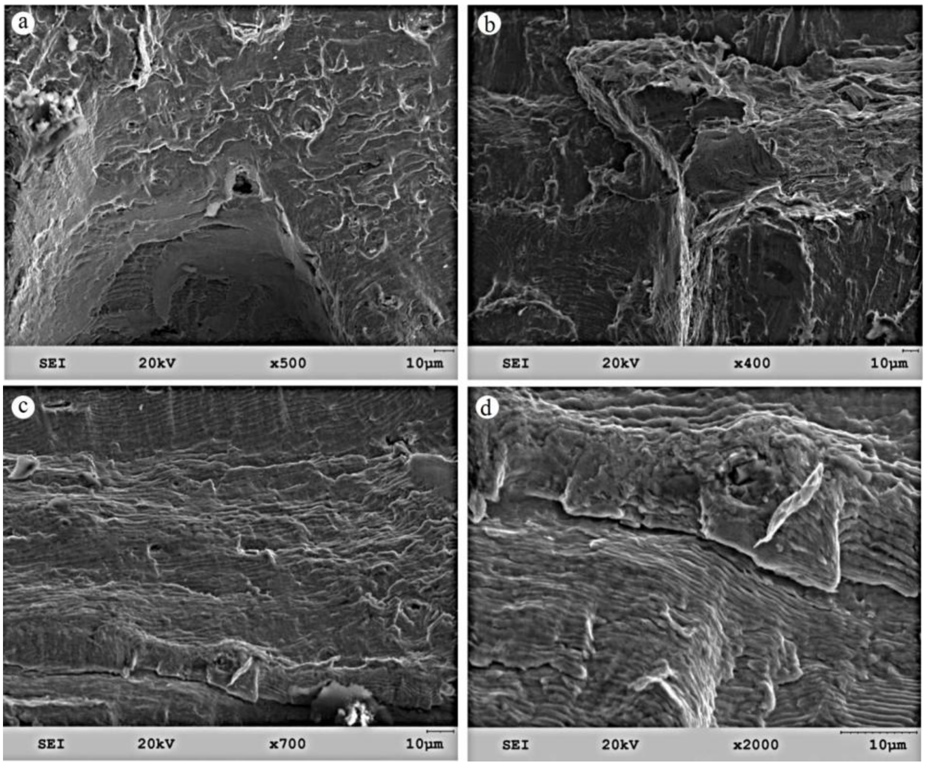


Fig. 10. Microstructure of low-cycle fatigue fracture of the initial steel

Microstructure of low-cycle fatigue fracture as the initial and modified steel is mixed. Along with the fatigue grooved relief we observed areas typical for the destruction by static loads, which is typical for low-cycle fatigue [7]. We have identified the differences between the fractures of steel in the initial (Fig. 10) and modified (Fig. 11) states. In the first case we seen continuous grooves having scalloped structure (see Fig. 10,c,d), and also areas with a relief which is characteristic to static fracture and represented mainly brittle intergranular cleavage facets (see Fig.10,a,b).

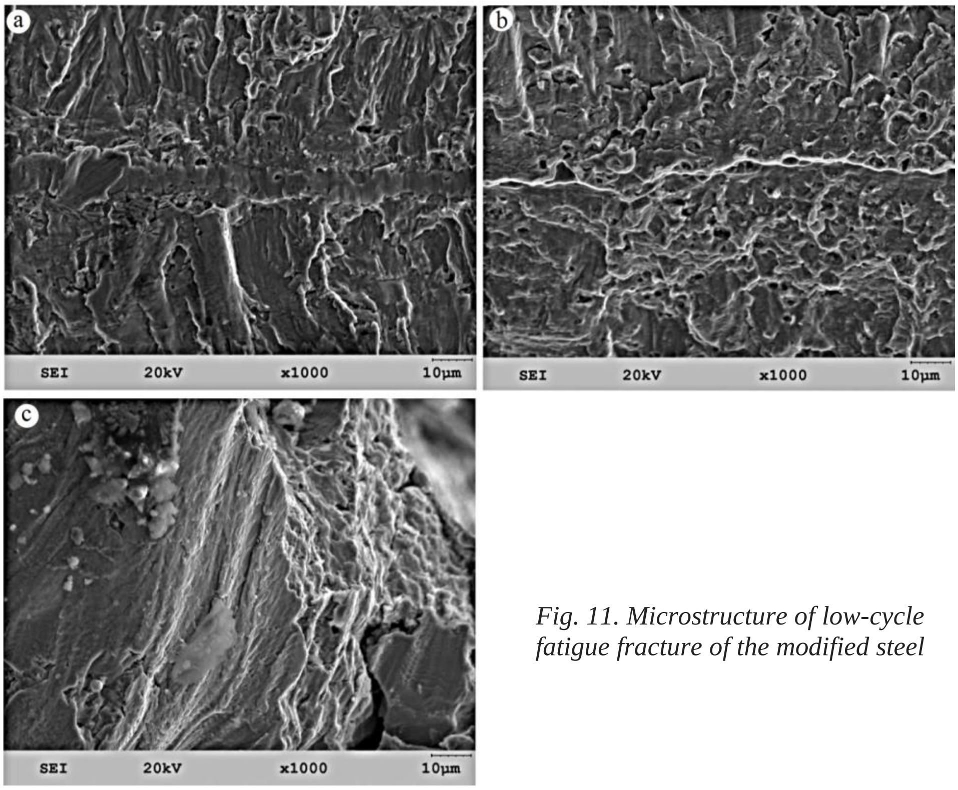


Fig. 11. Microstructure of low-cycle fatigue fracture of the modified steel

Grooved relief is expressed less explicitly at fracture of the modified steel. The grooves are intermittent in nature, reflecting the higher resistance of fatigue cracks propagation in comparison with the initial steel. The observed areas with a relief of static fracture have quasi-chipped (see Fig. 11,a) or low-power hole-like (see Fig. 11,b) structure. Certain elements of fracture along the faces of the grains also have a pit structure (see Fig. 11,c). All of this suggests a large energy intensity of fracture the modified steel in comparison with the initial steel at high stress amplitudes.

The positive effect of the modification on the fatigue fracture resistance of austenitic steel 08Cr18Ni10Ti should be connected with the following experimental results:

1 – correlation between the increase of strength properties and the endurance limit;

2 – decrease in the mean grain size and more uniform distribution of grain size;

3 – irregular character of grooves at the fracture surface microrelief.

Taking into account these results, consider briefly the possible causes observed changes in the fatigue characteristics of austenitic steel as a result of the modifying.

The analysis of the data in Figure 4 shows that the increase in the endurance limit $(\sigma_{RM}-\sigma_{RI})/\sigma_{RI}$ is ~ 10%, which agrees well with the change in the yield strength (8%) and a lesser degree with the change in tensile strength (2.3%) (see Table). This conclusion is consistent with the relationship of yield strength and physical endurance limit. It is manifested in the deceleration of the processes of shear formation and damageability due to creation stronger surface layer at the stage of microplastic deformation; the layer is a barrier for dislocations exiting on the surface [8, 9].

The authors [10] believe that the relationship between the endurance limit and grain size based on their data, responds well to the Hall-Petch relationship. In our case this regularity is shown in correlation of increasing the endurance limit to ~ 10% and of reducing the average grain size by about 25%.

Direct influence of nanoparticles of oxides on the characteristics of fatigue was manifested in the following.

It was marked above that shorter and winding grooves observed on fractographic figures at multicyclic fatigue and the intermittent grooves in low-cycle fatigue are due to higher energy intensity of fracture of the modified steel in comparison with the initial steel, especially at high stress amplitudes. According to [11–13] nanoparticles disposed within the body and at the grain boundaries impede propagation of cracks and increase the crack resistance due to contraction diverging cracks passing through the nanoparticle.

As it is known vacancies are intensively generated and redistributed to the sinks in the process of alternating deformation. The oxide nanoparticles are effective traps for vacancies in ODS steels [14]. When fatigue tests of modified steel, one would expect a decrease of vacancy flows to the top of the developing microcrack and reduce the rate of its growth. According to [3] the additives of nanosized zirconium oxides in steel 08Cr18Ni10T prevent the accumulation of phosphorus at the grain boundaries, which also contributes to slowing development of fatigue damage.

CONCLUSIONS

We have investigated fatigue on alternating cantilever bending steel 08Cr18Ni10T in the initial and in the modified state (additives of nanosized oxides of Zr and Y).

It was shown that the modified steel is characterized by essential increase (> 30%) of fatigue strength at the low-cycle fatigue and slight its change (~ 10%) when a large number of loading cycles.

Correlations of increasing endurance limit and reducing mean grain size which corresponds to the well-known Hall-Petch relationship for the fatigue tests is shown.

The positive role of nanoscale oxide additives to reduce the intensity of fatigue damage consists in braking microcracks, including due to the capture of deformation vacancies and preventing the accumulation of phosphorus in the grain boundaries.

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УСТАЛОСТЬ АУСТЕНИТНОЙ СТАЛИ, МОДИФИЦИРОВАННОЙ ОКСИДАМИ ЦИРКОНИЯ И ИТТРИЯ

С.И. Аксенова, Б.В. Борц, И.М. Короткова, А.В. Пахомов, В.И. Соколенко

Представлены результаты усталостных испытаний на консольный изгиб стали 08Х18Н10Т в исходном и модифицированном состояниях (добавки ноноразмерных частиц оксидов Zr и Y). Для модифицированной стали показано существенное увеличение усталостной прочности в области малоцикловой усталости и незначительное изменение этой характеристики при большом числе циклов нагружения. Наблюдаемые изменения усталостных свойств связываются с различием характера зарождения и развития усталостных повреждений.

ВТОМА АУСТЕНІТНОЇ СТАЛІ, ЩО МОДИФІКОВАНА ОКСИДАМИ ЦИРКОНІЯ І ІТРІЯ

С.І. Аксьонова, Б.В. Борц, І.М. Короткова, А.В. Пахомов, В.І. Соколенко

Представлені результати втомних випробувань на консольний вигин сталі 08Х18Н10Т у вихідному і модифікованому стані (добавки нанорозмірних часток оксидів Zr і Y). Для модифікованої сталі показано істотне збільшення втомної міцності в області малоциклової втоми і незначна зміна цієї характеристики при великому числі циклів вантаження. Спостережувані зміни втомних властивостей зв'язуються з відмінністю характеру зародження і розвитку втомних ушкоджень.