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Microstructural changes and fracture mechanisms in VT1-0 titanium in sub- and microcrystalline states after high-pressure treatment and fatigue testing

© M.V. Narykova[1], S.S. Manohin[2], V.I. Betekhtin[1], Yu.R. Kolobov[2], A.G. Kadomtsev[1], O.V. Amosova[1]

[1] Ioffe Institute,
St. Petersburg, Russia
[2] Federal Research Center of Problems of Chemical Physics and Medicinal Chemistry RAS,
Chernogolovka, Russia
E-mail: Maria.Narykova@mail.ioffe.ru

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The structure of VT1-0 samples was studied after fatigue tests under cyclic loading in sub- and microcrystalline states after additional treatment with high hydrostatic pressure. Fractographic studies of fatigue fracture surfaces have been performed using optical and scanning electron microscopy. Using the method of backscattered electron diffraction and transmission electron microscopy on thin foils, the evolution of the grain and dislocation structure, as well as the crystallographic texture of the studied material, has been studied. The parameters of the defective structure (nanopores) were determined using small-angle X-ray scattering and densitometry. The differences in the fracture patterns of the sub- and microcrystalline states of titanium VT1-0 under different fatigue loading parameters are discussed.

Keywords: titanium, VT1-0, microstructure, fracture, nanopores, fatigue.

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1. Introduction

Titanium and its alloys, due to unique combination of specific strength, corrosion resistance and biocompatibility, found wide application in aerospace industry, medicine and chemical machine building [1]. Technically pure titanium of VT1-0 grade holds a specific place and is used to manufacture medical implants, power equipment elements and structures, operating in aggressive media [2]. However, the traditional coarse-grained (CG) titanium has limited fatigue strength, which reduces the durability of the items under cyclic loads [3]. In the recent decades the attention of the researchers was concentrated on development of ultra-fine grain (UFG) and submicrocrystalline (SMC) structures in titanium, which demonstrate considerable improvement of mechanical properties. For example, paper [4] shows that the grain size of less than $1\,\mu$m increases the yield strength of titanium VT1-0 by $75-85\,\%$ compared to the CG-state. However, such materials are prone to recrystallization and degradation of the structure under thermocyclic exposures, which limits their application. Besides, as noted in paper [5], severe plastic deformation (SPD) methods used to produce UFG-structures often result in accumulation of internal stresses and microcracks, which reduces the fatigue resistance, and provides for low capability of the material to maintain the internal structure under thermal and thermomechanical exposure. In such materials the microstructure changes may take place, and the specimen grain size may increase in process of heat exposure or thermocyclic loading. A relevant area is search for post-treatments capable of clearing the defects of SMC structures without sacrificing their advantages. One of such methods is deemed to be exposure to high hydrostatic pressure. As shown in papers [6,7], pressure in the range of $1.1-5$ GPa may cure micropores and improve long-term strength (for example, in tensile tests in creep mode). Nevertheless, the impact of high hydrostatic pressure at fatigue behavior of titanium VT1-0 in SMC-state remains poorly studied.

Previously the authors found that after SMC titanium exposure to hydrostatic pressure, conventional endurance limit substantially increases in a certain area of its cyclic loading [8]. Therefore, the goal of this paper is to study the microstructural changes after fatigue tests under cyclic loading of VT1-0 alloy specimens in SMC and MC states previously exposed to high hydrostatic pressure, to compare this data with the data obtained previously for the source specimens. Special attention is paid to analysis of damage morphology and evolution of the dislocation structure under various loading modes. The paper results supplement the existing data [9–11] and may be used to optimize the technology for processing of titanium alloys used in critical structures.

2. Materials and research methods

An object of research were specimens of titanium alloy VT1-0 in SMC and recrystallized microcrystalline (MC) states before and after exposure to high hydrostatic pressure and subsequent tests in the cyclic loading mode. For structural research, specimens were used after cyclic loading in a wide range of stresses. Detailed description of the cyclic loading modes and specimen dimensions are published in paper [8].

The fractography of the damaged specimens fracture structure was investigated using scanning-electron microscopes Quanta 200 3D and Nowa NanoSEM 450 in topographic contrast mode using a secondary electron detector.

Measurements by method of small-angle X-ray scattering (SAXS) were carried out in a special small-angle X-ray installation with the help of a high resolution goniometer ($2''$). Filtered MoK_α radiation was used (wavelength $\lambda = 0.0707$ nm) with Kratky collimation in a narrow beam. Scattered radiation was detected using a scintillation counter. Measurements were carried out in the range of angles 0.9 ($0.9'$) — 10 ($10'$) min, therefore, a wide range of heterogeneity dimensions was covered (from ~ 5 to ~ 200 nm). The calculation of the volume share of scattering heterogeneities was carried out using Guinier approximation [12].

Density of specimens before and after exposure to high hydrostatic pressure was determined at room temperature by method of hydrostatic weighing with accuracy of 0.01 %. The working fluid was distilled water with density of 0.9982 g/cm^3 (at temperature 293 K).

The specimens were treated with high hydrostatic pressure at $P = 1.5$ GPa, and the working fluid was industrial oil, the healing time was 5 min (M.N. Mikheev Institute of Metal Physics of the Ural Branch of the Russian Academy of Sciences, Yekaterinburg).

3. Study results and discussion

3.1. Fractographic research of fracture surface

As it was shown in paper [8], pressure treatment introduces practically no changes to the shape of the Woehler curve for titanium specimens in MC state. In SMC state the pressure impact will not manifest itself in process of cyclic tests under small loads, which results in the fact that it practically does not change the fatigue strength of the specimens, however, has substantial impact at larger loads. The result of such treatment is significant increase of the number of cycles prior to damage — from two to three times. Therefore, in analysis of experimental data we will consider mostly two loading intervals: with larger and smaller loads and, accordingly, fewer or more cycles prior to damage N. For SMC of titanium the high load area will be conventionally defined at $\sigma \geq 650$ MPa. Under such tension the number of cycles to damage as per Woehler curve [8] is around $5 \cdot 10^5$ after treatment with high hydrostatic pressure and $2 \cdot 10^5$ — for the specimens not exposed to pressure. For MC states we will also consider the loading areas as conventionally separated by the tension value $\sigma = 500$ MPa. Under such tension the number of cycles prior to damage for MC specimens is around $3.7 \cdot 10^5$ and practically does not differ for the specimens before and after exposure to pressure.

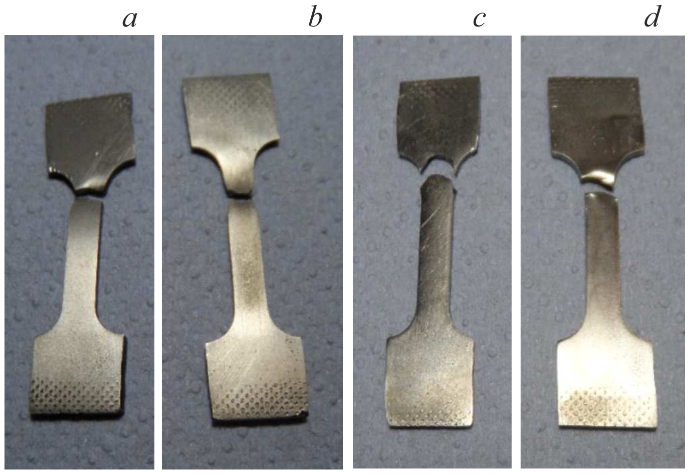

Figure 1. Optical images of specimens VT1-0, processed by pressure after cyclic tests: a — MC ($\sigma = 480$ MPa, $N = 7.8 \cdot 10^5$), b — MC ($\sigma = 630$ MPa, $N = 7.1 \cdot 10^3$), c — SMC ($\sigma = 633$ MPa, $N = 5.8 \cdot 10^5$), d — SMC ($\sigma = 733$ MPa, $N = 2.3 \cdot 10^5$).

Figure 1 presents images of fractures in VT1-0 specimens exposed to pressure with MC (Figure 1, a, b) and SMC (Figure 1, c, d) structure, after cyclic testing. Figure 2 provides the results of fractographic studies of the fracture surface in titanium alloy specimens for various fatigue loading ranges.

As you can see from the results of optical metallography (Figure 1, a, b), the break line in specimens with MC structure is even, inclined to the axis of the specimen approximately by angle of 6–8 degrees. In the damage zone the fracture surface morphology indicates quasi-viscous nature of the fracture. The difference from the specimens not exposed to pressure is the absence of many cracks. The grain boundaries are sharp, which is specified for a brittle rather than a viscous fracture.

For specimens with SMC structure (Figure 1, c, d), a U-shaped form of the fracture is specific, which is pronounced for specimens with small load — less than 650 MPa (Figure 1, c) and more even fracture with inclination of around 8 degrees for specimens with larger load (Figure 1, d). Such U-shaped form of the fracture is likely related to heterogeneous formation of microcracks on the side surface of the specimen. In the damage zone the fracture surface morphology indicates quasi-brittle nature of the fracture for the specimens with small load and quasi-viscous nature for the specimens with larger load (Figure 2). The difference from the specimens with MC structure is the availability of microcracks specific river pattern fracture, which indicate a brittler nature compared to the specimens with MC structure. It should be noted that for an SMC specimen with the minimum load the

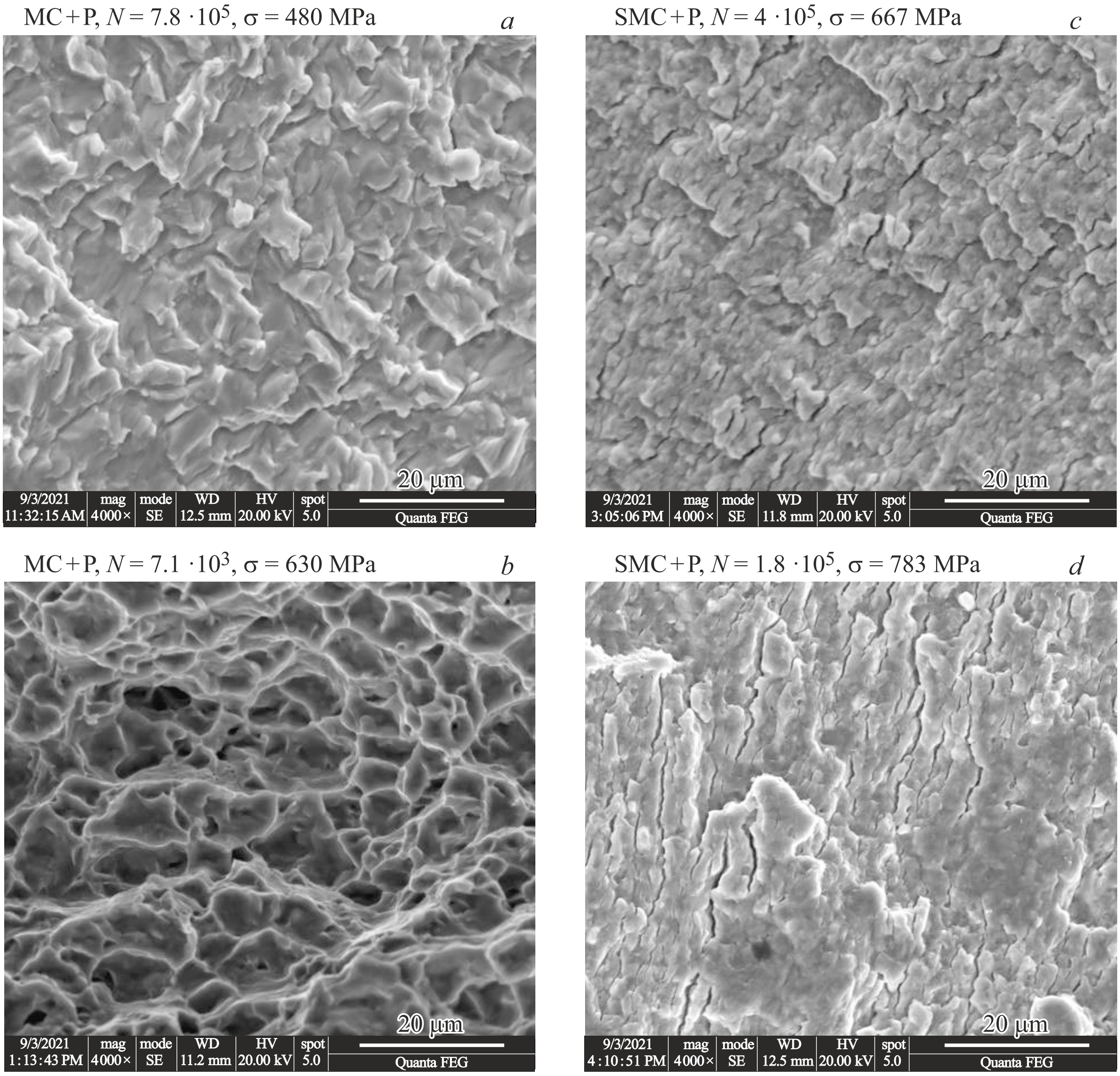

Figure 2. Electron microscope images of the damage surface perpendicular to the fracture surface of VT1-0 titanium alloy specimens in MC (a, b) and SMC (c, d) states, after pre-pressure treatment (+P).

fracture is brittler compared to other specimens. A brittler fracture of an SMC specimen with minimum load is due to the fact that exposure to pressure, while reducing the total number of defects, strengthens the role of the boundaries in the process of failure. Under long-term cyclic loading with small amplitudes (high number of cycles prior to failure), the damage is gradually accumulated primarily in the grain boundaries, and not in the volume of grains. This is promoted by higher density of dislocations near the boundaries after exposure to pressure, which is confirmed by the data of electron microscopic studies conducted in this paper. In contrast to the tests with high loads, which activate the plastic deformation mechanisms in the grain body, under low loads the intergranular damage prevails, which makes the fracture look more like the brittle damage. Such behavior, as we know, is specific for submicrocrystalline materials, where the grain boundaries play a key role in the deformation and failure mechanisms.

3.2. Investigation of the morphology of the specimen surface

Figures 3 and 4 presents the results of a study of the morphology of the surface perpendicular to the fracture surface of titanium samples pre-treated with high hydrostatic pressure after fatigue testing for tension in MC and SMC states.

The surface of the specimens with the initially smooth surface and shallow specially applied cracks (left after polishing with subsequent use of paper with graduation 2000, are directed along the axis of the specimen — load

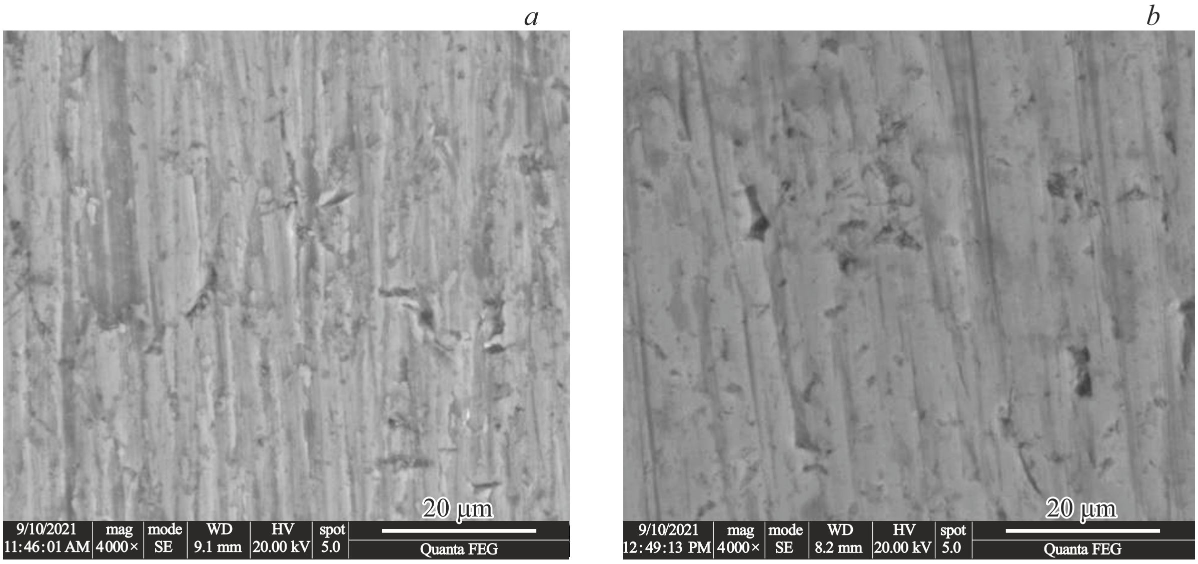

Figure 3. Electron microscope images of fracture surface in specimens of VT1-0 titanium in MC (*a*) and SMC (*b*) states after pre-pressure treatment and fatigue tension tests, $N = 2 \cdot 10^6$. Specimens were not brought to damage.

direction under cyclic tests) remains practically unchanged for the specimens with the low load that were subjected to more than $2 \cdot 10^6$ cycles and did not damage (Figure 3). This is fair for the specimens with both MC and SMS structure. In the specimen with the MC structure few nucleating microcracks were found, which were oriented perpendicularly towards the load direction.

In the specimens with higher load and MC structure (fractured specimens) the curvature of the surface cracks is observed, which indicates the change in the surface relief, and the height of irregularities of the surface relief increases as the applied load increases up to the complete disappearance of the cracks (Figure 4, *a*). This is observed for the specimen with maximum load, which withstood the least number of cycles before failure. Except for the surface microcracks, micropores are observed, the density and size of which are maximum for a specimen with the maximum load. For the specimen with the MC structure the plastic yielding area is around 1.5–2 mm, while in the specimens with smaller load it varies within several hundreds of microns.

For the specimens with the SMC structure the plastic yielding area is much smaller than for the specimens with the MC structure and does not exceed 250 μm. In the specimen with the SMC structure at maximum load in contrast to the similar one with the MC structure, there is no zone of surface relief change observed.

3.3. Investigation with methods of transmission electron microscopy and EBSD

Studies of the texture with the method of electron backscattered diffraction (EBSD) patterns analysis demonstrated that all titanium specimens exposed to pressure both in the initial state and after cyclic tensile testing detected the axial texture with the texture axis $\langle 01\bar{1}0 \rangle$. This is fair for the specimens with the MC structure and for the specimens with the SMC of titanium. The presence of such texture is related to the method of bar manufacturing, namely, combination of radial displacement and flat-and-edge rolling.

As it was shown in paper [8], the MC and SMC titanium specimens grain size did not change substantially after the cyclic tests. By way of example, Figure 5 shows data for the MC specimens of titanium before and after fatigue testing that have been previously exposed to pressure. Figure 6 shows images of the grain structure of MC and SMC titanium before and after fatigue testing (for two loading ranges) of pressure-treated samples. In MC titanium the density of dislocations is low, and after the testing it increases substantially. You may detect both individual dislocations and dislocations localized in small-angle boundaries. Density of dislocations is higher in the specimen with low load that withstood more cycles before failure.

In the SMC specimen the level of internal stresses in the specimen is high. In the specimens after the cyclic testing the structure will not change in principle, however, the level of internal stresses will rise compared to the initial specimen, which may be qualitatively judged upon by increase in the density of dislocations and extinction contours with small curvature radius.

It should also be noted that the level of internal stress (density of dislocations inside the grains and presence of bending extinction contours) in the specimens after the exposure to hydrostatic pressure is much higher than the

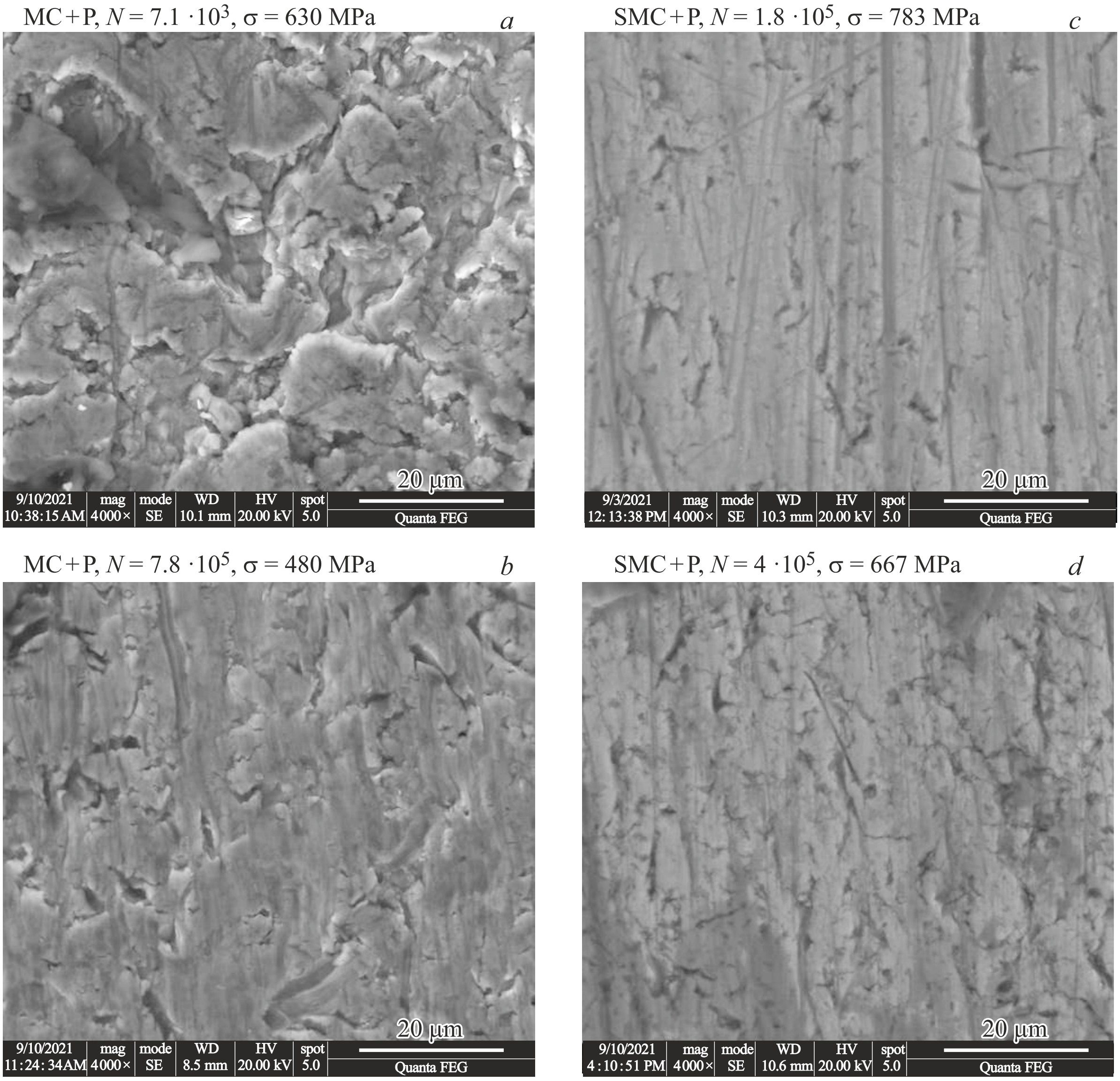

Figure 4. Electron microscope images of fracture surface in specimens of VT1-0 grade titanium in MC (*a, b*) and SMC (*c, d*) states after fatigue tension tests to damage.

stress level in the specimens that were not exposed to pressure.

3.4. Investigations by method of small-angle X-ray scattering and densimetry

Dependence of scattering intensity I on scattering angle φ for MC and SMC states before and after exposure to high hydrostatic pressure is shown in Figure 7. You can see that curves (*2*), corresponding to the pressure-exposed state, are located below the curves (*1*), corresponding to the initial state. As it was shown in paper [13], use of high pressure remains an effective method to reduce material porosity. Analysis of indicatrices of small-angle X-ray scattering, presented in Figure 7, *c* using Guinier appoximation [12] demonstrated that after exposure to pressure the volume fraction of scattering heterogeneities in MC and SMC decreased. According to the estimates, their specific size prior to pressure treatment was around 20 nm. The volume fraction of heterogeneities after exposure to high hydrostatic pressure for SMC state reduced by 0.2 %, for MC this value is significantly smaller — less than 0.1 %. After application of pressure used as well to clarify the cavity nature of discontinuities, in SMC state you may note the reduction in size of scattering heterogeneities (with their stable concentration), which amounted to, according to the estimates, $\sim$ 10–15 % — down to 16–17 nm. For MC state these changes are negligible and are less than 5 %.

Data obtained by the SAXS method were confirmed by the results density determination by hydrostatic weighing. Thus, density of MC specimen increased

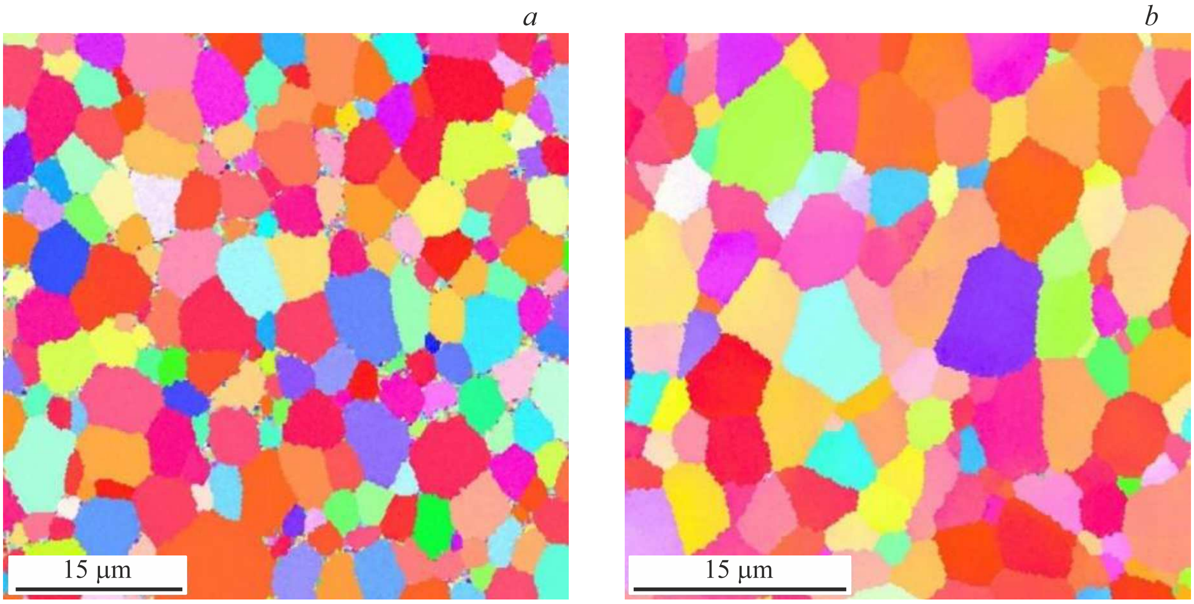

Figure 5. Microstructure of titanium MC specimen after exposure to pressure to (a) and after (b) fatigue testing at $\sigma = 560$ MPa, $N = 1.1 \cdot 10^6$.

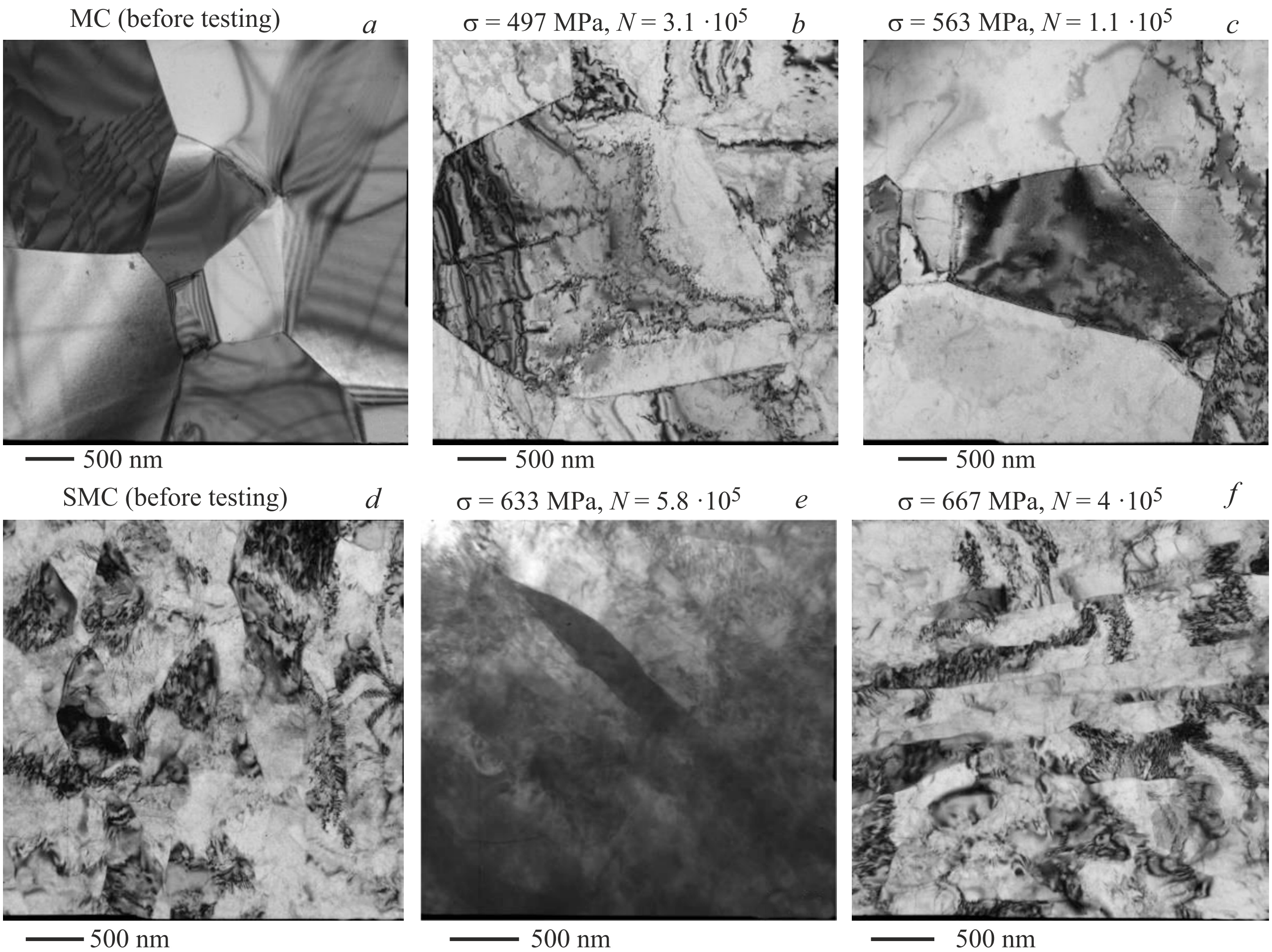

Figure 6. TEM-images of grain structure of the specimens exposed to high hydrostatic pressure of titanium VT1-0 with MC (a, b, c) and SMC (d, e, f) structure, before and after cyclic tensile strength testing.

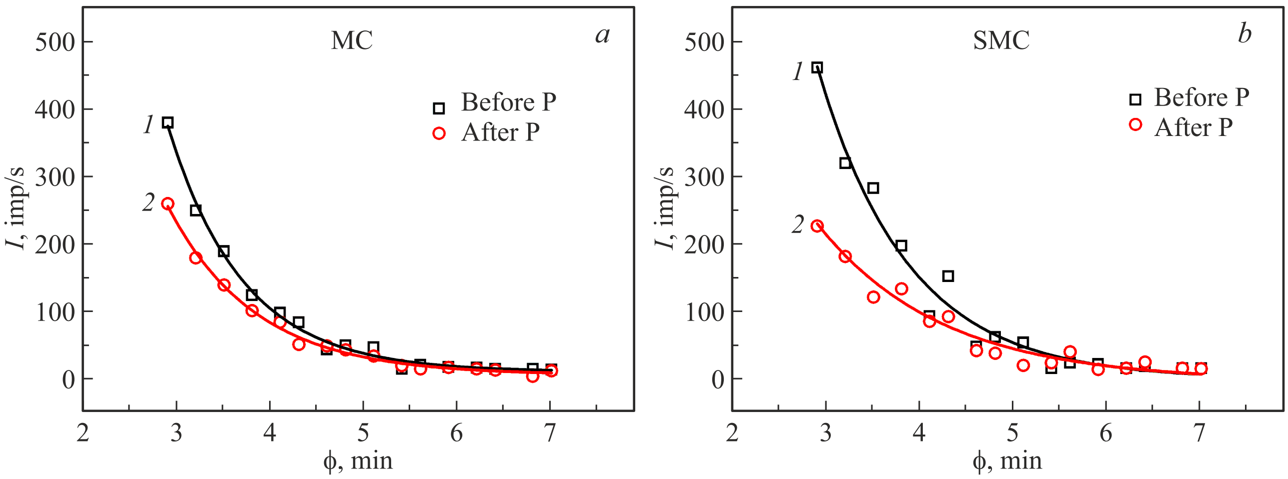

Figure 7. Indicatrices of scattering for MC (*a*) and SMC (*b*) specimens of VT1-0 before (curve *1*) and after (curve *2*) exposure to high hydrostatic pressure.

Comparative analysis of failure of MC and SMC specimens of titanium in the range of high loads in fatigue loading mode

Parameter	MC state ($N \leq 5 \cdot 10^5$)	SMC state ($N \leq 5 \cdot 10^5$)
Nature of failure	Quasi-viscous failure	Quasi-brittle failure
Microcracks	Large, oriented across	Fine on side faces of the specimen, along the boundaries
Internal stresses	Low level	High level
Zone of plastic of strain	Wide (up to 2 mm), expressed plastic flow	Narrow (less than 250 μm, localized flow)
Relief	Surface microcracks are observed micropores with high density	No zone of surface relief change is observed
Micropores	Micropores with diameter $1-5\,\mu$m	Submicron defects along grain boundaries

from 4.4987 to 4.5036 g/cm^3 after pressure, for SMC state — from 4.4963 to 4.5054 g/cm^3. The obtained results for variation of density determined by densitometric method, amounted to accordingly 0.11 % and 0.2 % for MC and SMC specimens. Higher density (before pressure) for MC state is likely due to the fact that the MC structure was formed from SMC state by recrystallization annealing (at $T = 550^\circ$C). Thermal treatment, as you know [14–16], is the effective method to cure micro- and nanopores, which may be formed in process of large plastic deformations in metal materials [17–21], which results in the increase of their density.

4. Results and discussion

Above it was shown that the highest impact under fatigue testing was found in higher loads, when the number of cycles to failure is relatively small — less than $5 \cdot 10^5$. Let us summarize some data for analysis on the results of structural investigations after fatigue testing of MC and SMC specimens in the area of high loads into a table.

Failure of VT1-0 specimens after exposure to high hydrostatic pressure in both investigated states occurs in accordance with the mixed mechanism. Besides, in the recrystallized MC state the quasi-viscous failure of specimens prevails, especially in the rupture area under maximum applied loads. In SMC state the prevalent failure of specimens is qualitatively close in the failure pattern to the quasibrittle one.

A noticeable reduction is found in the number of microcracks per unit of surface area in the fracture area of all investigated types of specimens compared to the specimens not exposed to pressure. This is especially noticeable for specimens with MC structure.

Investigation of the morphology of the side surface of specimens before and after cyclic tests by SEM method found formation of a surface relief related to the localization of plastic flow of material in the planes of non-crystallographic shear. It was found that the size of the area with the changed surface relief near the break line for the specimen exposed to pressure is smaller than the corresponding one for the specimens not exposed to pressure. The fact that in process of specimen failure the grains practically do not extend in process of uniaxial strain in the direction of the applied load, and the absence of the specific relief in the form of parallel bands, oriented at a certain angle to the direction of the applied stresses, makes it possible to assume that the mechanism of formation of the surface relief is grain boundary sliding. Besides, a total decrease in the number of microcracks and pores per unit of surface was recorded in the pressure-exposed specimens compared to previously investigated specimens that were not exposed to pressure. Absence of the meaningful change in the number of cycles before failure in the MC state, despite the decrease in the density of cracks by 20–30 % after exposure to pressure is explained by complex nature of fatigue failure, where the decisive role is played both by the number of cracks and the dynamics of their development. Exposure to pressure decreases the number of initiated pores and cracks, probably, simultaneously modifies the microstructure so that the mechanism of growth and distribution of the dominant crack changes. This causes preservation of the total number of cycles before failure.

The methods of transmission electron microscopy found that the grain size and the evolution of the structure in process of cyclic testing in the specimens previously exposed to pressure are not in principle different from the corresponding specimens not exposed to pressure. However, the level of microstresses related to high density of dislocations and extinction contours, is much higher in the specimens exposed to pressure. The seeming contradiction between the increase in the fatigue durability and growth of internal stresses in SMC titanium after exposure to high pressure is likely explained by the fact that hydrostatic pressure removes micropores and discontinuities being the main stress risers in nucleation of fatigue cracks, which prevails over the negative effect of increased internal stresses.

5. Conclusion

1. Differences were found in the nature of failure of MC and SMC specimens of titanium VT1-0 at different parameters of loading in the cyclic fatigue mode. As the stress increases, the morphology of the MC titanium surface reflects the classic viscous failure, whereas for SMC titanium the combination of brittle and plastic mechanisms is specific. At low loads (and large quantity of the cycles to failure) quasi-brittle failure prevails for both states.

2. Exposure to high hydrostatic pressure for MC titanium will not change the failure mechanism. For SMC specimens the pressure eliminates nanopores, reduces structure heterogeneity and increases resistance to crack nucleation.

3. Pressure exposure is most effective for the SMC state, improving the microstructure homogeneity and suppressing defects.

These features are important for predicting the durability of the items from titanium alloys in real operating conditions.

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Conflict of interest

The authors declare that they have no conflict of interest.

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