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# Research of fatigue failure of sub- and microcrystalline titanium VT1-0 after its treatment with high hydrostatic pressure

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The fatigue failure of titanium with submicrocrystalline (SMC) and microcrystalline (MC) structure formed during severe plastic deformation after exposure to high hydrostatic pressure was investigated. The features of the pressure effect on the conditional fatigue limit for SMC and MC titanium were identified and analyzed.

**Keywords:** VT1-0, submicrocrystalline titanium, ultrafine-grained structure, fatigue, internal stresses, nanopores.

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## 1. Introduction

Titanium, in virtue of its features (high strength and corrosion resistance with small specific weight) finds rather extensive application in industrial manufacturing. Besides, high-strength titanium with submicrocrystalline (SMC) structure is used in some critical areas of engineering and medicine.

One of the main methods to produce high-strength SMC metal materials is severe plastic deformation (SPD) — tremendous plastic deformation under quasi-hydrostatic conditions [1,2]. However, SPD leads to nanoscale voids (such as pores, cracks) in metals. Such voids were found in some papers [3–8]. The authors [9] believe that the voids arising from SPD are a sort of channels for dissipation of a portion of great energy introduced into the metal by SPD. Without this dissipation, if SPD extend increases, the metal integrity may be lost (it will fracture). For example, from multiple passes of equal channel angular extrusion — one of the most popular SPD methods.

As demonstrated in [10], the voids produced by SPD has practically no effect on the high values of static strength (tensile strength, yield strength). However, under a long-term load such „original" voids formed in the process of SPD may become the „foci" for development of damageability causing lower durability and thermal stability of high-strength SMC metal materials, i. e. reduction of their operation resource.

In [3,5] it was shown that application of high hydrostatic pressure caused decrease in the volume of nanoscale voids formed under SPD and increase in the durability of titanium and other SMC metals under a long-term load in the mode of uniaxial elongation.

This paper for the first time studied the healing effect of hydrostatic pressure when titanium is tested in the mode of fatigue cycling, which is most often implemented when operating universal metal items. For high-strength SMC titanium, which may be used to make heavy duty parts in aerospace field and medical field to make implants introduced into the human body, fatigue cycling analysis is especially relevant.

This paper studied the effect of high hydrostatic pressure at the features of fatigue damage of SMC titanium, prepared under SPD, and for comparison, of the same titanium with microcrystalline (MC) structure.

## 2. Materials and research methods

A study object was the specimens of titanium alloy VT1-0 in SMC and MC states before and after treatment with high hydrostatic pressure. The total content of admixtures (C, N, Fe, O, H, Al, Si) was $\sim 0.3$ wt.%. The SMC structure of titanium was produced using the original technology of cross rolling in the combination with the rolling for the specified profile [11]. The MC structure was produced after annealing of titanium with the SMC structure for an hour at 823 K. Microscopic studies showed that the average grain size for SMC titanium was 190 nm, for MC titanium — $2-3\,\mu$m. 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 Ural Branch of Russian Academy of Sciences).

To study the fatigue properties, double-T specimens were made with the working area thickness of 1 mm and width of 3 mm. The Instron Electropulse E3000 testing machine equipped with an electromagnetic drive was used in a loading mode with a frequency of 50 Hz at room temperature. The tests were carried out according to the tensile loading scheme in a symmetric sawtooth cycle in the loading region $0.9\sigma_{\max}-0.2\sigma_{\max}$.

The volume of voids (nanopores and cracks) was assessed by the precision method of density measurement using the analytical scale Shimadzu 120D with an attachment SMK-301; relative accuracy $\Delta\rho/\rho$ was 0.01%. Information about the presence and parameters of nanopores was obtained by the method of small-angle X-ray scattering (SAXS), which was used in previous works on the identification of the nanoporosity arising from SPD [3].

## 3. Experimental results and discussion thereof

Let us consider and analyze the experimental data on the effect of high hydrostatic pressure at fatigue damage of titanium with SMC and MC structure.

The figure shows fatigue curves in the area of loads $\geq 450$ MPa and $\leq 2\cdot 10^6$ testing cycles. One can see that these dependencies for titanium with SMC structure are of complicated nature. In the area of high (620−800 MPa) stresses and up to $\approx 10^5$ testing cycles the fatigue curve for the specimens having been exposed to the pressure is much higher than for the same specimens before pressure application. The produced data show that for these testing conditions the Conventional fatigue limit (CFL) after pressure treatment increases; the maximum growth of CFL is $\approx 2.5$ times.

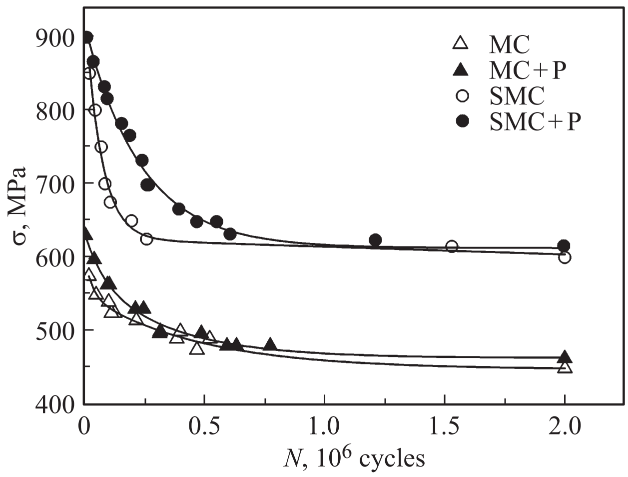
With the load over 620 MPa and growth of the number of cycles to $2\cdot 10^6$ and more the fatigue curves for titanium before and after pressure application come close. One may only say that pressure application does not decrease, but even slightly ($\approx 1.05$ times) increases CFL and therefore stabilizes its value as the duration of cyclic loading increases.

For titanium with MC structure the effect of pressure application at fatigue curves was found only at relatively short test cycles, and the strengthening effect is substantially smaller than for SMC state (see Figure).

Let us analyze the possible reasons for the found features of hydrostatic pressure effect at fatigue characteristics of titanium with SMC and MC structure.

As it was already noted, in process of SPD both high-strength ultra-fine grain (UFG) structure and nanoscale porosity are formed. Healing of this „original" nanoporosity under the effect of high hydrostatic pressure causes higher durability (service life) in high-strength SMC metal materials when loaded in stretching mode for a long period of time [3]. Therefore, this paper studied the effect of hydrostatic pressure at healing of the „original" nanoporosity (formed under SPD) in SMC titanium, which was tested in the fatigue loading mode often implemented in practice.

Using methods of SAXS treated according to [12,13], and precision measurement of density, SMC titanium (and MC titanium, for comparison) specimens were tested before and after exposure to high hydrostatic pressure. According to SAXS data for SMC titanium given in [14], the volume




Dependence of stress on the number of $N$ cycles prior to damage during fatigue tests of alloy VT1-0 in MC and SMC states, and also after their treatment with high hydrostatic pressure (+P).

share of nanopores is $\sim 4\cdot 10^{-4}$. Similar studies carried out for titanium in the MC state practically did not reveal the nanoporosity presence. Obviously, high-temperature annealing, which was used to obtain MC titanium, led to significant healing of nanopores.

The precision method of hydrostatic weighing was used to measure the density of titanium and the share of this density variation under pressure. It was found that the density prior to pressure was 4.4963 (for SMC), and afterwards it increased to 4.4983 g/cm$^3$. The total variation of density was $4.5\cdot 10^{-4}$. This deconsolidation value is well-matched with the SAXS results and confirms that the main share of SAXS is due to scattering on nanopores[1]. No significant density rise was observed for MC titanium, which is most probably due to low-temperature annealing for its manufacturing, which also reduces nanoporosity of metal materials.

With account of data on the effect of the pressure at nanoscale porosity, let us consider and analyze their connection with the results of fatigue tests. Electron microscopic studies demonstrated that after cyclic testing the structure (SMC and MC) would not change drastically. Substantial increase of CFL for the specimens exposed to pressure may unambiguously be related to the decrease in the nanoscale voids after exposure to high pressure. Significant reduction of effect from the CFL growth at stress below 620 MPa and growth of the test cycles is obviously due to the appearance of structural factors that mitigate the strengthening effect of healing.

The following data also confirm the effect of nanoscale porosity at fatigue tests. Experimental points „stress — number of cycles" (figure) were approximated by dependence of $\sigma = \sigma_0 + A\exp(-N/t)$ type. The date of estimated coefficients $(A, t)$ and errors in their detection upon approximation are shown in the table.

[1] The similar result was obtained for SMC aluminum subjected to hydrostatic pressure. Porosity reduction upon healing according to SAXS data — $\approx 2.5\cdot 10^{-3}$, and by density measurement — $3\cdot 10^{-3}$.

Estimated values of parameters $\sigma_0, A$ and $t$ under approximation of curves „number of cycles to damage/average load per cycle" with function $\sigma = \sigma_0 + A\exp(-N/t)$

| | MC | SMC | MC+P | SMC+P |
|---|---|---|---|---|
| $\sigma_0$ (MPa) | $450 \pm 8$ | $611 \pm 8$ | $471 \pm 5$ | $614 \pm 13$ |
| $A$ | $108 \pm 8$ | $331 \pm 23$ | $157 \pm 7$ | $312 \pm 19$ |
| $t$ | $(422 \pm 75) \cdot 10^3$ | $(76 \pm 9) \cdot 10^3$ | $(213 \pm 24) \cdot 10^3$ | $(241 \pm 31) \cdot 10^3$ |

As one can see from the fatigue curves, the pressure treatment for SMC state provides substantial effect on the progress of the curves. The result of such treatment is significant increase of the number of cycles prior to damage — from two to three times, which is obviously an important positive result.

It is also necessary to note a significant change for the SMC state in the value $t$ in the exponential curve of $\sigma = \sigma_0 + A\exp(-N/t)$ function, to which the fatigue curves were approximated with preservation of $A$ pre-exponential factor value. In this case parameter $t$ is related to the material structure. Increase of this parameter by more than 3 times — from $76 \cdot 10^3$ to $241 \cdot 10^3$ implies a significantly more difficult origination of cracks, which is most probably related to reduction of the quantity of defects in the specimen after exposure to pressure.

It was shown previously that nanoporosity formed under SPD has practically no effect on the characteristics of strength under „quick" loading (ultimate strength, microhardness etc.). Note that at the minimum number of cycles $\approx 2 \cdot 10^4$ the absolute values of increase after pressure treatment for both types of the structural states were close and made around 50 MPa. One may suggest that CFL increase is most probably due to not only healing of nanopores (even though this fact is also important), but the growth of internal stresses after exposure to hydrostatic pressure, which may be thoroughly concluded by increased density of dislocations and extinction contours with a small radius of curvature.

As the duration of the test increases, the difference between CFL for SMC titanium before and after the pressure varies non-monotonically. One of the possible reasons for such behavior are the high internal stresses, the sources of which may be the high angle boundaries ($\varphi > 15°$) and their triple junctions formed under high plastic deformations. Long-term tests result in relaxation of internal stresses, the main methods of which are the processes of defect evolution, such as formation and displacement of dislocations, rearrangement of grain boundary defects, rotation turns of grains, formation of vacancies and their coagulation into nanopores etc. [15]. As a result the volume share of nanopores in SMC titanium specimens after the pressure becomes quite close in the value for a specimen that has not been exposed to the pressure. The effect from healing of the original porosity reduces during long-term tests, and the accumulation of deconsolidation becomes substantial. Its value comes close to the critical value of around 1.2% regardless of the additional exposure to high hydrostatic pressure.

## 4. Conclusion

It was found that after SMC titanium exposure to hydrostatic pressure, CFL confidently increases in a certain area of its cyclic loading. CFL growth according to data of SAXS and density measurement is due to healing of some nanoscale porosity formed in process of SPD during SMC structure formation.

As the stress decreases, and the number of cycles to damage rises, the effect of CFL growth noticeably decreases. The completed analysis makes it possible to relate the reduction of CFL to formation of high internal stresses in process of SMC metal production and testing.

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### Conflict of interest

The authors declare that they have no conflict of interest.

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