Peculiarities of structure and properties of titanium during fragmentation and reversal in a torsion process under high pressure

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Abstract. Influence of a reverse rotation of the moving striker of a Bridgeman chamber (torsion under high quasi-hydrostatic pressure at room temperature) on the structure and mechanical properties of technically pure titanium (VT 1-0) was studied. It was established that a change in the rotation direction of the moving striker during torsion had a significant effect on the conditions for separating the ß-phase, as well as on the structural characteristics of deformation fragments and dynamically recrystallized grains formed during large plastic deformations. Influence of the deformation method on the internal distortions of structure and on the mechanical properties was analyzed.

1. Introduction
Increasing the strength of pure metals, including titanium, to the level of strength of heavily doped alloys is possible by various extreme effects on the bulk material. One of the common methods of creating giant levels of deformation is high-pressure torsion (HPT) in a Bridgman chamber [1]. A special feature of metals and alloys after HPT is formation of fragmented structures with submicron size of highly disoriented fragments [2], as well as simultaneous occurrence of dynamic recrystallization processes at room temperature [3].

Plastic deformation methods can be roughly divided into two large groups: continuous and fractional. With a continuous method, in the process of applying an external load, a solid is continuously plastically deformed to the necessary degree of shape change (for example, torsion, compression, single-axis tension, and bending). With the second method, a mechanical stimuli on a solid is of a short-term nature with a possibility of realizing only one degree of plastic deformation per cycle. In this case, plastic forming processes are, as a rule, fractional and occur with the help of several cycles (passes). As an example, we can use hot or cold rolling, as well as forging or extrusion.

With a fractional method, not so much attention is paid to the effect of the number of cycles in the deformation process, such as temperature or deformation rate. However, the processes of elastic
relaxation occurring during a “pause” between individual passes, even at room temperature, can significantly affect the defect structure and, therefore, the properties of the material subjected to a certain total degree of deformation. In particular, this applies to cases when, in the process of external influences, the material is subjected to very large plastic deformations [4].

When using a method of equal-channel angular pressing (one of the popular fractional methods of creating large plastic deformations), only some articles report the number of passes through which a very large resulting deformation was achieved [4]. In [5], it was theoretically shown that non-monotonic (in particular, cyclic) loading of a material leads, with other things being equal, to less intensive grinding of grains and less tendency to fracture compared to the process of quasi-monotonic loading.

It is known that using HPT (at a pressure of more than 3.4 GPa [6]), the α-phase of titanium transforms into a simple hexagonal structure (ω-phase) [7]. During HPT, the determining parameters for ω-phase formation are the number of revolutions of movable anvil (plastic deformation value) \(N\) and the value of quasi-hydrostatic pressure in Bridgman chamber \(P\) [8, 9]. In metals of group IV, the HPT process is essentially the only method that leads to the complete phase transformation \(\alpha \Rightarrow \omega\). This made it possible, in particular, to determine the mechanical properties of ω-phase (elastic constants [10] and slip systems during plastic deformation [11]). In [12], it was found that shear stress during HPT facilitates \(\alpha \rightarrow \omega\) transformation in titanium and accelerates formation of ω-phase nuclei. The thermal stability of ω-phase was also studied there and it was found that ω-phase persists after removing deforming stresses and can be observed in the structure for a long time at room temperature, but undergoes a reverse transformation \(\omega \rightarrow \alpha\) after a short-term annealing at 155 °C. During HPT, the authors of [12] observed the following orientation relationship between α- and ω-phases: \((0001)_\alpha || (11\overline{2}0)_\omega; \ [11\overline{2}0]_\alpha || [0001]_\omega\).

The purpose of this article is to experimentally evaluate the effect of fragmentation and direction (reversibility) during torsion under high hydrostatic pressure in a Bridgman chamber.

2. Material and experimental technique

As a test material we selected technically pure titanium VT1-0 (α-phase), where the structure formation processes during plastic deformation is minimally affected by impurity atoms or excess phases. Samples in the form of plates 50 μm thick were subjected to HPT deformation \((P = 6 \text{ GPa})\) in a Bridgman chamber at room temperature \((293 \text{ K})\) with a 1 rpm rotational speed of the moving striker.

The experiments were carried out according to the diagram shown in Figure 1. The total deformation of a sample in all cases corresponded to two full turns of the movable anvil \((N = 2)\) and the true deformation \(e\) made in accordance with expression (1) the value of \(e = 7\).

\[
e \approx \ln \left( \frac{2\pi r N h_0}{h^2} \right)
\]  

where \(r\) is a sample radius; \(h_0\) is an initial sample thickness; \(h\) is a final sample thickness.

Option a in Figure 1 represents a continuous deformation in one pass \(n\), equal to two full turns of the movable anvil \((N = 2, n = 2)\). In case of option b, a sample was subjected to deformation in two passes in the same direction of the movable anvil rotation \((\leftrightarrow)\). Whereby, each pass consisted of 1 full turn \((N=2, \leftrightarrow, n=1)\). Option c represents four passes in one direction of the anvil rotation \((N=2, \leftrightarrow, n=1/2)\). Options c and \(\varnothing\) were similar to options \(b\) and \(c\), with the only difference that the directions of rotation alternately changed to opposite ones \((\leftrightarrow\leftrightarrow)\): \(c - N=2, \leftrightarrow, n=1; \varnothing - N=2, \leftrightarrow, n=1/2\). In the fractional deformation process, the duration of ‘pauses’ between individual cycles was \(20 \pm 1 \text{ min}\) at room temperature.

Structural studies were carried out after HPT using a JEM 200CX transmission electron microscope with an accelerating voltage of 160 kV. Microhardness measurements HV were performed with the help of a LECO M400A microhardness tester with a load of 50 g and a loading time of 5 sec. All tests of the local structure (transmission electron microscopy (TEM)) and microhardness were performed in areas approximately corresponding to a half of the radius of disk-shaped samples.
3. Test results

3.1 X-ray structural analysis

According to the results of X-ray structural analysis of VT1-0 alloy, it was found that the original structure consists entirely of α-Ti. The microstructure of titanium after HPT is a mixture of α- and ω-phases. Based on a detailed analysis of X-ray patterns, the volume fractions of α- and ω-phases ($V_\alpha$, relative units) were determined. The volume fraction of ω-phase at two full revolutions of the anvil ($N = 2$) is 0.32.

With a one direction deformation «→» $N=2$, $n=1/2$, the volume fraction $V_\omega$ equals 0.4, then its quantity decreases to 0.29 ($N=2$, $n=1$), and with $N=2$, $n=1/2$ its number increases and reaches the value of 0.32.

With «↔» deformation the volume fraction of ω-phase $V_\omega$ gradually increases from 0.2 ($N=2$, $n = 1/2$) to 0.32 ($N=2$, $n = 2$).

![Figure 1. Diagram of the experiment: a - continuous deformation; b, c - deformation of «→» type with a different number of fractional deformation cycles; d, e – deformation of «↔» type with a different number of fractional deformation cycles; n – is the number of revolutions per one fractional deformation cycle.](image1)

![Figure 2. Dependence of the volume fraction of ω-phase $V_\omega$ on the value of fractional deformation $n$ during non-reversible «→» (1) and reversible «↔» (2) deformation](image2)
Figure 2 shows that increase in the number of cycles leads to a noticeable increase in the volume fraction of \( \omega \)-phase \( V_\omega \) in the case of non-reversible deformation \( \leftarrow \rightarrow \). In case of reversible deformation of \( \leftarrow \leftarrow \) type, fractional deformation leads to a decrease in the number of \( \omega \)-phase.

3.2 Transmission electron microscopy

In [13], a ‘two-phase mixture’ model was proposed in order to explain specifics of the structure formed during HPT. Its essence lies in the fact that this structure consists of a mixture of two ‘phases’: the first includes fragments having a deformation origin (DO) [1], and the second consists of a group of grains formed as a result of continuous dynamic recrystallization (R) [14]. Each of these ‘phases’ is characterized by a certain volume fraction and its own size distribution.

Figure 3 shows distribution histograms of recrystallized grains and deformation fragments for each fractional deformation option, obtained on basis of TEM data. The resulting histograms are, in fact, combined and consist of two distribution histograms: one for DO and the other for R, respectively. Analysis of TEM images, as the case of continuous deformations [15], shows that a dotted distribution curve corresponds to DO, and a dash-dotted curve corresponds to R.

For each deformation mode, the quantitative share of DO and R was determined by calculating the relative areas under Gaussian distributions. The calculated values of share DO \( C_\phi \) and R \( C_R \) are shown in Figure 4a and b, depending on the value of fractional deformation cycle \( n \), for \( \leftarrow \rightarrow \) experiments (Figure 4a) and \( \leftarrow \leftarrow \) experiments (Figure 4b).

Using the procedure of dividing the experimental histograms into two components (Figure 3), it is possible to trace how an average grain size corresponding to DO and R will change as the number of HPT cycles changes. Figure 5 shows dependences of an average grain size DO \( D_\phi \) and R \( D_R \) on \( n \) for deformation experiments of \( \leftarrow \rightarrow \) type (Figure 5a) and \( \leftarrow \leftarrow \) type (Figure 5b).

Dependencies \( D(n) \) for both types of fractional deformation are of a similar nature: the average sizes of DO and R have a characteristic minimum proportionate to \( n=1 \), increasing (more so for \( \leftarrow \rightarrow \)) with both the decrease and the increase in value \( n \).

The results obtained are in good agreement with the results obtained in [16] on technically pure iron. Dependences \( C(n) \) and \( D(n) \) presented in Figure 4 and 5 demonstrate the effect of fractional deformation and can be explained in the framework of energy concept of structural phase transformations in HPT [17], taking into account the fact that an increase in the fragmentation (during deformation \( \leftarrow \rightarrow \)) reduces the elastic energy of the deformed material and, as a result, complicates the flow of dynamic recrystallization. The change in the loading nature (\( \leftarrow \leftarrow \) instead of \( \leftarrow \rightarrow \)) leads to the fact that as the fractional deformation increases, the dynamic recrystallization, on the contrary, decreases. This is most likely due to facilitation of the nucleation of recrystallized grain boundaries capable of migration under alternating fractional reversible torsional deformations.

Figure 6 presents the values of fraction R \( C_R \), calculated during the research with the help of TEM methods, depending on the fractional deformation cycle \( n \) for experiments of \( \leftarrow \rightarrow \) and \( \leftarrow \leftarrow \) types. Accordingly, the proportion of deformation fragments \( C_\phi = 1 - C_R \). It is obvious that for \( \leftarrow \rightarrow \) the fractional effect under megaplastic deformation leads to a noticeable decrease of \( C_R \) to 0.34 with \( n = 1/2 \) and a corresponding increase in \( C_\phi \) up to 0.66, which after continuous deformation (\( n = 2 \)) were almost equal (\( C_R = C_\phi = 0.45 \)). In case of deformation experiments with reversal of \( \leftarrow \leftarrow \) type, the fragmentation effect has a completely different character: \( C_R \) value tends to increase with saturation at small values of \( n \) (\( C_R \Rightarrow 0.65 \)), and \( C_\phi \) value decreases with saturation at small values of \( n \) (\( C_\phi \Rightarrow 0.35 \)).
Figure 3. Histograms of grain and fragment size distribution for various deformation options.
Figure 4. Ratio of quantitative shares of recrystallized grains $C_{p3}$ and fragments $C_{\phi}$; a – $\leftarrow\rightarrow$ deformation; b - $\leftrightarrow$ deformation

Figure 5. Average size of grains and fragments, depending on $n$, a – $\leftarrow\rightarrow$ deformation; b - $\leftrightarrow$ deformation

Figure 6. Share of recrystallized grains depending on the fractional deformation value $n$ with irreversible $\leftarrow\rightarrow$ (1) and reversible $\leftrightarrow$ (2) deformations
Figure 7 presents the dependence of microhardness $HV$ at the point corresponding to the midpoint of the sample radius ($r = 2$ mm), on the deformation in one cycle $n$ for deformation experiments of $\leftarrow\rightarrow$ and $\leftarrow\leftrightarrow$ types. It is obvious that an increase in the number of cycles leads to a noticeable decrease in $HV$ value, and the effect of fractional deformation is more pronounced for reverse deformation of $\leftarrow\leftrightarrow$ type ($\Delta HV/HV > 10\%$).

**Figure 7.** Dependence of microhardness $HV$ on fractional deformation value $n$ under irreversible $\leftarrow\rightarrow$ (1) and reversible $\leftarrow\leftrightarrow$ (2) deformation

4. Conclusions

1) It was shown that fragmentation (number of passes) and direction of torsion (clockwise or counterclockwise) under HPT in a Bridgman chamber at room temperature, have, other things being equal, a noticeable influence on the nature of the structure and mechanical properties of technically pure titanium.

2) An increase in the number of cycles leads to a noticeable increase in the volume fraction of $\omega$-phase $V_{\omega}$ in case of irreversible deformation $\leftarrow\rightarrow$. In case of reverse deformation of $\leftarrow\leftrightarrow$ type, fractional deformation leads to a decrease in the number of $\omega$-phase.

3) Relationship between the structure regions corresponding to the deformation fragments and recrystallized grains can be explained in view of the energy concept of structural phase transformations under large deformation, taking into account the fact that increasing the fractional deformation and changing the direction of deformation ($\leftarrow\leftrightarrow$ instead of $\leftarrow\rightarrow$) reduces the elastic energy of the deformed material and contributes to the processes of dynamic recrystallization.

4) An increase in the number of cycles leads to a noticeable decrease in the microhardness value $HV$, and the effect of fractional deformation is more pronounced for reversible deformation of $\leftarrow\leftrightarrow$ type.
Acknowledgments
The authors are grateful to RFBR for financial support (Grant No. 18-08-00640a). The work was also carried out in the course of a state assignment of the Ministry of Education and Science of the Russian Federation No. 2017/13 (2097).

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