

Mechanical properties of the nanostructured Ti processed by combination of the severe plastic deformation methods

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DOI: 10.26565/2222-5617-2018-28-06

The polycrystalline Ti Grade2 was subjected to combination of different methods of the severe plastic deformation, such as equal channel angular pressing, quasi hydro extrusion and rolling, and eight different structural states were produced. For each state were measured the following parameters: average grain size, microhardness (at 300 K) and mechanical characteristics in uniaxial compression at 300, 77 and 4.2 K. The concept of mutually complementary modes of plastic deformation differing in the set of active slip systems is introduced. The combination of the modes of plastic deformation was found, which gives the maximal values of the mechanical characteristics at temperatures 300, 77 and 4.2 K. It was shown that decrease of temperature of the preliminary deformation from 300 down to 77 K leads to improvement of the mechanical characteristics of the Ti Grade 2 samples in the whole studied temperature range.

Keywords: deformation, polycrystalline Ti Grade2, equal angular pressing, quasi-extrusion, rolling, mechanical characteristics

У полікристалічному Ti Grade 2, шляхом комбінації різних методів інтенсивної пластичної деформації при 300 і 77 К, таких як рівноканальне кутове пресування, квазігідроекструзія і прокатка, були отримані вісім різних структурних станів. Для кожного стану виміряні наступні параметри: середній розмір зерен в мікроструктурі, значення мікротвердості (при 300 К) і механічні характеристики в ході одновісного стиску при 300, 77 і 4,2 К. Введено уявлення про взаємно додаткові моди пластичної деформації, що відрізняються набором діючих систем ковзання. Знайдений набір мод пластичної деформації, дія яких призводить до максимально високих значень механічних характеристик при температурах 300, 77 і 4,2 К. Показано, що зниження температури попередньої деформації від 300 до 77 К призводить до поліпшення механічних характеристик зразків Ti Grade 2 у всьому дослідженому температурному інтервалі.

Ключові слова: деформація, полікристалічний Ti Grade 2, рівноканальне кутове пресування, квазіекструзія, прокатка, механічні характеристики.

Introduction

Development of the new methods for production of the high strength states of materials by grain refinement is one of the actual tasks of modern material science. For this purpose the treatment of the various metallic materials is used by means of severe plastic deformation (SPD), which allows produce microstructures with submicron, and in some cases with nanoscaled grain sizes [1-17]. While, using the SPD methods at ambient temperature leads to saturation of the process of grain refinement at strains of $e \sim 8-10$ due to intensification of the dynamic recovery processes at large strains. But influence of the dynamic recovery on the microstructure changes during SPD can be decreased by decrease of the SPD temperature down to cryogenic values, which leads to increase of the yield and ultimate strength of the deformed materials [1, 2]. Additional decrease of average grain sizes and increase of the strength in metallic materials can be achieved by consequent change of

deformation mode during SPD, which leads to activation of the new slip systems. So, in [3] for Al-0.13 wt.%Mg alloy, subjected to ECAP (strain ~ 10) at 298 K, which followed by compression at 77 K, the record small average grain size of ~ 180 nm was achieved.

The present paper is devoted to investigation of the possibilities of grain refinement and corresponding increase of the yield and ultimate strength of the polycrystalline Ti Grade 2 by consequent application of the mutually complementary modes of the severe plastic deformation: the simple shear (during ECAP), the axisymmetric deformation (during quasi hydro extrusion at 300 and 77 K), and also by the pure shear (during rolling at 77 K). The polycrystalline Ti Grade 2 was chosen as investigation material due to its wide applications, and due to different acting slip and twinning systems, which can be activated by different deformation types, used in this work.

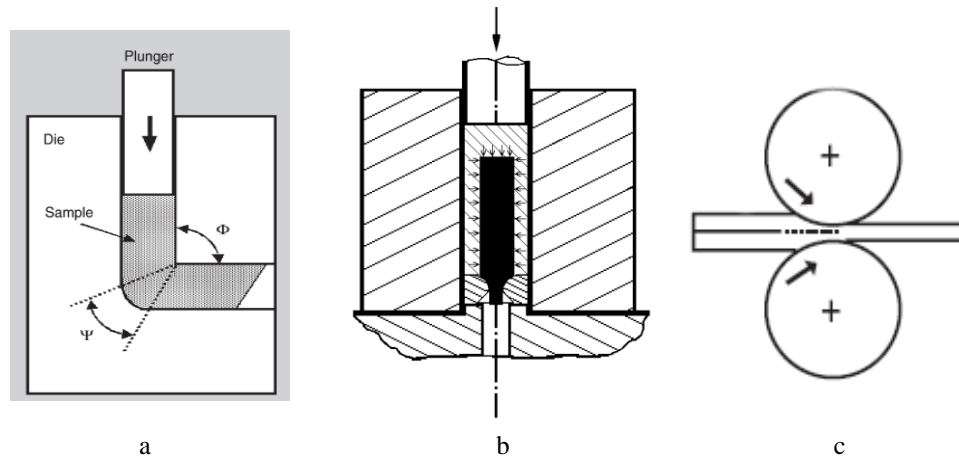


Fig. 1. Types of the severe plastic deformation used in this work: a) Equal channel angular pressing (ECAP); b) Quasihydroextrusion (QHE), working substance – metallic indium; c) Cryorolling (CR) in liquid nitrogen (77 K).

Materials and methods

The polycrystalline rods of Ti Grade 2, produced by hot forging, with average grain size of 20 μm were used as the initial material (**state 1**). Content of main impurities (in wt. %): O – 0.25; N – 0.03; C – 0.08.

From this material by combination of different types of the severe plastic deformation, which are schematically shown in Fig. 1 and described in [1, 2, 12], were produced seven different structural states (states 2-8).

State 2 was produced by 8 ECAP passes at 720 K.

States 3 and 4 were produced by QHE at 300 K by strain $\epsilon \sim 32\%$ (state 3) and $\epsilon \sim 55\%$ (state 4).

States 5 and 6 were produced by QHE at 77 K by strain $\epsilon \sim 38\%$ (state 5) and $\epsilon \sim 52\%$ (state 6).

State 7 was produced by consequent application of ECAP (8 passes at 720 K) and QHE ($\epsilon \sim 57\%$ at 77 K).

State 8 was produced by consequent application of ECAP (8 passes at 720 K) and rolling at 77 K to 50 %.

Vickers microhardness was measured by PMT-3 instrument with load of 100 g and time 10 s. Mechanical tests were carried out in uniaxial compression with initial strain rate $4 \cdot 10^{-4} \text{ s}^{-1}$ at cylindrical samples of 6 mm length

and 3 mm diameter at deformation machine MRK-3 (stiffness $7 \cdot 10^6 \text{ N/m}$) at temperatures 300, 77 and 4.2 K. During compression deformation the yield strength, ultimate strength and corresponding ultimate strain were registered. Measurements of the grain sizes were realized by electron microscope JEM-100CX II.

Experimental results

The average grain sizes and values of yield strength of the Ti Grade 2 samples in uniaxial compression of different structural states at the three temperatures are shown in Table 1. It should be noted that in state 8 after rolling the plate width was 1 mm, which is not sufficient for compression test and it was not carried out for this state.

Analysis of the experimental data listed in Table 1 shows the significant dependence of $\sigma_{0.2}$ and d on different SPD parameters: its type, value and temperature. It is turned out that minimal average grain sizes and maximal values of the yield strength for all studied temperatures were observed for state 7, which was produced by subsequent application of the mutually complementary modes of the severe plastic deformation:

Table 1.

Average grain sizes d and yield strength $\sigma_{0.2}$ in compression of Ti Grade 2 at temperatures 300, 77 and 4.2 K for the different structural states.

Temperature, K	Yield Strength $\sigma_{0.2}$, GPa						
	1. Initial, $d \approx 20 \mu\text{m}$	2. ECAP (8 passes at 720 K) $d \approx 0.4 \mu\text{m}$	3. QHE, (300 K, 32 %) $d \approx 2 \mu\text{m}$	4. QHE, (300 K, 55 %) $d \approx 1.5 \mu\text{m}$	5. QHE, (77 K, 38 %) $d \approx 0.5 \mu\text{m}$	6. QHE, (77 K, 52 %) $d \approx 0.4 \mu\text{m}$	7. ECAP, (8 passes at 720 K + QHE, 77 K, 57 %), $d \approx 0.1 \mu\text{m}$
300	0.42	0.64	0.52	0.60	0.63	0.73	0.86
77	0.67	1.05	0.82	0.89	0.86	1.00	1.22
4.2	0.74	1.22		0.99			1.38

simple shear (ECAP, 8 passes at 720 K) and axisymmetric deformation (carried out by QHE at 77 K to 57 %). For this structural state the typical stress – strain curves in uniaxial compression at temperatures 300, 77 and 4.2 K are shown in Fig. 2.

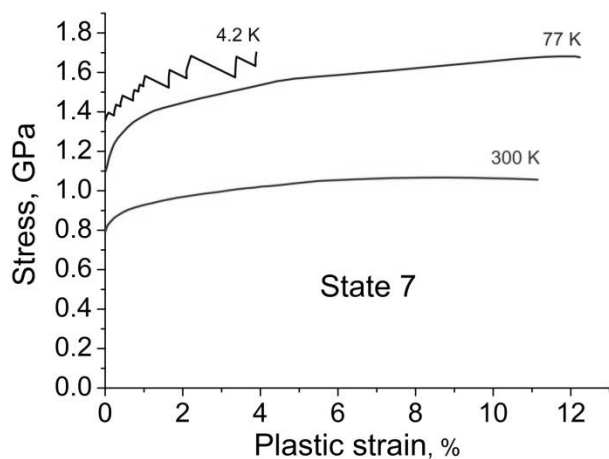


Fig. 2. Typical stress-strain curves in compression of Ti Grade 2 in state 7 for temperatures 300, 77 and 4.2 K

It can be seen from Fig. 2 that the smooth type of plastic deformation is changed by serrated, jump-like deformation at temperature 4.2 K. Such changes of the stress-strain curve type during transition to the liquid helium temperatures are typical [18] for deformation of titanium in this temperature range.

Yield strength values for different structural states of Ti Grade 2 in the forms of diagrams (for temperatures 300, 77 and 4.2 K correspondently) are shown in Fig 3 (a-c).

In Fig. 4 the microhardness values can be seen of Ti Grade 2 for different structural states, measured at 300 K.

It can be seen from Fig. 3 and 4 that values of microhardness and yield strength for the different studied structural states are in good correlation. And the maximal values of microhardness and yield strength at 300 K were observed for the state 7, produced by the consequent application of the mutually complimentary modes of the severe plastic deformation: simple shear (ECAP, 8 passes at 720 K) and axisymmetrical deformation, realized by QHE at 77 K to 57 %.

In Fig. 5 the typical microstructures are shown for the two structural states: state 2 (after ECAP) (a) and state 7 (after ECAP + QHE at 77 K) (b).

As follows from Fig. 5 a, the structure with grain sizes of ~ 0.4 μm is formed in the result of ECAP. Azimuth blurring of reflexes, observed in the diffraction pattern (insert to Fig. 5a), is caused by stresses from defects both inside the grains and at grain boundaries. Typical

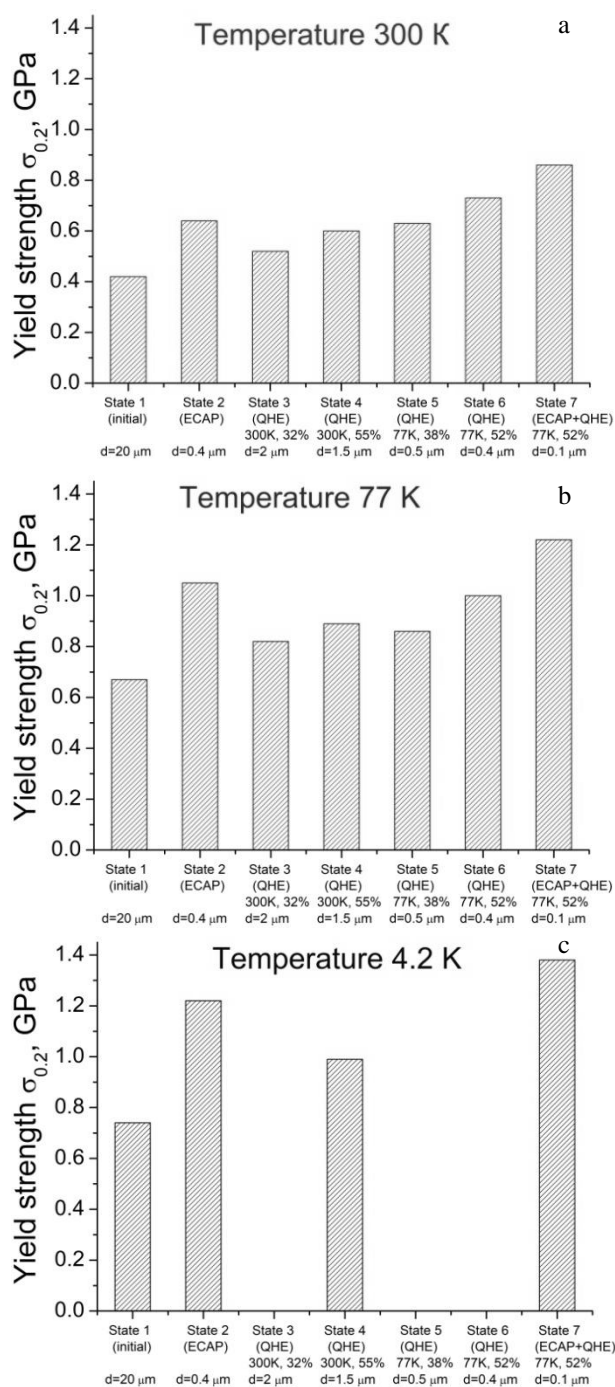


Fig. 3. Yield strength in compression of the Ti Grade 2 in different structural states at temperatures 300 K (a), 77 K (b), 4.2 K (c).

microstructure of the state 7 (ECAP + QHE at 77 K) indicates the significant grain refinement (Fig. 5 b) down to ~ 0.1 μm. Diffraction pattern in this case (insert to Fig 5b) has specific circular dot pattern, typical for the ultrafine grained structure.

Discussion of results

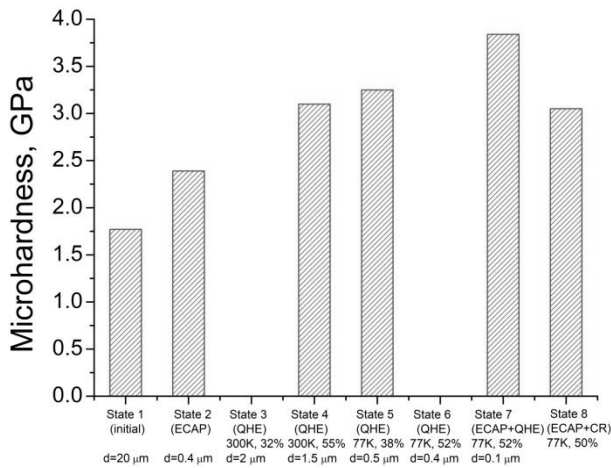


Fig. 4. Vickers microhardness at 300 K of the Ti Grade 2 in different structural states

It is known that the processes of mechanical treatment of metals can be described by combinations of different modes of plastic deformation, such as axisymmetrical deformation, simple and pure shear [1, 10, 19-23] etc. In this work the set of plastic deformation modes was realized by the consequent application of different deformation methods – ECAP, rolling and QHE.

It is reasonable that the consequent application of these deformation methods should provide the activation of maximal quantity of the slip systems [2], i.e. maximal accumulation of deformation in the material, maximal grain refinement and obtaining the high yield strength values. The experimental results of this work confirm these suggestions and indicate the influence of the SPD temperature on the measured parameters. It is seen from the Table 1 that combination of ECAP (simple shear deformation) and QHE (axisymmetrical deformation), i.e. use of mutually complimentary deformation modes of SPD, significantly increase the influence of SPD on the microstructure and mechanical properties. So, in this case (Table 1, state 7) grain size values are decreased in comparison with the initial state from 20 μm down to d = 0.1 μm, and $\sigma_{0.2}$ values at 300 K increased in two times – from 0.42 GPa to 0.86 GPa (Table 1, states 1 and 7).

The consequent action of other deformation types: ECAP + rolling (state 8) (i.e. simple and pure shear) also significantly (almost at ~ 20%) increased the microhardness value (Fig. 4).

The case of QHE can be used to consider the influence of the SPD temperature on the values d and $\sigma_{0.2}$. It is seen (Table 1, states 4 and 6) that decrease of QHE temperature from 300 down to 77 K leads to decrease of d in 4 times (from 2 μm down to 0.5 μm), while the $\sigma_{0.2}$ increases at 20 %.

More strong grain refinement at lower temperature of SPD is realized due to decrease of the dynamic recovery processes, which are caused by thermally activated cross slip or climbing of dislocations. Moreover, as follow from [1], increase of grain boundary mobility with decrease of SPD temperature influences on the grain refinement. Physical mechanism of such increase of the grain boundary mobility during SPD at cryogenic temperatures is not clear yet [1]. The authors of [1] suggested, based on the results of molecular dynamic simulation [24, 25] that at cryogenic temperature at conditions of active loading, the structure of the boundaries is heavily disordered. In the result, the grain boundaries are in nonequilibrium state, which can lead to increase of the diffusion coefficients and to accelerated grain boundary migration [25]. However, this suggestion needs additional confirmation, as the question about the activation energy, required for grain boundary diffusion at cryogenic temperatures [1] is not clarified yet.

One of the possible hypothesis, explaining the origin of the high mobility of grain boundaries at cryogenic temperatures, consists in counting of difference in coefficients of the thermal expansion of the grain boundary area and the grain interior: coefficient of the thermal expansion of the grain boundary area in several

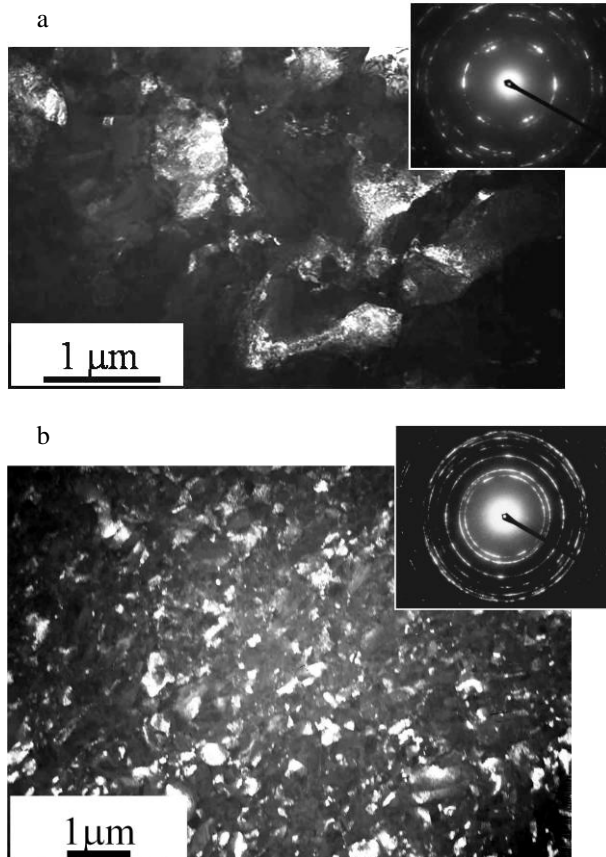


Fig. 5. Typical dark field transmission electron microscopy images and correspondent diffraction patterns of the Ti Grade 2 structure: state 2 (after ECAP) (a); state 7 (ECAP + QHE at 77 K) (b).

times larger in comparison with the coefficient of the grain interior [25, 26]. According to the Gruneisen relation [27], it leads to the increased isothermal compressibility of the grain boundary area, which was experimentally found for nanocrystalline iron [28] and nanocrystalline nickel [29]. Difference in the coefficients of thermal expansion of the grain boundary area and of the grain interior during the cooling below 300 K, should lead to the uniform compression of the grain boundary area and to increase of atomic volume in the grain boundary area [29]. This effect simplifies the diffusional mobility of atoms and decreases the resistance to the grain boundary sliding at cryogenic temperatures, which can explain the high mobility of the grain boundaries in these conditions. It is obvious, that these peculiarities can amplify the processes of grain refinement at cryogenic temperatures.

Conclusions

1. The concept is suggested of influence of the mutually complementary modes of plastic deformation, which are characterized by different sets of acting slip systems, on the microstructure and mechanical properties of Ti Grade 2.

2. The combination of the SPD methods is found, which leads the polycrystalline Ti Grade 2 to the nanostructured state with the minimal average grain size and with the maximal values of the yield strength. So, by consecutive use of simple shear (during ECAP) and axisymmetrical deformation (during QHE at 77 K) the decrease of average grain sizes is registered from 20 μm in initial state down to 0.1 μm , which accompanied by increase in two times of the yield strength values (from 0.42 GPa to 0.86 GPa).

3. Use of the cryodeformation during QHE allows to slow down the dynamic recovery. In the result, after QHE at 77 K, the resulted average grain size (0.5 μm) is in 4 times smaller, than after QHE at 300 K (2 μm), and the yield strength after QHE at 77 K increases at 20 %.

4. The explanation is suggested of the mobility increase of grain boundaries at cryogenic temperatures, which simplify of process of grain refinement.

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