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LOW TEMPERATURE FEATURES OF MECHANICAL PROPERTIES AND FAILURE OF DIFFERENT COMMERCIAL PURITY NANOSTRUCTURED TITANIUM PROCESSED BY ECA PRESSING

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Abstract. Low temperature mechanical characteristics of different purity nanostructured titanium produced by the equal channel angular pressing (ECAP) method have been studied under active deformation (quasistatic uniaxial tension and compression) and under failure. Values of the yield stress and the uniform plasticity at 300, 77, and 4.2K for nanostructured (NS) and polycrystalline coarse-grained (CG) titanium of different commercial purity have been compared. The failure type of Grade 2 Ti under tension and compression has been established.

1. INTRODUCTION

Successful technical use of titanium and its alloys as structural materials is conditioned by their high specific strength and high plasticity, which are not decreased at cryogenic temperatures due to the specific character of titanium deformation mechanism. Using of an intensive plastic deformation by the equal channel angular pressing allows decreasing the average grain size *d* from 10-20 μm down to *d* of 0.1-0.8 μm [1]. Such decreasing of a grain size leads to essential increasing of titanium mechanical characteristics in a nanostructured state comparatively with a polycrystalline state, with conserving a large enough ductility [2]. In this work, influence of NS titanium purity and type of deformation (quasistatic uniaxial tension and compression) on the temperature dependences of the yield stress σ_0, the ultimate uniform plasticity ε_{unif}, and failure have been experimentally studied at temperatures 300, 77, and 4.2K.

2. MATERIALS AND METHODS

Initial materials for investigations were polycrystalline titanium of different commercial purities: Grade 2, with 0.16% weight content of O_2, and Grade 4, with 0.34% weight content of O_2, - processed by hot forging.

The Grade 2 titanium was studied in the two structural states:

- Initial state 1 – polycrystalline titanium with the average grain size *d* of 20 μm;
- State 2 – with the average grain size *d*=0.8 μm along ECAP direction and *d*=0.39 μm across ECAP direction, processed by intensive plastic deformation of initial titanium by 8 runs of ECAP at 450 °C.

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The Grade 4 titanium also was studied in the two structural states:

- Initial state 1 – polycrystalline titanium with the average grain size *d* of 15 μm;
- State 2 – with the average grain size *d*=0.85 μm along ECAP direction and *d*=0.35 μm across ECAP direction, processed by intensive plastic deformation of initial titanium by 8 runs of ECAP at 450 °C.

Mechanical characteristics were investigated under uniaxial tension and compression with the $4{\cdot}10^{-4}\,s^{-1}$ rate of a relative deformation (quasistatic straining), with a stiff straining machine at 300K, 77K (in liquid nitrogen) and 4.2K (in liquid helium 4). The micro specimens for tension had the 6 mm gage length and a square cross section of 0.7×1.5mm. Specimens for compression were rectangular prisms 2×2×6 mm. Specimens were cut from rods after ECAP along (||) and across (⊥) ECAP axis.

Macroscopic yield stress σ_0 has been determined by the method of tangent intersection on the initial and stationary parts of the σ-ε curve, where σ is a stress acting on the specimen and ε is a plastic deformation. The ultimate uniform plasticity ε_{unif} value has been determined as difference between the deformation of the neck formation beginning and the deformation corresponding to the yield stress on the σ-ε curve under tensile deformation.

The type of fracture was specified, and the fracture surface morphology was studied with the TESLA BS-300 scanning electron microscope (SEM).

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1. Influence of the average grain size *d* on the yield stress σ_0 temperature dependence

The temperature dependences of the yield stress σ_0 in state 1 and 2 are shown in Fig. 1 (a-d) for Grade 2 and Grade 4 specimens at 300, 77 and 4.2K under uniaxial tension and compression, along and across the ECAP direction.

Decreasing of the grain sizes after ECAP from *d*=20 μm down to *d*=0.3 μm leads to 1.5–2 times increment of the yield stress σ_0 for Grade 2 Ti. The increment of the impurity content in Grade 4 Ti (increasing of oxygen content from 0.16% weight to 0.34% weight) leads to the σ_0 increment in comparison with Grade 2, while decreasing the σ_0 (*d*) dependence. For Grade 4 specimens, decreasing of d from 20 μm down to *d*=0.3 μm leads only to 1.1–1.5 times increment of the σ_0. Increasing of the σ_0 under changing from state 1 to state 2 depends not only on the impurity content, but also on other concerned parameters: the orientation of the specimens relative to ECAP axis, type of the deformation (tension-compression), and the temperature of the experiment (Fig. 1 (a-d)).

As one can see from Fig. 1, the temperature dependences of the σ_0 in the temperature range 300–4.2K for Gr. 2 and Gr. 4 Ti in the nanostructured state 2 are similar to the dependences for coarse-grained Ti Gr. 2 and Gr. 4 in state 1. The exception is the σ_0 (T) under compression of NS specimens Grade 4 (⊥) in the temperature range 77–4.2K (Fig. 1d), when $\partial\sigma_0/\partial T$ for NS state 2 is much larger than $\partial\sigma_0/\partial T$ for state 1.

The identity of the σ_0 temperature dependences in states 1 and 2 in the majority of the investigated cases for Ti Gr. 2 and Gr. 4 gives us the opportunity to consider that the same mechanism controls the kinetics of plastic deformation in this temperature range. It was previously established [3] that thermally activated overcoming of impurity atoms by dislocations is the dominant mechanism in the polycrystalline Ti (for Ti with O_2 atomic content 0.06–3%) in the range of *T* from 300 down to 4.2K. The polycrystalline Ti has additional barriers below the room temperature due to dislocations of other glide systems and twinning [3].

The activation volume *V* values [4] of the dislocation glide and the deformation dependence of V for NS Ti VT 1-0, produced by ECAP, also confirm that thermally activated overcoming of short-range barriers by intra-grain dislocations is the controlling process for the NS Ti plastic deformation, as well as for the polycrystalline Ti, in spite of the small grain size (*d*~0.3 μm) of the NS Ti.

Anomalous high yield stress values under compression of NS Gr. 4 (⊥) specimens, shown in Fig. 1d, can indicate that in this case the processes, controlling the rate of plastic deformation, are nucleation and annihilation of dislocations at the grain boundaries rather then thermally activated motion of intra-grain dislocations. Obviously, in the NS Ti, under subsequent decrease of the average grain size down to *d*<0.1 μm the nucleation and the annihilation of dislocations (especially at low temperatures) should control the process of plastic deformation. In these cases the anomalous high yield stress and the cryogenic strength can be expected as it took place in ECAP Ti VT 1-0 with grain size *d*~0.1 μm under cooling below 77K [1]. Apparently, the ex-

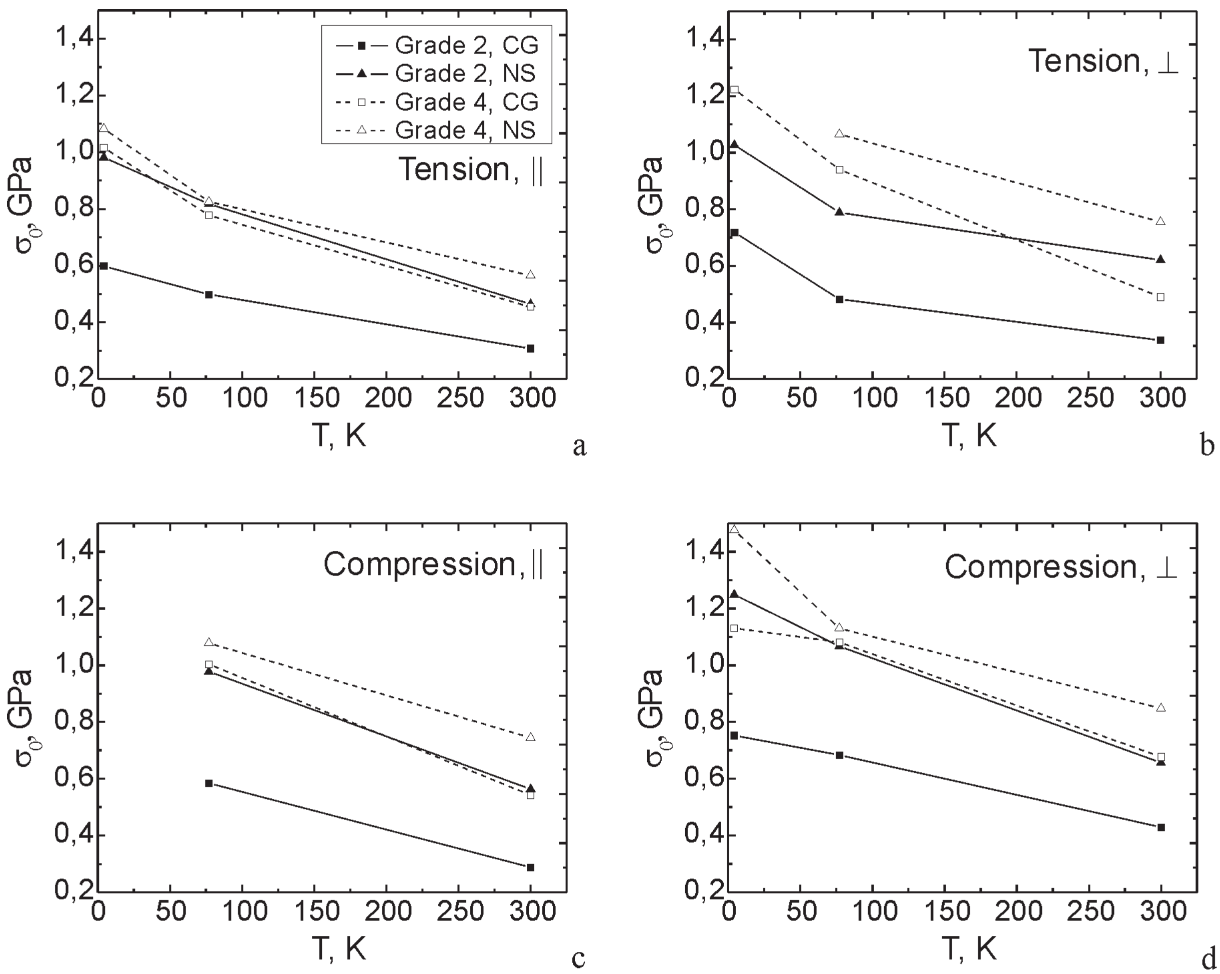

Fig. 1. Temperature dependences of the yield stress for Grade 2 and Grade 4 Ti, along (||) and across (⊥) ECAP axis in the coarse-grained (CG) state 1 and the nanostructured (NS) state 2.

traordinary increase of σ_0 in this case is due to changing of the deformation mechanism. And the acting mechanism is not so much inter-grain glide below 77K (as it was assumed in [2]) as nucleation and annihilation of dislocations at grain boundaries. Similar result was obtained recently in NS Ni and Co with the d<0.1 μm grain size [5].

3.2. Discrepancy of the σ_0 under compression and tension

The plastic deformation asymmetry, which is well known in literature as S-D effect [6], consists in a discrepancy of the yield stress values under uniaxial compression (σ_{0C}) and tension (σ_{0T}). Presence of S-D effect was related in literature to the crystal volume increase of in the course of plastic deformation [6]. The typical value of S-D effect for polycrystalline materials is several tens of percents on average [6]. This effect should be weakened in ultra fine-grained metallic materials due to a larger contribution to deformation of the grain-boundary dislocations (GD), in comparison with polycrystalline materials. The contribution of GD to the bulk effect in the course of plastic deformation is smaller (comparing to lattice dislocations) due to the small vector Burgers value.

The values of S-D effect can be characterized by the relation

$$\Delta = \frac{(\sigma_{0C} - \sigma_{0T})}{0.5(\sigma_{0C} + \sigma_{0T})}, \qquad (1)$$

and Δ has maximal values for Grade 2 and Grade 4 specimens across ECAP axis at 300K. In the state 1, Δ=24% for Grade 2 and 32% for Grade 4. The Δ

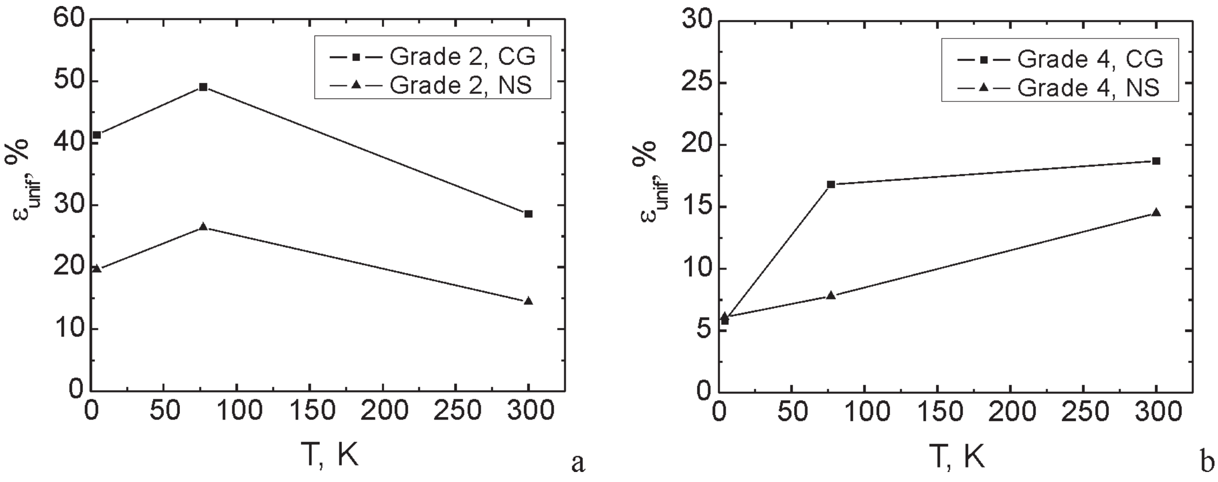

Fig. 2. Temperature dependences of the plasticity ε_{unif} for Grade 2 and Grade 4 Ti (||) in the coarse-grained (CG) state 1 and the nanostructured (NS) state 2.

value in the state 2 is several times smaller (Δ=5% for Grade 2 and Δ=11% for Grade 4). This result can be an indirect evidence of the noticeable contribution of GD in specimens' deformation both for Grade 2 and Grade 4 titanium. This conclusion is in the accord with previous data on S-D effect for Ti VT1-0 [7] in the states 1 and 2.

3.3. Temperature dependence of the plasticity for the nanostructured Ti

Fig. 2 shows temperature dependences of the plasticity ε_{unif} for titanium of different purity in the nanostructured and initial states. It is seen that for the Grade 2 Ti (Fig. 2a), ε_{unif} increases under cooling from 300 to 4.2K with the maximum at 77K. In state 2 ε_{unif} increases from 14 to 19% under cooling from 300 to 4.2K (with the 26% maximum near 77K). Values ε_{unif} for state 1 are nearly two times larger than for state 2. For Grade 4 Ti in state 2, ε_{unif} decreases monotonously from 14% at 300K down to 6% at 4.2K (Fig. 2b). In state 1, the ε_{unif} value decreases from 19% to 17%. ε_{unif} for the Grade 4 state 2 at 4.2 K is practically the same as for state 1.

Presented data show that the plasticity of the nanostructured and polycrystalline titanium essentially depends on the oxygen content in Ti. Increasing of the oxygen concentration in the nanostructured titanium decreases the ε_{unif} values and drastically changes its temperature dependence. This can be caused by a damping influence of the impurity on the probability of double cross-slip and on the twinning process in the titanium.

Increased plasticity under uniaxial tension of Grade 2 Ti (in states 1 and 2) and Grade 4 (in the state 1) at 77K was explained in [8] by the stimulating influence of the thermal anisotropy internal stresses upon the mobile dislocation multiplication by the double cross-slip mechanism in the polycrystalline state (for large grains with d=15 μm), and by the heterogeneous nucleation of mobile dislocations and mechanical twins at the grain boundaries (for small grains with d=0.35 μm).

3.4. Features of the low-temperature failure of NS Grade 2 Ti

Fractographic analysis of shear failure surfaces of nanostructured specimens under compression reveals following regularities: presence of unstable ductile sliding-off shear of one part of the specimen relative to another, presence of specific 'vein' pattern on the shear failure surface, observed by means of SEM. Similar regularities had been observed in VT1-0 Ti and were described earlier [9].

Under tensile deformation, the type of failure depends on the temperature (Fig. 3). The shear failure along the plain, oriented at the angle of 45° to tension axis, has been observed at the macro scale at 77 and 4.2K (Fig. 3 a-d). Typical 'vein' patterns have been observed by SEM observations of the shear

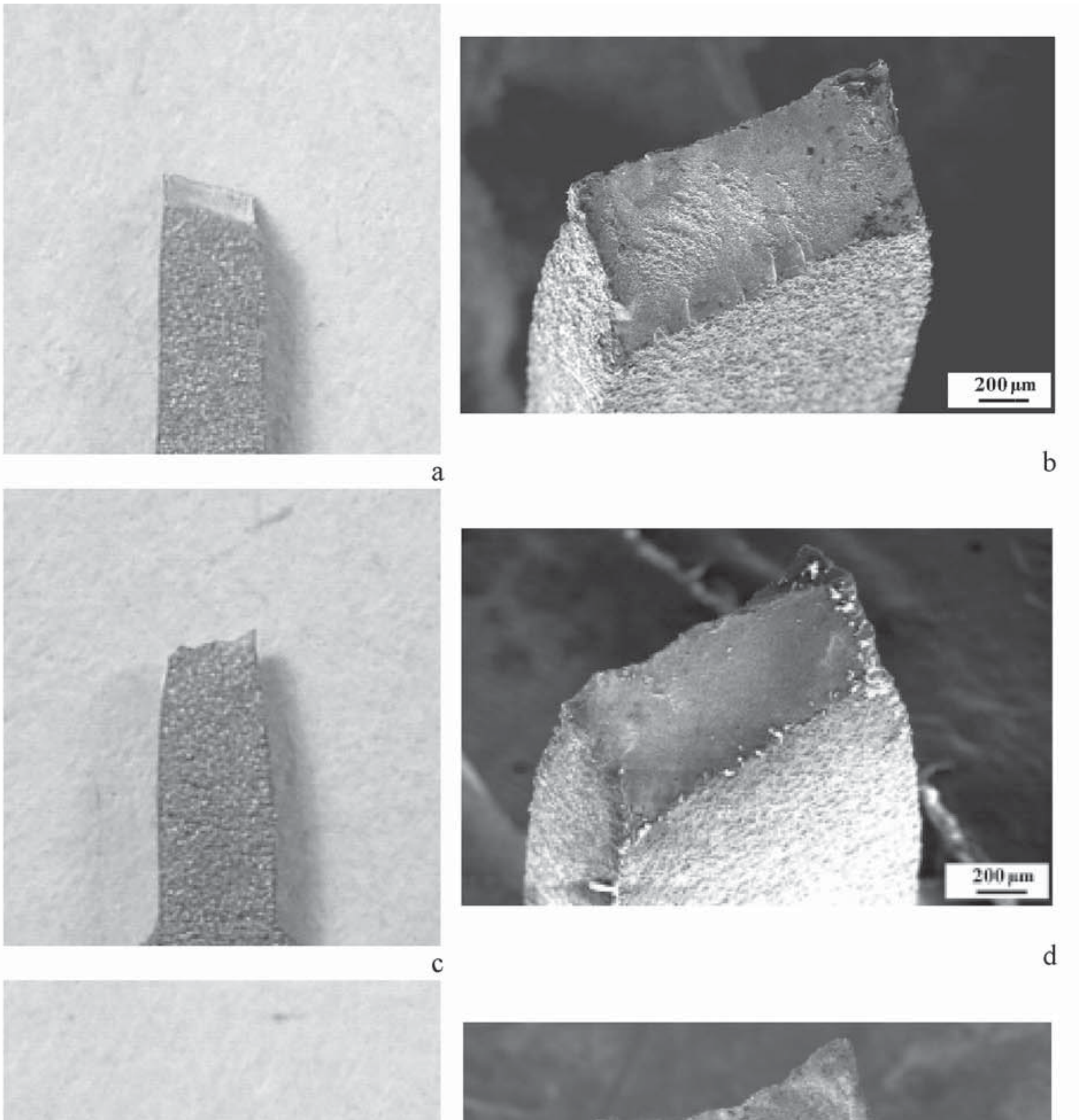

Fig. 3. Macroview of the failure regions (a, c, e) and the macrofailure surfaces (b, d, f) of the nanostructured Grade 2 Ti specimens (tension) at 4.2K (a, b), 77K (c, d), and 300K (e, f).

failure surfaces (similar to the case of the failure under compression).

At 300K the failure surface has two regions (Fig. 3 e-f): the normal rupture at the angle of 90° to the tension axis and the region of a shear failure (the 45° angle to the tension axis). 'Vein' patterns have been observed on the shear failure surfaces by SEM, and 'chevrons' (cup and cone failure) on the normal rupture surfaces. Thus, these data on the failure surface morphology are in agreement with observed increasing of the titanium macroscopic plasticity under cooling to the liquid nitrogen temperature.

4. CONCLUSIONS

The yield stress increase in Grade 4 Ti in comparison with Grade 2 Ti has been detected under the impurity content increase (O_2 content, from 0.16% weight to 0.34% weight) in the temperature range 300–4.2K.

Decreasing of the average grain size from *d*=20 μm down to *d*=0.3 μm after ECAP leads to increasing of the σ_0, which depends on the purity of titanium. For Grade 2 the σ_0 value in the nanostructured state is 1.5–2 times larger than the polycrystalline titanium σ_0. For the Grade 4 titanium this increment is much smaller (σ_{NS}/σ_{CG}=1.1–1.5).

Plastic deformation in the NS titanium at cryogenic temperatures has a thermally activated character, as well as in the CG titanium.

Observed differences in the temperature sensitivity of the σ_0 (in the 77–4.2K temperature range) for Ti in the nanostructured and polycrystalline states lead to the conclusion about different contributions of intra-grain, inter-grain glide, and twinning to the deformation of these structural states of Ti at the cryogenic temperatures.

It has been established that the value of plasticity and its temperature dependence in the nanostructured titanium depend essentially on the technical purity of titanium, and absolute values ε_{unif} are smaller in comparison with the corresponding polycrystalline analogues.

The value of the yield stress under compression of Ti specimens (across the ECAP axis) has been established to exceed the σ_0 values under tension (S-D effect). S-D effect value for the nanostructured Ti is smaller than for the coarse-grained polycrystalline Ti.

The increased plasticity under the uniaxial tension of Grade 2 Ti (in states 1 and 2) and Grade 4 Ti (in the state 1) at 77K is explained by a stimulating influence of the thermal anisotropy internal stresses upon the mobile dislocation multiplication by the double cross-slip mechanism in the polycrystalline state and by the heterogeneous nucleation of mobile dislocations and mechanical twins at the grain boundaries in the nanostructured state.

At 77 and 4.2K under tensile and compressive deformation of Grade 2 Ti specimens, the shear failure has been observed along the plain, oriented at the angle of 45° to tension axis. Regions of the normal rapture and the shear failure have been observed under tension of Grade 2 Ti at 300K.

A ratio of the yield stress and the plasticity resource values in nanostructured and polycrystalline states makes it possible to consider NS Ti as a perspective structural material for cryogenic applications.

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