Equal channel angular pressing at temperatures of 77-575 K of Titanium Grade 2: Microstructure and mechanical properties

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Abstract. Cylindrical samples of CP Titanium (Grade 2) were deformed by one, two and three passes of equal channel angular pressing (ECAP) each at temperatures 77, 300 and 575K, respectively. The microstructure of samples processed at 77K shows retardation of recrystallisation, high density of dislocations and deformation twins, diffuse and obscure grain boundaries compare to microstructure of samples processed at room and high temperature, where recrystallised ultrafine equiaxed grains are observed.

Mechanical properties for all structural states of Ti were studied by microhardness measurements at 300 K, and uniaxial compression at temperatures 300, 170, 77 and 4.2 K. Higher levels of ECAP deformation (more passes of ECAP) lead to higher values of strength and hardness at all studied temperatures. Decrease of ECAP temperature leads to increase of strength characteristics in all cases. Influence of ECAP and compression temperatures on possible changes of deformation mechanism is discussed.

Keywords: titanium, cryogenic equal channel angular pressing, cryogenic properties, mechanical properties.

1. Introduction

Severe plastic deformation (SPD) of different metallic materials at low temperatures has been performed by different methods such as cryogenic rolling [1,2], multi-axial forging [3], high pressure torsion [4,5], ECAP [6,7], drawing [8] and others. The use of cryogenic temperatures for SPD processing is justified by suppression of dynamic recovery and enhancement of grain refinement in comparison to room temperature deformation. It is suggested that finer grains below 0.2 μm could be obtained if SPD processing performed at low temperature and, therefore, mechanical properties such as strength and ductility could be significantly enhanced [4].

In this work a comparative study of microstructure formation in CP Titanium (Grade 2) deformed by one, two and three passes of equal channel angular pressing (ECAP) at temperatures 77, 300 and 575K has been performed. The microstructure characterization was followed by comprehensive study of mechanical properties at the range of temperatures from room temperature to 4.2 K.

2. Material and Experimental Procedures

Commercially-pure titanium, CP-Ti Grade 2 (0.16 wt.% of O, which correspond to 0.48 at.%) in the form of extruded rod of 10 mm diameter was used in this work. Samples of 35 mm length were cut from the rod and dipped in to liquid nitrogen. Then the samples were dropped in the entry channel of ECAP rig and immediately pressed. The time between extracting the sample from liquid nitrogen and the end of pressing did not exceed 10 sec. Samples were subjected to one, two and three ECAP passes with cooling in the liquid nitrogen before each pass. The ECAP rig used for processing is described elsewhere [9,10].

The microstructures of the Ti samples ECAP-processed at different conditions were examined with conventional and high-resolution (HR) transmission electron microscopy.
Thin foil samples for TEM studies were prepared with a Struers Tenupol-5 twin-jet electropolisher using a solution containing 5% perchloric acid, 35% butoxyethanol and 60% methanol by volume. The applied voltage and temperature of the solution were maintained at 60V and -35°C, respectively, during the electropolishing process.

General characterisations of the deformed Ti matrices were carried out with a FEI Tecnai G2 T20 microscope operating at 200kV. Selected area electron diffraction (SAED) pattern of samples were obtained using an aperture size of approximately 10 µm on image. A JOEL 2100F FEG microscope operating at 200kV was employed to record HRTEM images of the defect structures within the grains.

Electron backscatter diffraction (EBSD) analysis was carried out on electropolished samples. Crystallographic data were analysed with a FEI Quanta 3D FEG scanning electron microscope (SEM) incorporating a TSL OIM™ system.

For the measurement the Vickers microhardness, samples were prepared by spark erosion, grinding and polishing from transversal (perpendicular to the extrusion direction) sections, the applied load was 2.0 N for 10 s, and the indentations were applied in intervals of 0.2 mm along 4 different radial directions from the centre to the edge of sample.

Compression samples of square 1.8 × 1.8 mm² cross-section and height of 3.5 mm were cut from ECAP billets and also from initial coarse grained (CG) Ti rod by spark erosion with the height parallel to extrusion direction. The mechanical characteristics of ECAP processed as well as of coarse grained Ti samples were studied by recording the load-displacement curves during compression with a strain rate 3·10⁻⁴ s⁻¹ in an MRK-3 deformation machine at ambient temperature of 300 K and cryogenic temperatures achieved with liquid nitrogen and helium. The load-displacement data were converted into engineering stress-strain curves σ(ε) relevant to initial dimensions of the sample.

During the measurement of the deformation curves in some samples the shear flow stress sensitivity Δτ/Δlnεa was measured by increasing the rate of deformation from \( \dot{\varepsilon} = 3 \cdot 10^{-4} \text{s}^{-1} \) by a factor of 6.6. The shear stress \( \tau \) was expressed as \( \tau = 0.5 \sigma \) [11]. For construction the temperature dependence the \( \Delta \tau / \Delta \ln \dot{\varepsilon} \) values were used at \( \varepsilon = 2 \% \).

According to the standard formula [12] the values of activation volume for the process of plastic deformation were calculated from the rate sensitivity of flow stress

\[
V(T) = kT \frac{\Delta \ln \dot{\varepsilon}}{\Delta \tau(T)}
\]

where \( k \) is the Boltzmann constant.

3. Experimental Results

3.1. Microstructure Characterisation

The bright-field (BF) electron images and the corresponding SAED patterns of the CP Ti samples ECAP-processed under different temperatures, \( T \), and number of passes, \( N \), are summarised in Fig. 1.
Figure 1. Representative microstructures of the CP Ti Samples processed under (a),(b),(c) 1 pass, (d),(e),(f) 2 passes and (g),(h),(i) 3 passes of ECAP at temperature of (a),(d),(g) 77 K, (b),(e),(h) 300 K and (c),(f),(i) 575 K. Inset in each figure represents the corresponding electron diffraction patterns.

Comparing the microstructures processed at the cryogenic temperature, i.e. 77 K, there exists an obvious trend that the density of strain contours (dark bands) in matrix grains increases significantly with the number of passes, see Fig. 1 (a), (d) and (g) for $N = 1, 2, \text{and} 3$, respectively. The strain contours are diffraction contrasts caused by the rotation of the lattice planes and thus their density reflects the magnitude of the residual stress fields. Detailed microscopic examinations of the specimen processed at $T = 77$ K for $N = 1$ revealed a highly dislocated matrix but no apparent dislocation cell structures could be observed therein. Well-defined cell structures or sub-grains only became visible in specimens processed with $N = 2$ or above. The development of highly misoriented cells or subgrains in the specimen...
processed at $T = 77$ K for $N = 3$ is evidenced by the occurrence of continuous diffraction rings (or ring segments) in its corresponding SAED pattern, Fig. 1 (g). The presence of zone-axis type reflection maxima in the SAED pattern suggests that a substantial portion of the parent grain remains unrefined even after 3 ECAP passes at the cryogenic temperature.

An increase of the ECAP temperature to 300 K led to considerable formation of low-angle grain boundaries in the sample processed with one pass ($N = 1$), which are inferred by the streaked diffraction spots in Fig 1 (b). This plausibly suggests that sub-grains having low misorientations were developed in the matrix. An increase in number of ECAP passes to $N = 2$ at the same temperature resulted in an extended formation of subgrains within the primary grain. These sub-grains, arranged preferentially in cluster forms, are manifested by the regions of mottled contrast having a sub-micron size-scale in the BF image, Fig. 1 (e). Further ECAP deformation by three passes resulted in a relatively more homogeneous microstructure consisting of very fine subgrains having low to moderate degrees of misorientation with one another, Fig. 1 (h).

ECAP processing at the elevated temperature of 575 K gave arise to a considerable change in microstructures compared with the lower temperature counterparts. Fig. 1 (c) indicates that an abundance of high-angle grain boundaries have emerged in the matrix upon a single ECAP pass ($N = 1$), most of which were found to be associated with lamella grains having a longer dimension of up to several µm (e.g. the grains marked by arrows). Such an evolution in microstructure is attributable to recrystallization process which occurs more readily at elevated temperatures. It is noted that further increase in $N$ caused the lamella grains to break down into smaller, more equiaxed ultrafine grains, see Fig 1 (f) and (i). The result of dark-field grain size analysis (> 300 grains) indicated that the sample processed at $T = 575$ K for $N = 3$ contains a distribution of ultrafine grains having an average size of around 219 nm. Such a value of an average grain size is typical for Ti after ECAP at room and higher temperatures [13,14].

The essential difference in the dislocation substructure and cells formation at cryogenic ($T = 77$ K) and room temperature ($T = 300$ K) was confirmed by TEM study of the microstructures at high magnification after two ECAP passes, Fig. 2. The microstructure after ECAP processing at 300 K revealed the formation of dislocation cells characterised by a non-homogeneous size distribution ranging from ~ 10 to 80 nm. In particular, the cells walls (~10 nm in thickness) tend to exhibit a complex diffraction contrast due presumably to concentration of entangled dislocations, Fig 2 (a). Conversely, a more homogenous cell size of ~ 50 nm in diameter was observed after the same number of ECAP passes ($N = 2$) at 77 K, Fig. 2 (b).

Figure 2. Typical defect structures inside the coarse grains of the Ti samples processed with room temperature and cryogenic ECAP: BF-TEM micrographs showing nano-scale cell structures developed after 2 ECAP-passes at 300 K (a) and 77 K (b), respectively.
Upon further ECAP processing to $N = 3$ at 77 K, some extremely fine crystallites (<100 nm) were noted to form in the matrix of the sample processed at 77 K. The boundaries associated with these crystallites are mostly of high misorientations. However, they are not well-defined in profile but rather obscure and serrated, see Fig. 3 (a). It remains questionable whether these boundaries would possess distinctly different properties from the equilibrium grain boundary counterparts. By comparison, Fig. 3 (b) shows the ultrafine grains that are typical of the sample processed at $T = 575$ K for $N = 3$. The interior of the grains are mainly free of dislocations due presumably to dynamic recovery. Although cell structures still exist in this sample, they were observed only in those heavily-deformed matrix grains that were not fully recrystallised. It is believed that the contrasting differences in the nano-scale structures resulted from these two temperatures (77 K vs 575 K) would lead to dissimilar mechanical properties of their bulk materials.

Figure 3. Ultrafine microstructures in the Ti samples processed with ECAP at temperatures of (a) 77 K and (b) 575 K, for 3 passes.

In contrast to its profound effect on the size-scale of the Ti grains, EBSD analyses reveal that ECAP temperature has a relatively small influence on the texture development of the Ti matrices even when the number of ECAP passes accumulates. Fig. 4 (a) and (b) compare the IPF maps and the corresponding pole figures of the Ti samples processed with one ECAP pass at 77 K and 575 K, respectively. Due to the circular geometry of the 3 mm disc samples used in this EBSD study, reference directions are not defined on the pole figures.
Figure 4. IPF maps and pole figures of the ECAP-processed Ti samples: (a) 1 pass at 77 K, (b) 1 pass at 575 K, (c) 3 passes at 77 K and (d) 3 passes at 575 K.

It can be seen from both sets of {0001} pole figures that poles of high intensities (Max = 10.22 for 77 K and Max = 12.22 for 575 K) are concentrated in discreet zones which correspond to a tilting angle ranging from 60 to 90 degrees with respect to the normal direction. Such a crystallographic texture is likely to be inherited from the prior texture of the raw α-Ti extruded rods, which commonly exhibit the so-called "cylindrical texture" where c-axes tend to align perpendicular to the extrusion axis. A minor but noticeable difference between these two set of texture plots is that the sample processed at 575 K shows less intense \(\langle 0\overline{1}0\rangle\) and \(\langle 1\overline{2}0\rangle\) poles aligned with normal direction than does the 77K counterpart.

As the number of ECAP passes increases, the texture analysis is complicated by the severe deformation of the cryogenically processed samples. Fig. 4 (c) shows the IPF map of the sample ECAP-processed at 77 K for 3 passes. A significant portion of the matrix was not readily indexed due plausibly to the presence of an extremely high density of dislocations. The pole figures reflect the textures of the remnant grains, which may not be representative of the entire matrix. On the other hand, Fig. 4 (d) clearly shows that the sample ECAP-processed at 575 K for 3 passes underwent extensive dynamic recrystallisation, resulting in the formation of sub-micron grains having a size of the order of 100 nm, in accord with the TEM observations. Albeit the high degree of recrystallisation, it is apparent that the texture of the recrystallised Ti matrix remains similar to those of the sample processed with 1 ECAP pass, cf. Fig. 4 (a) and (b), albeit that the intensity of the poles are substantially lower (Max = 4.78).
3.2. Mechanical Properties

Typical strain curves obtained in uniaxial compression at different temperatures of initial coarse grained and ultrafine grained Ti, which was processed by 3 passes of ECAP at temperatures 77, 300, 575 K, are shown in Fig. 5. These figures show clearly the contrast between smooth plastic flow at all moderately low temperatures and serrated plastic flow at temperature of 4.2 K. This serrated flow is typical at such low temperature of many metals and alloys including coarse grained Ti [15,16], and can be explained by localization of plastic deformation and collective and interrelated avalanche type motion of carriers of plasticity. The amplitude of serrations is considerably larger in ultrafine grained Ti in comparison with coarse grained structural state indicating larger concentration of barriers for carriers of plasticity in the ultrafine grained states.

![Figure 5. Typical stress-strain curves at different compression temperatures for initial coarse grained and ultrafine grained (3 passes of ECAP at temperatures 77, 300, 575 K) Ti.](image)

The strain curves (Fig. 5) exhibit two different stages: initial “parabolic” stage is changed at plastic strain values of 0.2-3% by extended nearly linear region. Increase of the deforming stress at the “parabolic” stage corresponds to increase of quantity of grains involved in process of plastic deformation, and transition to the linear stage corresponds to spreading of the plastic deformation over all grains and beginning of stable plastic flow [17,18]. Transition to the linear stage takes place at lower values of plastic strain in coarse grained Ti (at about 0.5%) while in ultrafine grained Ti at about 3%. That indicates more wide grain size distribution in ultrafine grained Ti and presence of nanograins with the size of below 100 nm, which require especially high stresses for plastic deformation.
Considerable plasticity is registered for all studied structural states of Ti (more than 15% at compression temperature of 4.2 K and more than 25% at higher temperatures).

Severe plastic deformation by ECAP leads to significant decrease of grain size in comparison with initial coarse grained state and correspondent increase of the material strength, clearly observed in Fig. 5. This effect seems even more pronounced in Fig. 6, where values of the yield stress are plotted for studied structural states and compression temperatures.

![Graphs showing yield stress at different compression temperatures and pass numbers.](image)

**Figure 6.** Yield stress in compression at temperatures 4.2, 77, 170, 300 K for coarse grained and ultrafine grained Ti, processed by 2 and 3 passes of ECAP at 77, 300, 575 K.

It can be seen from Fig. 6 that higher level of strain (more passes of ECAP) lead to higher values of strength at all studied temperatures. Decrease of ECAP temperature leads to increase of strength characteristics in all cases: yield stress after ECAP at 300 K is approximately 10-15% higher than after ECAP at 575 K for all studied compression temperatures. ECAP at 77 K gives additional 5-10% increase of strength relatively to ECAP at 300 K. Similar tendency is proven by microhardness measurements for all studied structural states of Ti (Table 1).
Table 1. Microhardness of initial coarse grained Ti and ultrafine grained Ti (after 1, 2, 3 passes of ECAP at temperatures of 575, 300, 77 K).

| State             | Microhardness, GPa |
|-------------------|-------------------|
| Initial coarse grained | 2.08              |
| ECAP at 575 K, 1 pass | 2.44              |
| ECAP at 300 K, 1 pass | 2.67              |
| ECAP at 77 K, 1 pass | 2.77              |
| ECAP at 575 K, 2 passes  | 2.39              |
| ECAP at 300 K, 2 passes  | 2.73              |
| ECAP at 77 K, 2 passes  | 2.69              |
| ECAP at 575 K, 3 passes  | 2.39              |
| ECAP at 300 K, 3 passes  | 2.92              |
| ECAP at 77 K, 3 passes  | 3.02              |

The yield stress is increased for all studied structural states by about 70-110% when temperature of compression decreased from room temperature to 4.2 K (Fig. 6) indicating the thermoactivated type of plastic deformation. Dependence of activation volume of plastic deformation on grain size can be identified from Fig. 7.

It can be seen from Fig. 7 that decrease of ECAP temperature and increase of ECAP passes leads to decrease of the activation volume values at all studied compression temperatures. 2 ECAP passes at 575 K give nearly the same the activation volume as coarse grained Ti indicating identical deformation mechanisms, while activation volume after 3 ECAP passes at 77 K considerably differ from coarse grained state indicating significant change of distance between the barriers for carriers of plasticity or even change of plastic deformation mechanism. All other studied structural states are between these two limiting cases, showing gradual change in activation volume (plasticity mechanisms).
4. Discussion of results

The microscopic mechanisms controlling the plastic deformation of coarse grained Ti are established quite well by now. In the studied temperature range (below 300 K) the kinetic of plastic strain in CP coarse grained Ti is controlled by thermally activated motion of dislocations over the local barriers formed by interstitial impurity atoms, mainly O and C [11]. Therefore, the dislocations in grain interior are mostly pinned at the impurity atoms, and dislocation segments between the pinning points are curved by the deforming stress. Dislocations repeatedly overcome these local barriers by thermal activation and start to move to be pinned by the next set of pinning points. Average length of the dislocation segment between the pinning points in coarse grained Ti can be written as [11]:

$$L = \beta b C^{-1/2},$$

where C is atomic concentration of oxygen; $\beta$ is empirical parameter ($\beta \approx 3$).
Severe plastic deformation by 2 ECAP passes at 575 K leads to formation of submicron grains in Ti. Decrease of average grain size after 2 ECAP passes at 575 K compared to initial coarse grained Ti leads to considerable increase of yield stress at all studied compression temperatures (Fig. 5, 6), while values of the activation volume are practically identical for these two structural states, which indicate that mechanism of plastic deformation after ECAP is also thermally activated overcoming of the impurity atoms. Decrease of the grain size in this case does not lead to change of the average length of the dislocation segment \( L \), which can be estimated using relation (2) taking into account the atomic concentration of O in Ti (0.0048) as \( L \approx 43 \text{ b} \), i.e. distance between the pinning points is considerably smaller than grain sizes, and grain boundaries cannot affect significantly the average dislocation segment length. Changes of grain size mainly cause change of level of internal stresses and correspondently the value of deforming stress. If the grain size decreased down to the dislocation segment length, the grain boundaries would not be acting as the additional pinning points for dislocations. In this case the grain boundaries appear to be the strong barriers for dislocations, which cannot be overcame by thermal activation and the change of deformation mechanism is expected. Decrease of ECAP temperature leads to diminished dynamic recovery [20,21], which gives more active accumulation of defects within grains, and to lower level of recrystallization (Fig. 1, 3), resulting is smaller grain sizes and higher strength values after ECAP. Another reason for smaller grains formation and higher strength after ECAP at lower temperatures is formation of twins, which is known to be active in Ti at low temperatures [22]. Considering that twin boundaries as well as the grain boundaries are effective barriers for dislocations, an increase of twin quantity leads to increase of strength.

After 1 pass of ECAP at 77 K the overwhelming part of the Ti volume remained coarse grained (Fig. 1 (a), 4 (a)), while strength and hardness of this structural state is considerably higher in comparison with initial coarse grained Ti and material after 1 pass of ECAP at 300 and 575 K. It can be explained by formation of rather thin layers of nanosized grains (cells) around the coarse grains in this structural state (Fig. 4a); these layers can be efficient barriers for dislocation motion, which leads to increase of strength. 1 ECAP pass at higher temperatures gives more efficient grain refinement - larger quantity of fragmented grains is observed (Fig. 1, 4) in comparison with material after 1 ECAP pass at 77 K, but typical size of these fragments of the grains is larger in comparison with nanosized grains after ECAP at 77 K. Higher level of severe plastic deformation (2 and 3 ECAP passes at 77 K) leads to formation of more developed ultrafine grained structures with minor portion of remnant coarse grains embedded in ultrafine grained matrix (Fig. 1, 4 (c)), and increase of hardness (strength). At higher ECAP temperatures, smaller quantity of the remnant coarse grains can be observed, while ultrafine grained subsystem has average sizes larger in comparison with ECAP at 77 K (Fig. 4 (c), 4 (d)), resulting in lower strength. Moreover, the grain boundaries of recrystallized grains with r low energy formed after ECAP at 300 and 575 K is easier to penetrate by dislocations in comparison with not recrystallized grains after ECAP at 77 K, which also explains observed higher strength at low ECAP temperature.

Decrease of the activation volume of plastic deformation is observed with decrease of ECAP temperature (Fig. 7). At compression temperature of 300 K the decrease of activation volume starts in the structural state of Ti, produced by ECAP at lowest temperature (77 K), while at lower compression temperatures changes of activation volume starts at higher ECAP temperatures (300 and 575 K). After 3 ECAP passes changes of activation volume are more pronounced in comparison with 2 ECAP passes. Two explanations of these observed changes of the activation volume are suggested:

(i) Decrease of ECAP temperature and/or increase of the number of ECAP passes leads to increase of defects (dislocations, forest of dislocations, subgrain boundaries, etc.) in grains due to suppressed dynamic recovery at lower ECAP temperatures. When the distance between these defects becomes comparable with the distance between the impurity atoms, the number of efficient pinning points for dislocations considerably
increases and the average length of the dislocation segments between the pinning points and the values of the activation volume decreases;

(ii) Decrease of the ECAP temperature and/or increase of number of ECAP passes leads to decrease in average grain size, which triggers the change of deformation mechanisms at sufficiently small grain sizes [23]. The concentration of oxygen does not control any more the process of plastic deformation in ultrafine grained Ti.

The effect similar to (i) was observed for coarse grained Ti with different concentrations of oxygen [11]. It was observed that increase of oxygen concentration leads to increase of the number of pinning points for dislocations, and decrease of average dislocation segment length, which results in increase of the strain rate sensitivity (decrease of activation volume). For different oxygen concentration [11] the biggest difference of the strain rate sensitivity (activation volume) was observed near 300 K, which decreased proportionally with decrease of strain temperature. However, in our case the difference in activation volume for different structural states increases with decreasing of compression temperature. This indicates that change of average distance between the pinning points (i) apparently does not determine the observed changes of the activation volume (Fig. 7), and the more plausible explanation of these changes is the change of the deformation mechanism triggered by decrease of ECAP temperature and increase of the number of ECAP passes. The values of the activation volume for different structural state of Ti and different strain temperatures (Fig. 7) can be considered as a kind of map of deformation mechanisms, which clearly indicates the temperatures and grain sizes where the change of the deformation mechanism starts. The available experimental data do not identify which specific deformation mechanism controls the plastic deformation of ultrafine grained Ti, but it can be speculated that nucleation of dislocations at grain boundaries is the dominant one as widely discussed in literature [24].

5. Conclusions
The results of this work can be summarized as follows:
- 1 and 2 ECAP passes produce complex structures, where areas of ultrafine grains coexist with remaining large grains.
- ECAP at 575 and 300 K results in rather equiaxed grains with clear grain boundaries due to action of the dynamic recovery, while ECAP at 77 K leads to diffuse and obscure grain boundaries;
- 3 ECAP passes results in more uniform microstructures with smaller average grain sizes;
- Decrease of ECAP temperature and increase of level of severe plastic deformation leads to higher strength characteristics in all studied cases; yield strength after ECAP at 300 K is approximately 10-15% higher in comparison with ECAP at 575 K for all studied compression temperatures. ECAP at 77 K gives additional 5-10% increase of strength in comparison with ECAP at 300 K;
- High ductility is registered for all studied structural states and compression temperatures;
- Considerable increase of strength with decrease of compression temperature indicates the thermoactivated plastic deformation;
- Thermoactivated motion of dislocations over local barriers (impurity atoms) in grains is the microscopic mechanism of plastic deformation of coarse grained Ti and ultrafine grained Ti processed by ECAP at higher temperatures.
- Decrease of ECAP temperature and increase of the number of ECAP passes leads to the change of the deformation mechanism, which can be defined from the values of the activation volume of plastic deformation for all studied structural states.
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