Microstructure and microhardness of the Ti15Mo alloy subjected to severe plastic deformation in α+β condition

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Abstract. Metastable β-titanium alloys are perspective candidates for use in biomedicine. For that purpose a high strength of these materials is required. The enhanced strength of these alloys can be achieved by subjecting the material to severe plastic deformation. However, due to the limited ductility of the metastable β-titanium alloys, deformation of the alloy in β solution treated condition at room temperature is sometimes inaccessible. In this study a Ti15Mo alloy in a two phase α+β condition was subjected to equal channel angular pressing at elevated temperature of 400°C. The resulting microstructure after N=4 and 8 ECAP passes consists of deformed β-phase with elongated α-phase particles. Despite the microstructure is not ultra-fine grained the microhardness of the deformed alloy exhibits higher values compared to the non-deformed counterpart.

1 Introduction
Metastable β-Ti alloys are nowadays industrially used in aircraft manufacturing industry, namely for construction of landing gears [1]. Binary Ti15Mo alloy investigated in this study is considered for biomedical applications as it contains only biocompatible elements [2]. Restriction on biocompatible elements results in lower strengthening by advanced alloying typical in high-strength alloys for aerospace industry [3]. Therefore, improvement of the strength can be achieved by significant grain refinement. For this purpose, severe plastic deformation (SPD) methods can be used which proved to be an effective approach to prepare ultra-fine grained (UFG) materials with high strength [4].

Equal channel angular pressing (ECAP) is a principal SPD method that uses simple shear deformation applied by pressing specimen through the die with the given angle [5]. For successful deformation, it is critical that material remains compact without cracking. Moreover, pressing force must be sufficient for pressing the material through the die. ECAP can be done at ambient temperature [6] or, if necessary at elevated temperatures [7]. Titanium alloys are very strong, extremely tough, with limited ductility and therefore very hard to deform by ECAP [8]. Critical is using ECAP die with the angle of 120° (i.e. not the most common 90°) [9, 10]. Despite some β-Ti alloys can be processed at room temperature [11], elevated temperatures are usually required [9, 10]. This brings another complication in case of metastable β-Ti alloys, as the material may undergo phase transformations during ECAP deformation. In this
study, Ti15Mo alloy in a fully aged condition (α + β) was used to avoid phase transformations during processing, namely formation of ω-phase which causes material embrittlement [3].

2 Experimental
The Ti15Mo alloy was supplied by ATI Corp. in a form of rod with a diameter of 10 mm. The material was thermally treated in order to get a duplex α+β microstructure. A 2-step thermal treatment (TT) consisted of annealing of the material above β-transus temperature for 1 hour followed by slow cooling below 552°C. The second step of the TT was a stress-relief annealing at 816°C for 10 minutes and consequent slow cooling below 552°C.

The deformation by ECAP was accomplished at Ufa State Aviation Technical University (USATU) in Ufa, Russia. The deformation was carried out at elevated temperature of 400°C in a die with channels intersecting at angle of $\Phi = 120^\circ$.

Samples after N = 4 and 8 ECAP passes were prepared for this study. The total achieved equivalent strain [12] was $\varepsilon_{\text{equiv}} = 2.66$ and 5.32, respectively. The principle of the ECAP process is schematically displayed in figure 1 and is described elsewhere [13].

Samples for scanning electron microscope (SEM) observations and microhardness measurement were prepared by mechanical grinding and polishing followed by a three-step vibratory polishing. The scanning electron microscope Zeiss Auriga CrossBeam operated at 10 kV was used for microstructural observations. The microstructure was studied in two planes – parallel to the processing (rolling direction) and perpendicular to the processing (normal direction) as schematically displayed in figure 1. Microhardness measurements were carried out using the automatic microhardness tester Qness Q10a by Vickers method; 0.5 kgf load, 20 indents per sample were applied.

![Figure 1. Schematic representation of the ECAP process and orientation of the sample (rolling-RD and normal-ND direction).](image)

3 Results
Figure 2 shows the initial microstructure of the Ti15Mo alloy in α+β condition. The α-phase particles appear darker in the image due to the chemical contrast (Mo depletion). α-particles are elongated with the length of few micrometers and submicrometer width.
Figure 2. BSE micrograph of the as-received Ti15Mo alloy in α+β condition.

In Figure 3 a and b the microstructure of Ti15Mo alloy after N=4 ECAP passes in both rolling and normal directions is shown. Elongated α-phase particles are only partly refined. Individual grains of β-phase cannot be resolved. α-phase particles appear bigger and more equiaxed in the normal direction when compared to the rolling direction.

Figure 3. BSE micrograph of the Ti15Mo alloy after N=4 ECAP passes in rolling direction (a) and normal direction (b)

Figure 4 a and b shows the microstructure of the Ti15Mo alloy after N = 8 ECAP passes in both rolling and normal directions, respectively. The microstructure is highly deformed and β-phase is significantly refined as it is well visible due to changes of the grey contrast. The microstructure is not homogeneous, areas with smaller α precipitates appear. Nevertheless, in normal direction less amount of α precipitates, which are even bigger, are present.
Evolution of the microhardness with the ECAP processing is shown in figure 5. The microhardness was measured both in rolling and normal directions. The microhardness of the Ti15Mo alloy in α+β condition reaches 360 HV [14] and is displayed by a dashed line in figure 5. The microhardness after N = 4 ECAP passes increases up to 383 HV and 374 HV in rolling and normal direction, respectively. The microhardness slightly increases with increasing number of ECAP passes, however, the increase is not significant. Microhardness of N = 8 condition is not homogeneous, which is consistent with the observation of the non-homogeneous microstructure in SEM.

Figure 4. BSE micrograph of the Ti15Mo alloy after N=8 ECAP passes in rolling direction (a) and normal direction (b)

Figure 5. Evolution of the microhardness with the number of ECAP passes in both normal and rolling directions (microhardness of the non-deformed Ti15Mo alloy is displayed by a dashed line)
4 Discussion
Deformation by 8 passes by ECAP was possible thanks to using the die with the outer angle of 120°, processing temperature of 400°C and initial material with thermodynamically stable α + β phase composition. Processing of β solution treated material is comparatively more complicated, probably due to the formation of the α-phase during ECAP processing, which causes material embrittlement [15]. After 8 ECAP passes, α-phase particles were deformed, but internal structure suggesting grain refinement was not observed. This is consistent with observation of Ti6Al7Nb alloy in α + β condition [16]. On the other hand, β matrix is refined which can be inferred from SEM observations, especially in figure 4(b).

The microhardness was measured using indentation according to Vickers with the load of 500g, which results in indents with the approximate diagonal length of diagonal of 50 μm. One indent therefore spans over several α-phase particles. Microhardness of the Ti15Mo alloy in α + β condition is superior to the microhardness of the Ti15Mo in solution treated β condition due to the presence of stronger α-phase and mainly due to strengthening by phase interfaces [3, 16]. The microhardness increases with increasing number of ECAP passes which can be attributed to the grain refinement of the β matrix. Microhardness of Ti15Mo alloy in α + β condition processed by ECAP is lower than the microhardness of β solution treated Ti15Mo alloy processed by high pressure torsion (HPT) – up to 510 HV [17]. HPT processed material is characterized by more refined microstructure with grain size of few hundreds nm and most importantly by the presence of the α-phase. On the other hand, α-phase is not present in this condition. It can be concluded that presence of the α-phase in β solution treated material after extreme refinement by HPT is decisive for achieving the microhardness which is significantly higher than in α + β aged structure processed by ECAP. Similar results were obtained in metastable β aerospace Ti-6.8Mo-4.5Fe-1.5Al alloy [18]. In this alloy, the presence of the α-phase results in maximum microhardness of 550 HV, while fine α + β structure is characterized by microhardness of 500 HV, which is caused both by solid solution strengthening and very fine α particles [18]. Comparable microhardness was not achieved by ECAP processing of Ti15Mo alloy. Note that despite the specimen is rotated successively between individual ECAP passes around its longitudinal axis, significant differences both in microstructure and microhardness are achieved in rolling and normal directions.

5 Conclusion
Biomedical metastable β Ti15Mo alloy was successfully prepared by 8 passes of equal channel angular pressing (ECAP) at 400°C using the die outer angle of 120°. The following conclusion can be drawn from this investigation:

- Fully aged two phase thermodynamically stable α + β condition of Ti15Mo alloy can be processed by ECAP easier than β solution treated material.
- α-phase particles are significantly deformed after N = 8 passes, but there is no sign of grain refinement within these particles.
- β-phase matrix was significantly refined by ECAP processing.
- Microhardness increases with the strain imposed by ECAP processing, but it does not reach the microhardness of Ti-6.8Mo-4.5Fe-1.5Al aerospace alloy in aged condition.

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