EVOLUTION OF MICROSTRUCTURE IN Ti15Mo ALLOY DEFORMED BY HIGH PRESSURE TORSION DURING LINEAR HEATING

Kristína BARTHA[1], Josef STRÁSKÝ[1], Jozef VESELÝ[1], Veronika POLYAKOVA[2], Irina SEMENOVA[2], Miloš JANEČEK[1]

[1]*Department of Physics of Materials, Charles University, Prague, Czech Republic, EU,* kristina.bartha@met.mff.cuni.cz

[2]*Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, Russian Federation*

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Abstract

The influence of severe plastic deformation on ongoing phase transformations in metastable β-Ti alloy Ti15Mo was investigated in-situ during linear heating by electrical resistance and complemented by ex-situ microstructure observations using transmission electron microscopy. Several non-monotonic regions in the temperature dependence of electrical resistance were identified and associated with corresponding phase transitions, namely the reversible diffusionless shuffle transformation of athermal ω phase, followed first by diffusion controlled growth of isothermal ω phase and subsequently its continuous dissolution. Formation of α phase at high temperature is associated with increase of the electrical resistance. The influence of severe plastic deformation on the phase transformations sequence is discussed in detail. For ex-situ microstructural observations different conditions were selected based on electrical resistance measurement. It was revealed, that lattice defects introduced by severe plastic deformation strongly influence the phase transitions during heating of the alloy.

Keywords: Metastable *β* Ti alloy, high pressure torsion, phase transformations, microstructure, electrical resistance

1. INTRODUCTION

Titanium alloys are known to be attractive materials for numerous industrial applications. High strength, low density and excellent corrosion resistance provide a large potential for a variety of applications in automobile and airspace industry [1]. Due to a low elastic modulus some Ti alloys are also widely used in orthopedics and implantation surgery [2].

Pure titanium is an allotropic material which undergoes a phase transformation at 882 °C from the low-temperature α phase (hexagonal close-packed structure) to the high-temperature β phase (body-centered cubic) [3].

In recent years, the main interest has been focused on development and investigation of a group of metastable β-Ti alloys. These alloys contain sufficient amount of β stabilizing elements (e.g. Mo, V, Fe, Nb) which can suppress the martensitic β → α transformation and retain the high temperature β phase in a thermodynamically metastable state even at room temperature after quenching [4]. A series of non-equilibrium phase transformations may occur in these alloys, which, if properly controlled, may influence the microstructure and mechanical properties of the material. In some metastable β-Ti alloys, tiny nanometer-sized particles of metastable ω phase may form during quenching. The ω phase has hexagonal structure (not close-packed) and belongs to the *P6/mmm* space group [5]. It is formed by a reversible displacive shuffle transformation [6] and referred to as ω athermal (ω_{ath}). Athermal ω particles are coherent with β matrix [7] and have a form of prolate spheroids. By ageing to higher temperatures (above ~ 150 °C, depending on the chemical composition of the alloy), when the diffusion becomes dominant, ω particles start to grow irreversibly and form so called ω isothermal phase (ω_{iso}). These particles are more chemically stabilized [8] and can serve as preferential

nucleation sites for α phase particles during further annealing at suitable temperatures (above 400 °C); therefore, the ultimately equilibrium α + β composition can be achieved [9,10].

Severe plastic deformation (SPD) is an attractive method which introduces a high density of lattice defects into the bulk material and causes fragmentation of its microstructure and significant grain size reduction up to the submicrometer or even nanometer range [11-13]. One of the most popular methods of SPD is the high pressure torsion (HPT). It proved to be the most effective technique of grain refinement due to the introduction of extremely high plastic strain by torsional deformation [14,15].

In our previous studies phase transitions have been thoroughly investigated in several coarse-grained metastable β-Ti alloys (both single- and polycrystals) using several experimental techniques (microscopy, X-ray diffraction, electrical resistance, etc.) [16,17]. Recently, we have focused on the influence of SPD on the kinetics of ongoing phase transformation, in particular on the precipitation of α phase [18]. However, a detail study addressing all aspects of the influence of SPD on phase transitions is still missing. In order to fill this gap, we have selected a simple binary metastable β-Ti alloy Ti15Mo and employed in-situ electrical resistance measurement complemented by ex-situ observation of the microstructure in selected conditions by transmission electron microscopy to assess the details of the kinetics of phase transitions in severely deformed material.

2. EXPERIMENTAL

Ti15Mo alloy, which was subject of the investigation, was supplied by Carpenter, Co, USA in a form of rods with the diameter of 10 mm. The supplied rod was solution treated at 810 °C for 4 hours. Cylindrical specimens with height of approximately 5 mm were cut from the solution treated (ST) material (hereafter referred to as undeformed condition). These specimens were first pressed with a high pressure of 6 GPa to double their diameter. HPT deformation was subsequently performed at room temperature (RT) and the same pressure by a single turn at USATU Ufa, Russian Federation. The resulting HPT-deformed samples had a shape of discs with the diameter of 20 mm and approximate height of 1 mm.

The measurement of electrical resistance was performed using a standard four-point method employing a self-constructed apparatus. The voltage and electrical current were measured simultaneously using nanovoltmeter Keithley 2182 and SourceMeter Keithley 2400 devices, respectively. The relative error of such a measurement is lower than 10^{-4} within each measured point and acquisition rate is two experimental values/second. The details of the apparatus and the measurement are given elsewhere [19]. The measurement was performed in-situ during linear heating with the heating rate of 5 °C/min from room temperature up to 750 °C.

Thermal treatment to selected temperatures based on results of the measurement of electrical resistance was carried out in a furnace with the same heating rate of 5 °C/min. During heating the specimens were sealed in a quartz tube filled with Ar to prevent contamination. After reaching the desired temperature, the specimens were immediately quenched in water.

Post mortem microstructure observations were performed using transmission electron microscope (TEM) Jeol JEM 2200 FS operated at the acceleration voltage of 200 kV. Thin foils for TEM observations were prepared from the periphery part of the HPT-deformed sample (where the deformation reaches its highest values; $\varepsilon_{vM} \sim 36$) by twin-jet electro-polishing (TENUPOL-5) unit at the temperature of -20 °C and finished by low voltage Ar ion milling using Leica EM RES102 ion polisher.

3. RESULTS AND DISCUSSION

The evolution of the relative electrical resistance with temperature for undeformed and HPT material is shown in **Figure 1** (note that absolute electrical resistivity cannot be determined due to a complex shape of the sample) [20]. The relative electrical resistance was calculated by dividing the measured resistance by the

reference value of the electrical resistance at the temperature of 40 °C. The solid and dashed line represent the evolution of the relative electrical resistance of the undeformed and HPT-deformed Ti15Mo alloy, respectively. An unexpected behavior consisting of several stages of monotonic dependence of the electrical resistance on the temperature were found. Note, that similar behavior was observed in our previous work by Zháňal et al. [17] on coarse-grained Ti15Mo alloy and the microstructure changes controlling the behavior of electrical resistance in individual temperature ranges were discussed in detail.

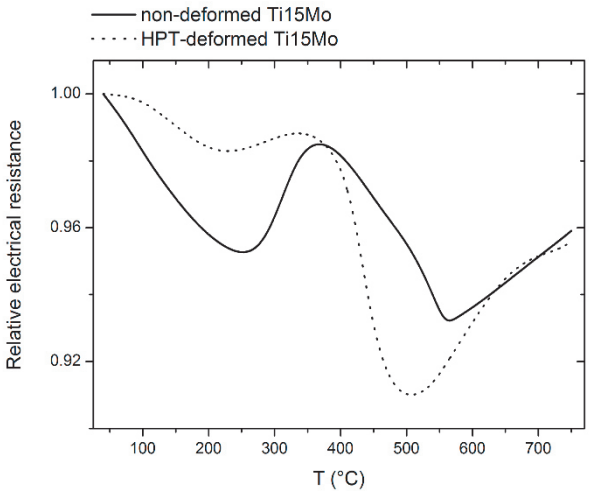

Figure 1 Temperature dependence of the relative electrical resistance of the undeformed (solid line) and HPT-deformed (dashed line) Ti15Mo alloy

In the first stage (from RT to 250 °C) the decrease of the relative electrical resistance, which outweights the phonon scattering effect, was observed and attributed to the dissolution of the ω_{ath} particles [17]. The continuous dissolution of ω_{ath} precipitates during heating is accompanied by the partial release of the elastic strain field formed originally during quenching around ω_{ath} precipitates and results in easier electron drift in the β matrix [21]. As a result, the electrical resistance decreases with increasing temperature. The decrease of electrical resistance in HPT material is smaller due to the high density of lattice defects introduced to the material by severe plastic deformation. These defects act as additional scattering centers for electrons even after dissolution of the ω_{ath} phase.

The second stage (from 250 °C to 365 °C) is characterized by an increase of the relative electrical resistance with increasing temperature and is related to the formation and growth of ω_{iso} precipitates [22]. Both the formation and growth of these precipitates result in increasing amount of β/ω interfaces [23] which represent additional obstacles for electron drift, and thus, together with the phonon contribution, contribute to the increase of the electrical resistance in this stage.

The third stage (from 365 °C to 560 °C) represents a decrease of the relative electrical resistance in both undeformed and HPT samples. It is caused by decreasing amount of β/ω interfaces due to simultaneous growth and dissolution of the ω_{iso} precipitates [24]. For HPT sample the decline of the relative electrical resistance is more pronounced. Besides the dissolution of the ω_{iso} phase, recovery of defects and possible recrystallization of the microstructure enhanced by the presence of lattice defects may play a significant role in deformed material. A complete dissolution of the ω_{iso} phase at 560 °C reported also in [17] is accompanied by an abrupt change in the monotonicity of the evolution of electrical resistance with temperature. The above mentioned change is also achieved in HPT-deformed material; however, it is shifted to lower temperatures. This may be attributed to shifted β → α transformation to lower temperatures in HPT-deformed material [18].

In the fourth stage (from 560 °C to 750 °C) an increase of the electrical resistance of the undeformed material is almost linear, suggesting that the nucleation and growth of the α phase has only a limited effect on the electrical resistance due to its relatively low volume fraction and large particle size. On the other hand, heating of the HPT-deformed sample shows a small peak between 600 °C and 700 °C. This can correlate with the increased volume fraction of α phase compared to the undeformed counterpart; the deformation and induced lattice defects enhance the precipitation of the α phase due to preferential nucleation of the α phase at defects and accelerated growth due to enhanced diffusion along lattice defects [25]. The decrease of the slope of the relative electrical resistance curve after heating to temperature above 650 °C of the HPT sample can be associated with the dissolution of the α phase in the vicinity of the *β*-transus temperature.

For further investigation of the microstructure post mortem in HPT-deformed material three characteristic temperatures were selected based on the behavior of electrical resistance at 350 °C, 500 °C, and 650 °C.

Figure 2 shows TEM bright field images of HPT samples after heating to three selected temperatures of 350 °C, 500 °C, and 650 °C, respectively. The HPT-deformed sample heated to 350 °C in **Figure 2 (a)** contains a heavily deformed and ultra-fine grained (UFG) structure. **Figure 2 (b)** representing the HPT sample heated to the temperature of 500 °C indicates that the microstructure remains UFG, however, small grains of the α phase are already visible. The presence of the α phase was also proved by X-ray diffraction [20] and are published elsewhere [26]. The sample heated to 650 °C shows a recovered, but still UFG microstructure containing grains of β and α phase with the grain size ~ 200 nm (note the lower magnification in **Figure 2 (c)** compared with **Figures 2 (a)** and **(b)**).

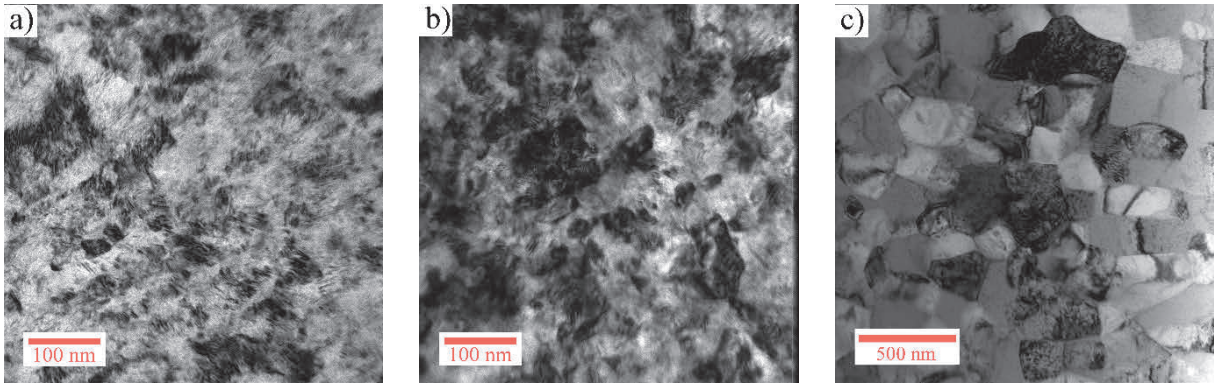

Figure 2 TEM bright field image of the HPT-deformed Ti15Mo alloy (a) heated to temperature 350 °C, (b) heated to temperature 500 °C, (c) heated to temperature 650 °C (note the lower magnification in this case)

4. CONCLUSIONS

The influence of severe plastic deformation on phase transformations occurring in a metastable β-Ti alloy Ti15Mo during heating was investigated in-situ by electrical resistance measurements and complemented by ex-situ microstructure observations using transmission electron microscopy. The following conclusions may be drawn from this experimental study:

- HPT deformation introduced a high density of lattice defects into the material and resulted in a strong grain refinement.
- The evolution of the electrical resistance upon heating is complex - it consists of several stages with alternating positive (normal behavior) and negative slope (inverse dependence).
- Lattice defects introduced by severe plastic deformation influence the phase transformations - they accelerate the precipitation (both nucleation and growth) of the α phase.
- Electrical resistance measurement proved to be a very sensitive technique for determination of the kinetics phase transformations in metastable β-Ti alloy.

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