In-Situ Observation of the Phase Transformations in Ti15Mo Alloy Deformed by High Pressure Torsion

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Abstract. Metastable β-Ti alloys including Ti15Mo alloy are perspective candidates for use in medical applications. During thermal treatment Ti15Mo alloy undergoes various phase transformations. After solution treatment it contains metastable β-phase and ω-phase. During annealing the ω-phase partially dissolves as well as stable α-phase particles are formed.

The solution treated Ti15Mo alloy was deformed by high pressure torsion (HPT) at room temperature. Significant grain refinement with grain size of ~100 nm was achieved even after 1/4 of HPT rotation. The effect of the ultra-fine grained (UFG) structure achieved by HPT on the phase transformations was studied by differential scanning calorimetry (DSC) and transmission electron microscopy (TEM) during in-situ heating. High density of lattice defects, dense network of grain boundaries as well as ongoing recovery and recrystallization upon heating significantly affected the phase transitions. Observation of the microstructure during in-situ heating in TEM revealed no representative changes in transparent part of the sample due to the “thin foil effect”.

Introduction

Metastable β-Ti alloys provide an advantageous balance of mechanical and physical properties compared to other titanium based alloys [1]. Among them, Ti15Mo is a thoroughly investigated binary alloy and a promising candidate for biomedical use [2]. Various thermal and mechanical treatments of titanium alloys were proposed to tailor their structure and properties. Similar to iron, titanium has different allotropic modifications at high and low temperatures and thermal treatment (TT) of these alloys leads to several phase transformations in the material.

It was previously reported that after quenching from a temperature above β-transus temperature (β-solution treated condition) Ti15Mo alloy primarily consists of β-matrix embedded with nanosized athermal ω (ωath) particles formed by diffusionless transformation [3, 4]. Upon heating, part of the ωath particles dissolves back to β-matrix which is associated for instance with the change in the electrical resistance [5, 6]. Further TT results in the stabilization of ω particles by diffusion process expelling Mo atoms – these particles are then termed as isothermal ω (ωiso) phase. The ωiso-phase particles tend to grow during annealing and simultaneously become more chemically stabilized [7]. Furthermore, under certain conditions ωiso-phase can serve as nucleation site for precipitation of the α-phase [8, 9]. Other preferential nucleation sites for α-phase are β/β grain boundaries and dislocations [10].

Severe plastic deformation (SPD) has been recognized as an effective way to introduce a high density of dislocations to the material and thus to increase strength [11]. Equal channel angular pressing (ECAP) and high pressure torsion (HPT) are the most widely used SPD techniques nowadays [12, 13].

Present study investigates the effect of the ultra-fine grained (UFG) structure induced by HPT deformation on the phase transformations in Ti15Mo metastable β-Ti alloy. Advanced methods of
in-situ differential scanning calorimetry (DSC) combined with transmission electron microscopy (TEM) during in-situ heating were used for this investigation.

Experimental

The rod (diameter approx. 10.5 mm) of the Ti15Mo alloy was supplied by Carpenter Technology Corp. The as-received material was solution treated (ST) at the temperature of 810°C for 20 min in a protective inert atmosphere and subsequently water quenched. The rod was cut to cylinders with the height of 5 mm. The samples were processed by HPT at Ufa State Aviation Technical University, Russia at room temperature and the pressure of 2 GPa. Disc-shaped samples with final thickness of 1 mm were prepared by N = 1/4 and N = 1 HPT turns.

Transmission electron microscope (TEM) Jeol JEM 2200 FS operated at 200 kV was used for microstructure observation. Thin foils for TEM study from the periphery part of the material after 1/4 and 1 HPT rotation were prepared by twin-jet electro-polishing (Tenupol-5) unit at -20°C and finished by low voltage Ar ion milling (Leica EM RES102 ion polisher).

Differential scanning calorimetry (DSC) measurements were performed on Netzsch calorimeter DSC 404 Pegasus in dynamic Ar atmosphere. Samples with the dimensions of 3.5x3.5x1 mm³ were prepared from solution treated Ti15Mo alloy and from the periphery part of the HPT (N = 1) deformed sample.

Results and Discussion

TEM observation of HPT-deformed (N = 1/4) Ti-15Mo alloy. TEM bright field (BF) image in Fig. 1 shows the microstructure of the HPT-processed (N = 1/4) Ti15Mo alloy. Ultra-fine grained structure with nanometer sized grains is observable already after N = 1/4 HPT turns. The microstructure of the Ti15Mo alloy after higher number of HPT rotations (N > 1/4) exhibits more deformed structure as it was reported in our previous work [14]. The non-uniform contrast of the grains in the BF image is associated with the distortion of the lattice [15]. The phase composition comprising β + ωath phases was confirmed by selected area electron diffraction and X-ray diffraction measurements (not shown here). Depending on the chemical composition, internal stresses and the density of lattice defects, the so-called stress-induced ω-phase can form in other metastable β-Ti alloys. In contrast to the ωath-phase, the stress-induced ω-phase has a plate-like morphology [16, 17]. During severe plastic deformation of less stabilized metastable β-Ti alloys other martensitic transformations such as β→α" can occur [18, 19]. Nevertheless, other authors reported the absence of the ω-phase in UFG material due to the reverse ω→β transformation during deformation [20].

In-situ differential scanning calorimetry (DSC). The DSC measurement of β solution treated and HPT deformed Ti15Mo alloy was performed during heating up to 700°C with the heating rate of 50°C/min. Fig. 2 shows the evolution of the DSC signal of solution treated (ST - solid black line) and HPT processed (HPT - dashed red line) Ti15Mo alloy during linear heating. The DSC signal is displayed for temperatures above 100°C in order to get appropriate data not influenced by fast heating rate at lower temperatures.

Heating up to approx. 225°C results in no significant change in the DSC signal. However, the electrical resistance measurement reported in [4] indicated the dissolution of the ωath-phase in this temperature range. The DSC signal cannot identify such changes, because the dissolution of the ωath-phase is a displacive transformation with negligible latent heat.

During subsequent heating, the ST sample exhibits an outset of exothermic process up to the peak around 400°C. This exothermic transformation of the ST Ti15Mo alloy is connected with the stabilization of the ωath particles by expelling Mo atoms – transformation to ωiso-phase [21]. In contrast, HPT deformed sample exhibits rather wide peak from 250 to 550°C. The reason could be the overlapping processes of the exothermic diffusion controlled ωiso-phase formation, exothermic process of α precipitation and exothermic processes of recovery and recrystallization. During fast...
heating (50°C/min), all these processes take place concurrently and individual peaks cannot be resolved in the DSC data.

At higher temperatures (above 400°C) ω-phase dissolves in ST Ti15Mo alloy. Evident endothermic DSC peak on the temperature 560°C suggests massive dissolution of the ω-phase. During further heating of the solution treated material, α-phase particles nucleate and grow until equilibrium condition for given temperature is achieved. As a consequence, at approx. 600°C for ST material, the moderate exothermic process is observable. In HPT deformed Ti15Mo alloy, the endothermic peak is missing, which can be attributed to earlier formation of α-phase due to preferential nucleation on lattice defects [14]. Small exothermic peak is apparent at the temperature of 550-600°C which can be attributed to the homogeneous precipitation α-phase particles in the β-matrix and their coarsening.

High density of lattice defects in UFG material introduced by severe plastic deformation accelerates the precipitation of the α-phase as it was reported in our previous work [14]. The effect of the UFG microstructure prepared by HPT on phase transformations upon thermal treatment was also studied in other materials [22].

Fig. 1: TEM bright field image of the Ti15Mo alloy after $N = \frac{1}{4}$ turn of HPT deformation – periphery part of the sample

In-situ TEM during heating of the HPT deformed Ti15Mo alloy. Based on the DSC measurement shown above, in-situ observation by TEM during heating was performed in order to elucidate the microstructure changes in the material during phase transformation. Sample from the peripheral part of the HPT material after $N = 1$ HPT rotation was prepared for TEM investigation. The specimen was heated analogously to the DSC measurement with the heating rate of 50°C/min up to temperature 700°C and subsequently isothermally aged for 30 minutes.

The BF TEM image on the Fig. 3(a) shows the highly deformed microstructure of the Ti15Mo alloy after 1 HPT rotation before heating. The individual grains of the UFG Ti15Mo alloy cannot be distinguished due to the overlapping grains through the thickness of the foil. In the Fig. 3(b), the microstructure of the sample after in-situ heating and isothermal ageing at 700°C is shown. It is apparent that the microstructure of the specimen in the observable area by TEM (i.e. transparent area for electrons) did not change significantly. The fundamental reason of the unchanged microstructure can be attributed to the “thin foil effect” [23]. The transparent part of the thin foil is in fact two dimensional object and therefore diffusion is significantly reduced. As the result, the nucleation of the α-phase cannot take place even at the temperature of 700°C. Annealing for longer times may provide sufficient driving force for diffusion and precipitation of the α-phase even in transparent part of the sample.
Summary

The microstructure of ultra-fine grained metastable beta Ti15Mo alloy deformed by high pressure torsion (HPT) and the effect of severe deformation on the ongoing phase transformations were investigated. The following conclusions can be drawn from this experimental study:

- Transmission electron microscopy proved severely deformed nanocrystalline microstructure of the Ti15Mo alloy already after $N = 1/4$ HPT rotation.
- Differential scanning calorimetry measurements indicated that ultra-fine grained structure has a significant effect on the phase transformations in metastable $\beta$-Ti alloys. During heating several physical processes take place concurrently, such as dissolution of the $\omega$-phase particles, recovery and recrystallization of lattice defects which results in overlapping of DSC peaks of phase transitions.
In-situ TEM experiment during heating up to 700°C and subsequent ageing for 30 minutes could not reveal significant changes in the transparent area of the thin foil. Precipitation of α-phase particles was completely suppressed in the TEM foil, due to lack of dislocations or due to reduced diffusion.

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