

Thermal expansion evolution of metastable β Ti-15Mo alloy during linear heating

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Abstract. Thermal expansion measurement was employed for characterization of phase transformations occurring in metastable β titanium alloy (Ti-15Mo) during linear heating with three different heating rates. Effects of the transformations on materials dimensions is discussed and compared with changes of lattice parameters of present phases obtained from in-situ powder neutron diffraction. The shrinkage of the material with increasing temperature at about 300 °C was attributed to the decrease of lattice parameter of β phase, which occurs due to diffusion of alloying elements out of ω phase. Complete dissolution of ω phase was detected at 560 °C regardless of the heating rate.

1 Introduction

Titanium alloys are used in a wide range of components for aerospace and chemical industry and as alternative structural materials for automotive sector due to their high specific strength and excellent fatigue and corrosion resistance [1,2]. Moreover, due to biocompatibility and partial or total superelasticity, β -Ti alloys are also considered for biomedical applications [3,4]. The only disadvantage, which hinders wider use of titanium, is its comparatively high cost, which is caused by high processing costs.

Titanium is a polymorphic material. The high-temperature phase has a body-centered cubic structure (bcc), so called β phase. The ambient temperature phase has a hexagonal close-packed structure (hcp) and is referred to as α phase. In recent years, the main interest has been focused on development and



investigation of the so-called metastable β alloys. These alloys contain sufficient amount of β stabilizing elements to retain the high temperature β phase at room temperature after quenching. β phase is in a metastable state and ultimately equilibrium $\alpha + \beta$ composition can be achieved by annealing treatment [1,4,5]. Several other structural phases can be found in metastable β Ti alloys. In the investigated Ti-15Mo, hexagonal (non-close packed) ω phase forms by a diffusionless shuffle transformation first described in [6]. The phase formed during quenching is referred to as athermal ω (ω_{ath}). Athermal ω particles have similar chemical composition and are coherent with β matrix [7]. ω particles are prolate spheroids and their main semi-axes have a few nanometers [8,9]. By heating up to certain temperature (around 110 °C in Ti-15Mo), β stabilizing elements (Mo in our case) are rejected out of ω particles. From the temperature at which the diffusion starts to play a role, ω phase is referred to as isothermal ω (ω_{iso}). ω_{iso} particles grow during annealing/heating and become more chemically stabilized i.e. they differ from surrounding β matrix in composition [10]. ω particles serve as preferential nucleation sites for α phase particles precipitation during annealing at suitable temperatures (above about 400 °C). Mechanical properties of metastable β titanium alloys are mainly determined by the distribution and the size of metastable ω particles or stable α precipitates in β matrix. The formation of the precipitates, their morphology, volume fraction, the size and the distribution are influenced by phase transformations occurring during heat treatment. Therefore, a thorough understanding of these processes is necessary to optimize the mechanical properties of the material through thermomechanical treatment.

This paper presents a study of phase transformations occurring in the metastable β titanium alloy Ti-15Mo (15 wt % (8.1 at. %) of molybdenum). Ti-15Mo alloy was chosen for this experimental study, because it is designed for biomedical use [3], and it represents a simple binary system that has been already widely investigated [11].

2 Materials and methods

The metastable β -Ti alloy (Ti-15Mo) used in this study contains 15 wt % (8.1 at %) of molybdenum and negligible amounts of impurity elements (less than 0.2 of O, 0.1 of C and Fe, 0.05 of Ni and 0.015 of H in wt % [12]). The material was solution treated (ST) at 900 °C for 4 hours in a quartz tube filled with high-purity Ar and quenched in water. This condition corresponds to the ST initial state for the investigation.

In-situ thermal expansion measurement (dilatometry) and in-situ powder neutron diffraction were employed to investigate phase transformations in Ti-15Mo alloy.

2.1 Dilatometry

Evolution of thermal expansion of Ti-15Mo was measured in-situ during linear heating with the heating rates of 1, 5 and 20 °C/min up to 850 °C utilizing the Linseis L75 PT vertical dilatometer. Simultaneous measurement of standard sapphire specimen was utilized to determine the thermal expansion of the apparatus, which was utilized to correct thermal expansion of investigated samples. Dilatometry is a technique for characterizing dimensional changes of a material caused by physical or chemical processes. From the dilatometric curve it is possible to analyze temperature and kinetics of phase transformations or to calculate the coefficient of thermal expansion.

2.2 Neutron diffraction

Neutron diffraction experiments were performed at the Institut Laue-Langevin (ILL), Grenoble, France, at the instrument D20 with an extremely high neutron flux utilizing neutrons with the wavelength of 1.54 Å. The data were obtained within [13] proposal. The detector at D20 covers a

scattering range of 153.6° , and allows to obtain the diffraction pattern in the order of seconds as a function of temperature, pressure or other parameters. The acquisition time of a single spectra was 30 s and the heating rate of $5^\circ\text{C}/\text{min}$ was utilized.

3 Results and discussions

3.1 The influence of heating rate on phase transitions measured by in-situ dilatometry

The evolution of thermal expansion of Ti-15Mo was measured in-situ during linear heating with the heating rates of 1, 5 and $20^\circ\text{C}/\text{min}$ up to 850°C . The results of all measurements of thermal expansion are shown in figure 1. All curves deviate from linearity for all studied heating rates. The deviation from the linear trend is shifted to higher temperatures for faster heating rates. Between $400 - 450^\circ\text{C}$ the material starts to expand faster, up to about 560°C , where the curves return to linearity up to 850°C . The point of return of the curves to the linearity at 560°C corresponds to a complete dissolution of ω particles [8].

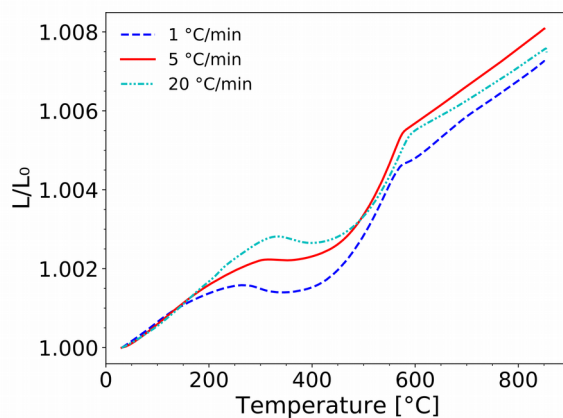


Figure 1. Thermal expansion of Ti-15Mo during heating with different heating rates.

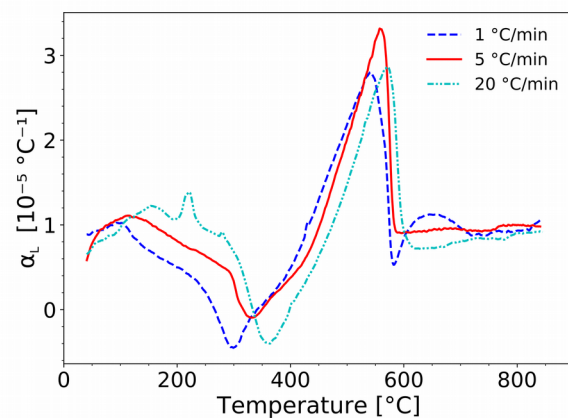


Figure 2. Thermal expansion coefficient of Ti-15Mo during heating with different heating rates.

Additional information can be obtained from the derivative of thermal expansion, which represents the linear expansion coefficient (α_L) of the material. The definition of the linear expansion coefficient is given by the equation:

$$a_L = \frac{1}{L_0} \frac{dL}{dT} \quad (1)$$

The evolution of α_L for all three heating rates is displayed in figure 2. Unfortunately, the heating rate of $20^\circ\text{C}/\text{min}$ is already beyond the performance ability of the apparatus, which resulted in influencing the results at lower temperatures. The furnace was unable to heat the specimen at low temperatures fast enough. The correct heating rate was reached at about 250°C , which is manifested by a peak at this temperature, which does not appear in the other curves. Otherwise all curves show similar trends. Interestingly, the values of global minima of α_L observed in temperature range $300 - 400^\circ\text{C}$ are negative, which corresponds to shrinking of the material with increasing temperature for all heating rates. This behaviour can be attributed to the decrease of lattice parameters of both β and ω phases,

which was also observed during isothermal ageing of LCB alloy at 300 °C [14]. At higher temperatures α_L increases and the dissolution of ω particles at 560 °C causes an abrupt decrease of α_L . Between 560 and 750 °C there is an apparent bump at α_L evolution measured with the heating rate 1 °C/min. This bump is probably connected to α phase formation and its subsequent dissolution towards β -transus as the slowest heating rate provides the longest time for α phase to precipitate. The last observable change of α_L at all curves, which probably corresponds to complete dissolution of α phase (β -transus), occurs at about 760 °C. At temperatures above 800 °C the values of α_L are nearly equal for all heating rates indicating that no additional transformations occur in the material.

3.2 Evolution of lattice parameters determined from powder neutron diffraction

Powder neutron diffraction of Ti-15Mo was measured in-situ during linear heating with the heating rate of 5 °C/min up to 850 °C. The evolution of the lattice parameter of β phase (bcc) is shown in figure 3 and the development of lattice parameters of ω phase (hexagonal elementary cell) is compared in figure 4. The scatter of the values of lattice parameters of ω phase at low temperatures (below 300 °C) is caused by extremely small dimensions of ω particles, which hinders fitting of diffraction patterns. ω particles start to grow at about 300 °C and the values of their lattice parameters are determined more easily. As a result the scatter of the values of lattice parameters is negligible for higher temperatures. The absence of values of lattice parameters of ω phase above 560 °C (see figure 4) is due to complete dissolution of this phase at 560 °C.

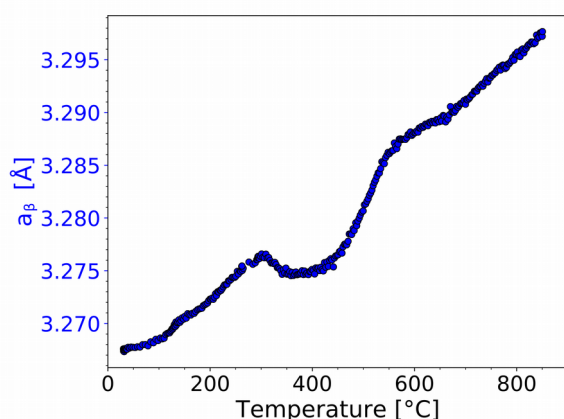


Figure 3. The evolution of a_β during heating with heating rate of 5 °C/min.

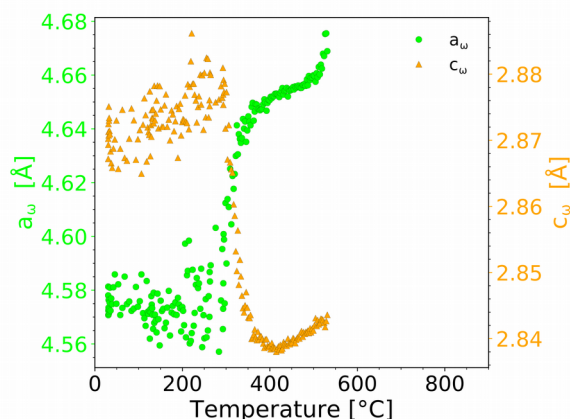


Figure 4. The evolution of a_ω and c_ω during heating with heating rate of 5 °C/min.

The comparison of evolution of volume of elementary cells of β and ω phases is shown in figure 5 as blue dots and green triangles, respectively. The increase of a_ω above 300 °C prevails over the decrease of c_ω and the volume of the ω cell increases. The volume of the elementary cell of β phase obviously follows the trend of its lattice parameter (cf. Figures 3 and 5).

The evolution of the lattice parameter of β phase has a dominant effect on thermal expansion of the material. This can be easily understood from figure 6, where the comparison of relative values of thermal expansion (red curve) and the evolution of relative lattice parameter of β phase (blue dots) is

presented. The lattice parameter closely follows the trend of dilatometric curve, even the values are very similar.

The evolution of lattice parameters of α phase (observed between 580 and 720 °C), was omitted because no distinct changes of samples dimensions were detected in this particular temperature range.

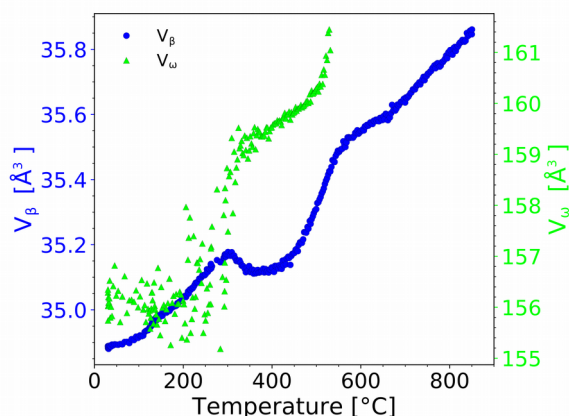


Figure 5. The comparison of evolution of volume of elementary cell of β and ω phases during heating with heating rate of 5 °C/min.

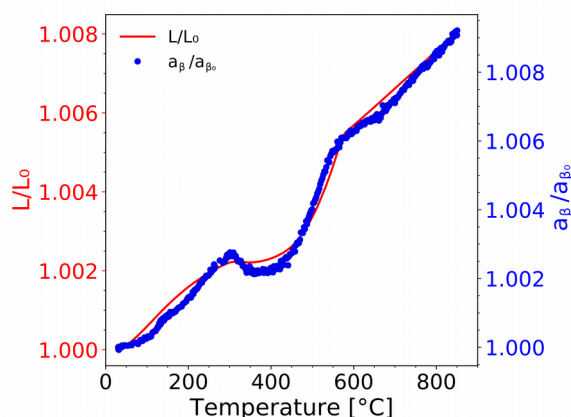


Figure 6. The comparison of thermal expansion and evolution of relative lattice parameter of β phase during heating with heating rate of 5 °C/min.

4 Conclusions

The main results of the present work can be summarized as follows:

- Dilatometry proved to be a suitable method for the detection of phase transformations occurring in titanium alloys.
- Around 300, 330 and 360 °C for the heating rates of 1, 5 and 20 °C/min, respectively, the material exhibits a decrease in length with increasing temperature for all measured heating rates.
- The dissolution of ω phase, which causes an abrupt decrease of α_L , was detected at the same temperature as in electrical resistance experiments (560 °C) presented in our previous work [8].
- The evolution of lattice parameters of β and ω phases during heating with the heating rate of 5 °C/min was determined from neutron powder diffraction experiment.
- The diffusion of alloying elements out of ω particles greatly affects lattice parameters of both β and ω phases.
- The thermal expansion of Ti-15Mo during heating is primarily affected by evolution of lattice parameter of β phase.

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