Experimental report

21/12/2018

Proposal: **DIR-155** **Council:** 4/2017

Title: Study of Phase Transformations inTI-15MO Alloy Leading to Inverse Resistivity Evolution

Research area: Materials

This proposal is a new proposal

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Samples: TI-15MO

Instrument	Requested days	Allocated days	From	To
D20	2	2	19/06/2018	21/06/2018

Abstract:

Study of phase transformations in Ti-15Mo alloy leading to inverse resistivity evolution

Introduction

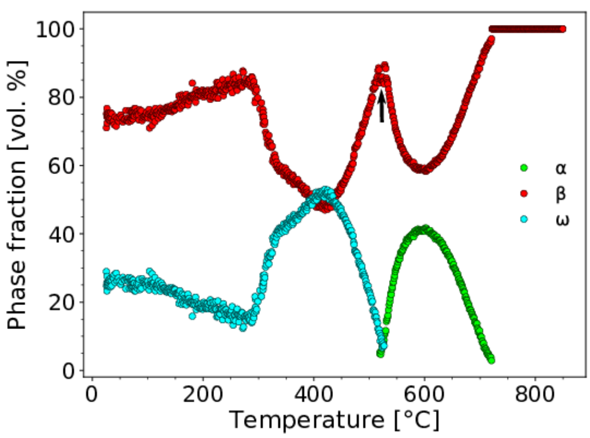
Metastable β-Ti alloys are of great interest to the automotive, aerospace and biomedical industries due to their outstanding mechanical properties. In particular, meta-stable β-Ti alloys have high strength, low density, excellent corrosion resistance, toughness and good hardenability as a result of ageing treatment involving several phase transformations. Therefore, investigation of these phase transformations is of significant importance. The mechanical properties of metastable β-Ti alloys are mainly determined by the distribution and the size of metastable hexagonal ω particles or stable hcp α precipitates in the bcc β matrix. The formation of the precipitates, their morphology, volume fraction, the size and the distribution depend on the heat treatment conditions. The phase transformations which are expected to occur during heat treatment of the studied meta-stable β titanium alloy Ti-15Mo are β ↔ ω, ω → α and β ↔ α. The kinetics and transitions sequence are not fully understood. Few samples for in-situ observations were prepared. The samples were aged at 900 °C for 4 hours and subsequently quenched in water. Such material consists of β phase matrix and extremely fine (∼ 1 − 4 nm in length) ω particles, which were formed during quenching. The aim of this study is to investigate the kinetics of the phase transformations to help us understood the inverse resistivity evolution which takes place during the ageing process. An in-situ neutron diffraction is an ideal tool for such a study where the information from whole sample volume needs to be considered.

Results of the experimental

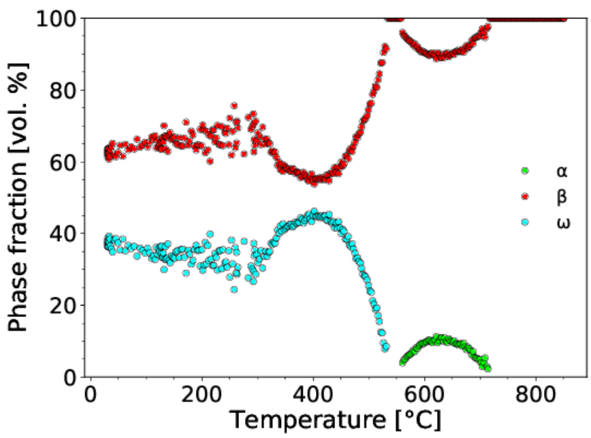
Two types of experiments aiming to determine the details of phase transformations occurring in Ti-15Mo alloy were performed:

a) the influence of the heating rate on the kinetics of phase formation,
b) investigation of kinetics of ω dissolution and α formation during ageing.

Evolutions of volume fractions of α, β and ω phases during heating with heating rates of 1.9 and 5 °C/min are shown in Fig. 1. For both heating rates, the volume fraction of ω phase initially decreases. The scatter of values at low temperatures is due to extremely small dimensions of ω particles, which hinders fitting of the diffraction patterns. At about 300 °C, the volume fraction of ω phase starts to grow, reaching its maximum volume at about 400 °C. The maximum volume



(a) Heating rate of 1.9 °C/min.



(b) Heating rate of 5 °C/min.

Figure 1: The evolution of volume fractions of α, β and ω phases during heating.

fraction of ω phase is 52 % (due to the presence of Nb peaks in diffraction pattern this value is incorrect; the correct value of volume fraction of ω phase at this temperature determined from the ageing experiment is 68 %) and 45 % for the heating rates of 1.9 and 5 °C/min, respectively. The volume fraction of ω phase continuously decreases up to 560 °C above which ω phase completely disappears. For the slower heating rate, a small temperature range (∼9 °C) of coexistence of α and ω phases can be observed. It is probably due to precipitation of α phase on grain boundaries. This fact is supported by a small decrease of volume fraction of β phase in this temperature range (marked with the black arrow in Fig. 1a), while the rate of decrease of the volume fraction of ω phase remains constant. The volume fraction of α phase reaches the maximum value of 41 % and 11 % above 600 °C for the heating rates 1.9 and 5 °C/min, respectively. During subsequent heating, α phase dissolves back to β phase, which is manifested by the decrease of the the volume fraction for T>600 °C and T>620 °C for the heating rate of 1.9 and 5 °C/min, respectively.

The development of volume fractions of α, β and ω phases in the material aged at 450 C is shown in Fig.2. The kinetics of α phase growth was determined from the Avrami equation [1,2]:

$$\zeta = A\{1-\exp[-k(t-\tau)^n]\} \quad ,$$

where ζ represents the volume fraction of nucleated phase, k is the growth rate constant and A corresponds to the saturation value of the precipitating phase. In the standard Avrami equation τ refers to the incubation period, however in this case it stands for a time, when ω particles grow to a

critical point and α phase begins to precipitate. With respect to shape of α precipitates, the exponent n was set $n = 1$, which corresponds to diffusion controlled growth of needles or plates [2]. The determined values of the coefficients from above mentioned equation are:

$$A = 0.51,$$
$$k = 0.24\ h^{-1},$$
$$\tau = 1,57\ h$$

The comparison of determined volume fraction of α phase with Avrami equation with fitted parameters is shown in Fig. 3. This analysis indicate that α phase starts to nucleate in Ti-15Mo after ageing for 94 minutes at 450 °C (the ageing time depends obviously on utilized heating rate). The saturated value of volume fraction of α was determined to be 51 %. After 8 and additional 11 hours (i.e. 19 in total) of ageing the volume fraction of alpha should be 40 % and 50 %, respectively. α phase grows at the expense of both β and ω phases, with the decrease of volume fraction of ω phase being slightly faster.

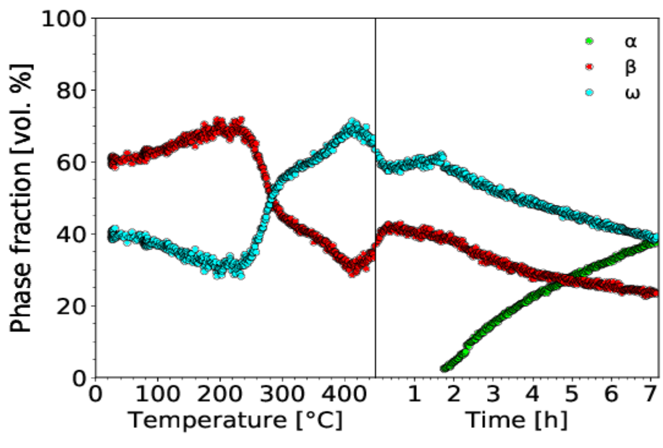


Figure 2: The evolution of volume fractions of α, β and ω phases during heating with the heating rate of 1.9 °C/min up to 450 °C followed by ageing for 7 h.

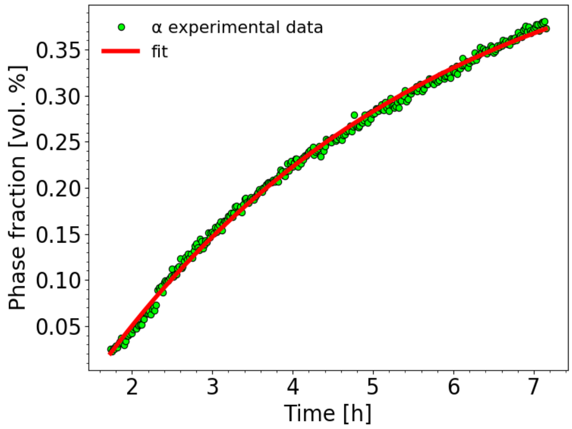


Figure 3: The comparison of measured volume fraction of α phase with Avrami equation with fitted parameters.

References

1. Avrami, M. Kinetics of Phase Change. II Transformation-Time Relations for Random Distribution of Nuclei. *J. Chem. Phys.* **8**, 212–224 (1940).
2. Christian, J. W. CHAPTER 12 - Formal Theory of Transformation Kinetics. in *The Theory of Transformations in Metals and Alloys* (ed. Christian, J. W.) 529–552 (Pergamon, 2002). doi:10.1016/B978-008044019-4/50016-7