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On the complementarity between resistivity measurement and ultrasonic measurement for in-situ characterization of phase transitions in Ti-alloys

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Abstract

We present the results of in-situ characterization of the phase transitions in metastable β-Ti alloy Ti5553 by contactless laser-based resonant ultrasound spectroscopy method and electrical resistance measurement in a four probe configuration. Phase transformations were studied during continuous heating from the room temperature to 700 °C with various heating rates. We showed that both methods provide complementary results and can be successfully used for observation of phase transitions in metastable β-Ti alloys.

Keywords:
Metals and alloys, Elasticity, Ultrasonics, Phase transitions

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1. Introduction

Mechanical properties of metastable β-Ti alloys are strongly dependent on the microstructure which in turn arises from different thermomechanical treatment. High variability of possible microstructure and low density result in a wide applicability of this class of alloys, ranging from orthopedic implants to the structural parts of aircrafts [1]. For this reason, it is crucial to have a reliable technique for the in-situ characterization of phase transitions and their kinetics in the material during thermo-mechanical treatment.

This study is focused on the Ti5553 alloy which belongs to the class of metastable β-Ti alloys and exhibits various solid-solid phase transformations. Nano-sized particles of athermal ω phase (ω_{ath}) form by diffusionless shuffle transformation during quenching from the temperatures above β-transus temperature. Upon subsequent heating, the particles of isothermal (ω_{iso}) phase are chemically stabilized by the diffusion of β-stabilizing elements into the surrounding matrix [2]. At higher temperatures, ω_{iso} particles serve as nucleation sites for precipitation of finely dispersed particles of α phase [3, 4, 5, 6]. The creation of α phase can be preceded by formation of orthorhombic α'' phase which existence was also observed in Ti5553 alloy [7, 8]. During further heating, the α phase gradually dissolves and the whole material is transformed into pure β phase at temperatures above β-transus temperature.

Measurement of electrical resistance is one of the most frequently used methods for in-situ observation of phase transformations in metastable β-Ti alloys. This method is well established for the study of kinetics of diffusion-driven formation of ω_{iso} particles [9] and it is successfully used for investigation of phase transitions during continuous heating of Ti15Mo [10], TIMETAL LCB [11, 12], Ti12Mo [13], β-Cez alloy [14] and Ti6Mo5Ta4Fe [15].

Another techniques used for in-situ study of phase transitions are small angle neutron scattering [16] or high energy synchrotron x-ray diffraction (SXRD) [17, 18, 19]. The advantage of the SXRD method is the possibility of evaluation of volume fractions of individual phases. Bruneseaux et al. [20] and Settefrati et al. [7, 21] proved a good complementarity of electrical resistance and SXRD measurement of transformation kinetics.

Further options of in-situ techniques are dilatometry [13, 22] or differential scanning calorimetry [23]. The in-situ techniques can be complemented by post-mortem investigation such as metallography or hardness measurement to obtain a deeper insight into the mechanisms of phase transformations.

The aim of this study is to compare the applicability of electrical resistance measurement with resonant ultrasound spectroscopy (RUS) [24, 25] for investigation of phase transformations kinetics in a selected metastable β-Ti alloy. Recently, RUS was utilized for in-situ observation of the influence of ω phase on the elasticity of TIMETAL LCB alloy [26]. It was shown that the high sensitivity of this method enables examination of formation and growth of ω_{ath} particles that is quite difficult to observe by previously mentioned conventional techniques. Furthermore, post-mortem study by RUS of TIMETAL LCB and Ti15Mo alloys after plastic deformation by high pressure torsion proved the occurrence of deformation-induced ω phase [27].

2. Material and methods

The experiments were carried out on the titanium alloy Ti5553 (Ti-5Al-5Mo-5V-3Cr in wt. %). The alloy was produced by VSPMO and provided to Institut Jean Lamour within the PROMITI project. The β-transus temperature of this alloy was 845 °C [21]. The alloy in the as-received state had a duplex microstructure – i. e. consisting of equiaxed α-particles (so-called primary α) and a mixture of lamellar α plates surrounded by some remaining β matrix. Afterwards, two initial states of microstructure were prepared. The specimens were either β solution treated at 890 °C or heat treated at 800 °C (below the β-transus temperature). In both cases, the treatment was terminated after 30 min by quenching to room temperature in helium gas. After the solution treatment, the microstructure at room temperature consisted of metastable β-matrix with finely dispersed particles of ω_{ath} particles. The heat treatment below the β transus resulted in 15 % of equiaxed α phase and only 85 % of metastable β phase [7].

Samples for RUS and electrical resistance measurements were prepared from both above described initial states of the material. Samples for RUS measurement, one from each initial condition, had a cylindrical shape with 4 mm diameter and 2.23 mm height. Four samples from the '890 °C/30 min' state and one from the '800 °C/30 min' state were prepared for electrical resistance measurement. They had also cylindrical shapes with 4 mm diameter and 40 mm height. All samples were subsequently studied in-situ by the two aforementioned methods in the temperature range from room temperature to 700 °C.

The temperature evolution of resonant spectra of samples was recorded by RUS using fully contactless laser-based RUS set-up described in detail

in [28]. The vibrations of the examined sample were generated by focused laser pulses and the modal response was detected by a laser vibrometer. The measured specimen was placed in a temperature chamber filled with low-pressure nitrogen atmosphere that enabled its temperature control. The average heating rate of both samples, '890 °C/30 min' and '800 °C/30 min', was 1.9 °C/min.

As discussed in [29], RUS resonant spectra carry mainly information on shear elastic constants (i.e. shear modulus G in case of isotropic material). Therefore, the elastic constants related to longitudinal motion of the material (volumetric changes, unidirectional tension/compression) can't be determined from resonant spectra with sufficient accuracy. For this reason, only temperature dependences of shear modulus G were evaluated from the measured resonant spectra by iterative inverse procedure that is described in detail in [24, 30].

Electrical resistance of all samples was measured by an in-house built dilatometer using a four-point configuration that allows simultaneous measurement of voltage and electrical current. The temperature was controlled by S-thermocouple spot-welded on the surface of the specimen. For the samples '890 °C/30 min', four different heating rates were chosen 1.4 °C/min, 1.9 °C/min, 3.9 °C/min and 6 °C/min. In case of the sample '800 °C/30 min', the heating rate of 1.9 °C/min was used.

RUS and electrical resistance measurement can be expected to bring slightly different but mutually complementary information on the processes taking place in the material. While electrical resistance measurements are sensitive both to the phase composition of the material and to the number and the structure of interfaces due to scattering of conduction electrons, the RUS data can be assumed as dominantly sensitive only to the volume fractions of individual phases. Hence, RUS results may help to separate the effect of the phase composition and the effect of the interfaces for the electrical resistance measurements.

3. Results and discussion

If the thermal expansion of the sample is neglected, the relative electrical resistance R/R_0 is equal to the relative electrical resistivity ρ/ρ_0 ($\rho = (RS)/l$), where R and ρ are immediate resistance and resistivity, R_0 and ρ_0 are resistance and resistivity measured at initial temperature, S is the cross-sectional area of the specimen and l is the length of the specimen.

The temperature dependence of relative resistivity of samples '890 °C/30 min' (relative resistivity= $(\rho - \rho_0)/\rho_0$, where ρ_0 is the resistivity measured at initial temperature 66 °C) is shown in fig. 1. It is obvious that the shape of the relative resistivity curves is strongly dependent on the heating rate.

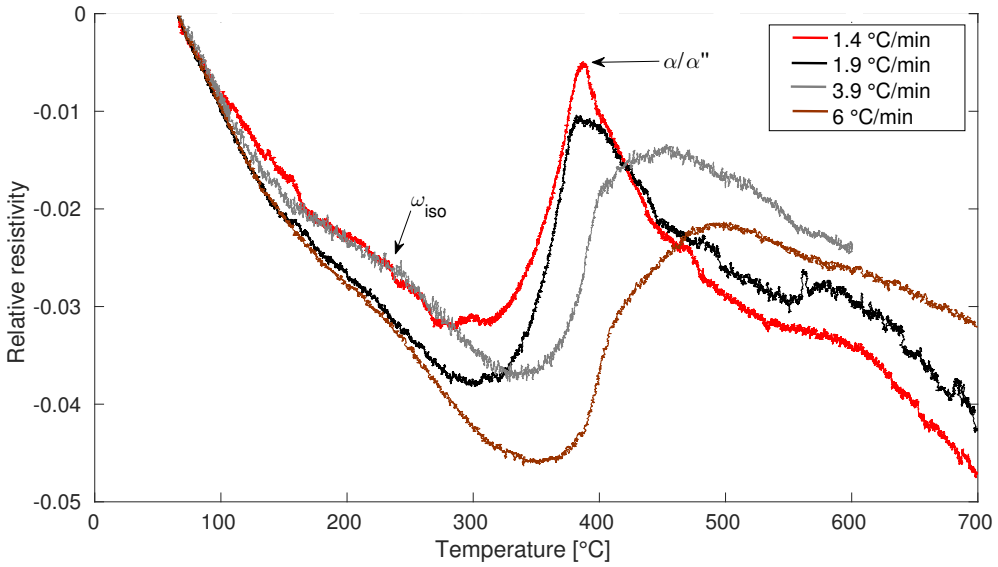

Figure 1: Temperature dependence of relative resistivity of samples '890 °C/30 min' during various heating rates – 1.4 °C/min, 1.9 °C/min, 3.9 °C/min and 6 °C/min. The peaks corresponding to formation of ω_{iso} and α/α'' phases are marked.

The decrease of relative electrical resistivity that is observed at the beginning of heating for all heating rates is attributed to the combination of anomalous behavior of β phase due to the phonon softening and vanishing of the particles of ω_{ath} phase [10, 12, 31, 32]. Reduction of β/ω_{ath} interfaces and related stress fields results in a drop of conduction electron scattering and therefore to the decline of relative resistivity.

The change of the slope of the decrease can be seen around 220 °C. The small peaks that are visible around this temperature for all heating rates are results of diffusion-driven formation of particles of ω_{iso} phase. As temperature increases, the precipitation of α/α'' phases occurs, while the ω_{iso} particles dissolve. The formation of α/α'' phase is visible on the relative resistivity curves as a sharp increase. However, both the position and shape of these peaks depend strongly on the heating rate; for the lowest heating

rate (1.4 °C/min), the peak is sharper and located to lower temperatures ($\sim$ 400 °C), while for the highest studied heating rate (6 °C/min), it is broader and located to higher temperatures ($\sim$ 500 °C). The difference in the position of this peak is clearly caused by the diffusion-driven mechanism of the formation of ω_{iso} and of α/α'' phases; i.e. at lower heating rates new phases have longer time for nucleation and growth.

Comparison of the evolution of resistivity and elasticity of sample '890 °C/30 min' is plotted in fig. 2. As it can be seen, variations of shear modulus present two distinctive peaks that are clearly associated with the formation of ω_{iso} phase and α/α''. The shear modulus increase is caused by the higher shear modulus of the ω_{iso} phase and α/α'' phase as compared to that of the β matrix, so their formation has well-detectable impact on the elastic properties of the whole material. The peaks of resistivity and shear modulus attributed to diffusional formation of ω_{iso} phase are approximately located at the same temperature. Nevertheless, the peak corresponding to α/α'' formation is shifted to a higher temperature by approximately 80 °C in RUS data. It can be observed that the temperature for which there is a peak of relative resistivity coincides with the largest variation of the shear modulus. This agrees well with the assumption that the relative resistivity evolution with temperature is dependent not only on the phase composition (i.e. volume fraction of the α/α'' phase), but also on the number and effective cross-section of the interfaces between the β-matrix and the α particles, as these parameters are probably the highest somewhere in the middle of the transition process.

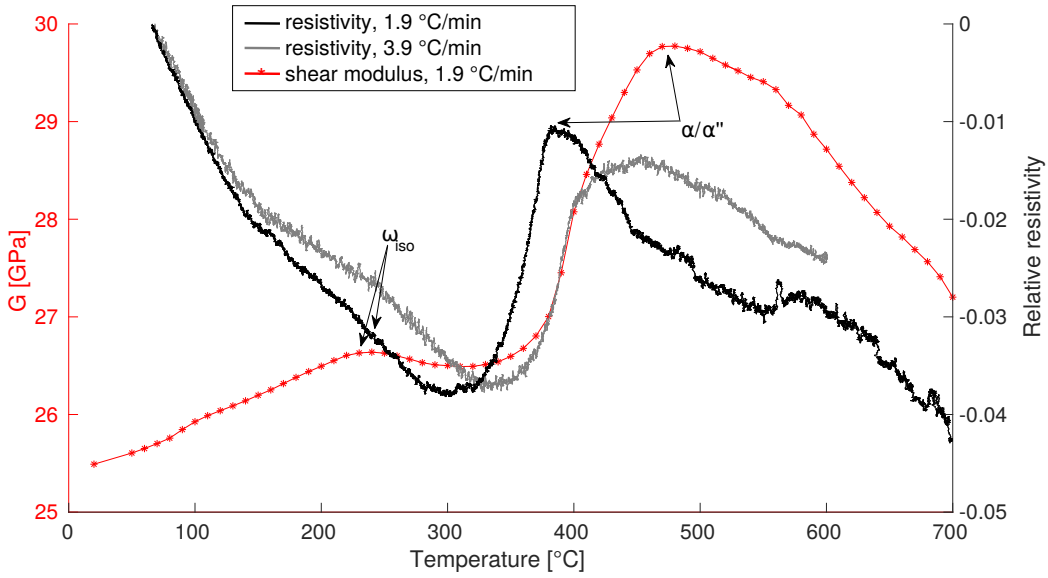

Figure 2: Comparison of relative resistivity (black curve) and shear modulus G of samples '890 °C/30 min' during heating with heating rate 1.9 °C/min. For the purpose of comparison, the relative resistivity (grey curve) of the sample '890 °C/30 min' with heating rate 3.9 °C/min is also plotted.

However, some part of this discrepancy could be also explained by the different way of temperature regulation of the experimental techniques. The temperature during electrical resistance measurement was measured by a thermocouple that was directly spot-welded on the specimen surface. The temperature was controlled by a PID regulator with four halogen lamps that were source of thermal radiation. This way of temperature regulation enabled rapid and precise control of temperature. As the loop time was equal to 10 ms, the time evolution of temperature during resistivity measurement was nearly linear. In contrast, the temperature of the sample during the RUS measurement was regulated via the nitrogen atmosphere by thermal conduction. This manner of temperature control was slower and the used heating rate of 1.9 °C/min was in fact an average rate; during the RUS measurement the dependence of temperature had a 'staircase' shape (heating to desired temperature and stabilization at this temperature). Formation of ω_{iso} and α/α'' phases could be very sensitive to this difference because, as was shown in fig. 1, even a slight change in the heating rate can significantly influence the kinetics of their formation. Such an interpretation is supported

by the fact that the location of the shear modulus peak for α/α'' phases from RUS measurements corresponds well to the resistivity peak for 3.9 °C/min, as is also shown in fig. 2. Nevertheless, in order to reliably separate the effect of the phase composition and the effect of the interfaces, it would be necessary to accompany the RUS and electrical resistance measurement by in-situ observations of the microstructure.

The complementarity of the RUS and electrical resistance measurement was also confirmed in case of sample '800 °C/30 min' (see fig. 3). The $\beta \rightarrow \omega$ and $\beta \rightarrow \alpha/\alpha''$ phase transformations were successfully detected by both methods. In the material heat treated under the β-transus temperature, the formation of ω_{iso} phase and α/α'' phase takes place at higher temperatures in comparison with the '890 °C/30 min' sample that initially contained only β and ω_{ath} phases. Similarly to sample '890 °C/30 min', peaks of relative resistivity and shear modulus corresponding to α/α'' do not coincide (the difference is roughly 40 °C). Moreover, the shear modulus of the sample '800 °C/30 min' in the initial state at room temperature is higher in comparison to the sample '890 °C/30 min' due to the fraction of α phase that causes stiffening of the whole material.

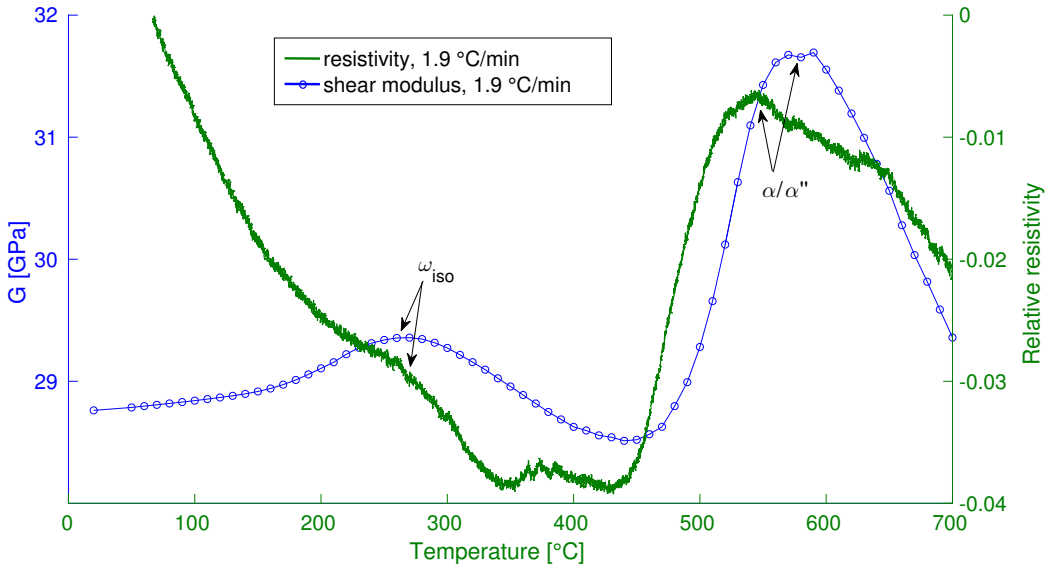

Figure 3: Evolution of relative resistivity and shear modulus G of samples '800 °C/30 min' during heating with heating rate 1.9 °C/min.

4. Conclusions

In summary, the results prove the complementarity of two experimental methods – resonant ultrasound spectroscopy and electrical resistance measurement. These methods were successfully utilized for the in-situ characterization of phase transformations of metastable β-Ti alloy Ti5553 during continuous heating from room temperature to 700 °C. It was shown that the phase transitions in this alloy are strongly dependent on the heating rate. The temperature of $\beta \rightarrow \omega$ phase transformation determined by both methods roughly correspond. In case of the temperature of $\beta \rightarrow \alpha/\alpha''$ transformation, the shift of peaks was observed which can be explained by the difference in temperature regulation or by the different sensitivity to the composition and effective cross-section of interfaces.

5. Acknowledgments

The authors wish to dedicate this paper to Michal Landa our colleague and friend who has passed away recently.

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- Phase transitions of Ti5553 alloy were studied in-situ.
- RUS and electrical resistance measurement are sensitive to structural processes.
- Both methods provide complementary results.
- Phase transitions in Ti5553 alloy are strongly dependent on heating rate.