In-situ characterization of growth of isothermal ω phase in metastable β-Ti alloy TIMETAL LCB

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Abstract

Metastable β-Ti alloys exhibit various solid-solid phase transitions. Our study is focused on the characterization of the diffusion controlled $\beta \rightarrow \omega_{iso}$ phase transition. The particles of ω phase play an important part in thermomechanical treatment since they serve as heterogeneous nucleation sites for precipitation of finely dispersed particles of hexagonal α phase. The in-situ observation of the growth of particles of ω phase could be difficult by conventional techniques. However, it was shown recently that the ω phase significantly influences the elastic constants of the material, and the different forms of ω phase have different effects on the elastic anisotropy, as well as on the internal friction coefficients. Therefore, the $\beta \rightarrow \omega$ phase transformation could be in-situ observed by the precise measurement of the tensor of elastic constants. In this contribution, we present the study of the kinetics of the $\beta \rightarrow \omega_{iso}$ phase transformation by resonant ultrasound spectroscopy. The polycrystalline samples of TIMETAL LCB alloy were in-situ examined by this technique during isothermal and non-isothermal ageing at temperatures up to 300 °C.

1. Introduction

Metastable β-titanium alloys are an important class of alloys that have found use in demanding applications such as aircraft structures, engines, orthopaedic and orthodontic implants. Their high strength and, simultaneously, low Young's modulus, good corrosion resistance, excellent biocompatibility and ease of fabrication provide significant advantages compared to other high performance alloys [1]. The metastable bcc β phase decomposes to form equilibrium hcp α phase precipitates.

Intermediate metastable phases, such as the ω_{iso} phase, form first and serve as heterogeneous nucleation sites for the equilibrium α phase.

Elastic properties (such as the low Young's modulus) of metastable β titanium alloys are strongly affected by the formation of elastically stiffer ω_{iso} particles in the original β–matrix [2]. As shown in [3], the Young's modulus of pure α titanium in some crystallographic directions can be as high as E = 220 GPa, and thus, even small volume fractions of ω particles can cause a significant increase in the modulus of the alloy. Formation of ω phase particles in metastable β–Ti alloys is a thoroughly studied phenomenon [4, 5]. However, the in-situ observation of the growth of particles of ω phase could be difficult by conventional techniques. This work presents the study of the kinetics of the $\beta \rightarrow \omega_{iso}$ phase transformation by resonant ultrasound spectroscopy – i.e. the possibility to observe the ω phase formation through the measurement of evolution of elasticity.

2. Material and experiments

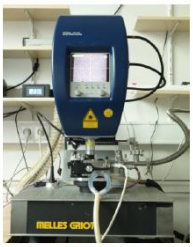
Four polycrystalline specimens of a metastable β-Ti alloy TIMETAL LCB (Ti-6.8Mo-4.5Fe-1.5Al in wt. %) were examined within this work. All of them were solution treated at 860 °C/4 h followed by water quenching. For samples details see table 1.

Table 1: Dimensions, density and velocity of quasi-longitudinal wave measured in z direction of the sample, all polycrystalline samples were considered as isotropic.

specimen	x [mm]	y [mm]	z [mm]	rho [g/cm3]	vqL (z) [m/ms]	Crystallographic symmetry
A	2.590	2.270	0.840	4.7	5.6	isotropic
B	2.630	2.350	0.750	4.7	5.6	isotropic
C	4.120	3.215	0.578	4.7	5.6	isotropic
D	4.127	3.225	0.479	4.7	5.6	isotropic

Elastic coefficients of the samples were determined by resonant ultrasound spectroscopy (RUS) [7]. The contactless experimental setup (see figure 1) is described in detail in [6]. RUS method is based on the fact that free vibrations of specimen contain information on elasticity of the material, which is represented by resonant frequencies of the specimen and corresponding modal shapes. From crystallographic orientation, density, dimensions and the first guess of elastic coefficients, it is possible to calculate resonant frequencies of the specimen. The complete set of sought elastic coefficient is obtained by minimization of the misfit between corresponding experimental and computed resonant frequencies (inversion procedure). The RUS method may be beneficially complemented by measuring the velocities of propagation of longitudinal waves in

directions perpendicular to the faces of the sample, and by involving these velocities into the inversion procedure. This combination of RUS and pulse-echo method, reported in [6], provides reliable results for any anisotropic material of arbitrary symmetry class.



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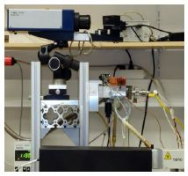

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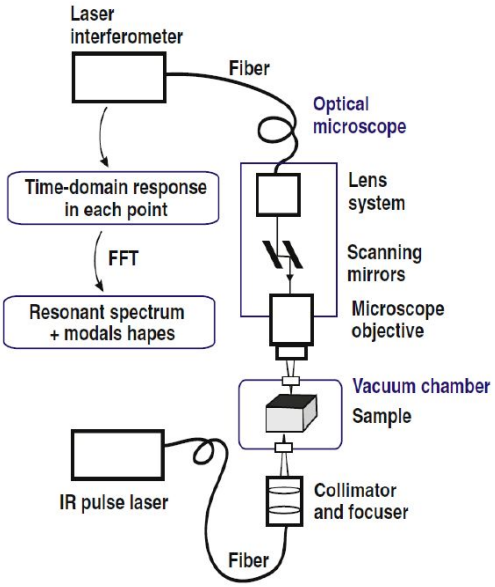

Figure 1: Schematic representation of the experimental part of Resonant Ultrasound Spectroscopy method.

Resonant spectra of free elastic vibrations of the samples were recorded in the frequency range from 0.2 MHz to 2 MHz, which covered sufficient number of resonant peaks for each sample. The symmetry of all samples was considered as isotropic with 2 independent elastic coefficients. The samples were first measured at room temperature in order to obtain resonant spectrum and corresponding modal shapes. During in-situ measurement, only resonant spectra at each temperature were detected and the evolution of elasticity was determined from shifts of corresponding resonant frequencies.

The temperature-time profile of in-situ measurement of all samples is shown in figure 2.

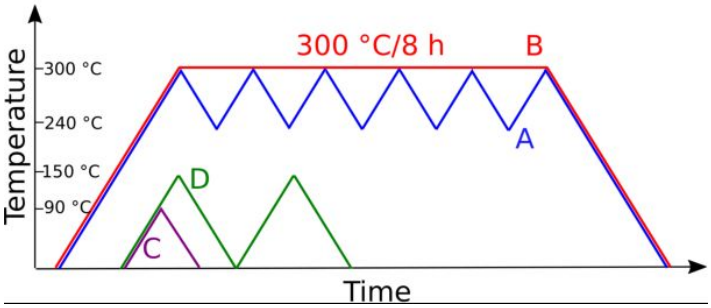

Figure 2: Temperature-time profile of all measurements.

One temperature cycle from room temperature to 90 °C was performed on sample C, two temperature cycles to 150°C were performed on sample D. Average heating/cooling rate was 1.4 °C/min. Determination of the reversibility/irreversibility of the microstructural processes taking place in the material was the aim of this in-situ measurement.

Afterwards, the specimen B was heated to 300 °C with average heating rate 1.4 °C/min, then isothermally aged at 300 °C for 8 hours and then cooled with cooling rate 1.4 °C/min. The sample A was, instead of isothermal ageing, subjected to temperature cycling between 240 °C and 300 °C with average heating/cooling rate 1.4 °C/min. This comparative measurement was carried out to closely examine the kinetics of the $\beta \rightarrow \omega_{iso}$ phase transformation.

3. Results and discussion

Elastic coefficients measured at room temperature of solution treated examined samples of TIMETAL LCB can be seen in table 2. The elastic constants of all samples are almost equal (considering the experimental error).

Table 2: Elastic coefficients of polycrystalline specimens of TIMETAL LCB at room temperature.

specimen	**C_{11} [GPa]**	**G [GPa]**
A	147.3 ± 1.0	29.9 ± 0.1
B	147.4 ± 1.0	29.8 ± 0.1
C	148.5 ± 1.0	29.3 ± 0.1
D	148.4 ± 1.0	29.5 ± 0.1

The solution treated samples were afterwards subjected to in-situ RUS measurement during thermal treatment described in section 2. Growth of ω phase causes the isotropisation and stiffening of the β-matrix as shown in [2]. Also, it was proved that long-term ageing of solution treated single crystals of TIMETAL LCB alloy at 300 °C does not cause any formation of α phase. Therefore, all changes of elasticity measured in this study could be attributed only to the changes of ratio between volume fractions of β and ω phases, or the evolution of the elasticity of these phases with temperature. In the case of in-situ measurement, only shear modulus was evaluated, since the resonant spectra carry mainly information on shear elastic constants and it is not possible to assess the value C_{11} with sufficient accuracy [6].

The temperature dependences of shear modulus *G* of samples C and D is shown in figure 3. While the behaviour of sample C during temperature cycle to 90 °C (fig. 3a) is linear and fully reversible (the difference between values measured during heating and cooling run is below the sensitivity of RUS measurement), heating to 150 °C causes the irreversible behaviour of sample D in both cycles (fig. 3b).

This indicates that there is a disappearance of ω_{ath} particles during heating and their reversible formation during cooling from 90 °C in case of sample C. In contrast, heating to 150 °C of sample D results in the diffusion controlled growth of ω_{iso} particles during which the rejection of β stabilizing elements occurs out of the ω phase particles to the surrounding β matrix [5]. This phase transformation induces elastic stiffening of the examined sample. Thus, it is apparent that heating to 150 °C is above the threshold of formation of ω_{iso} in polycrystalline TIMETAL LCB.

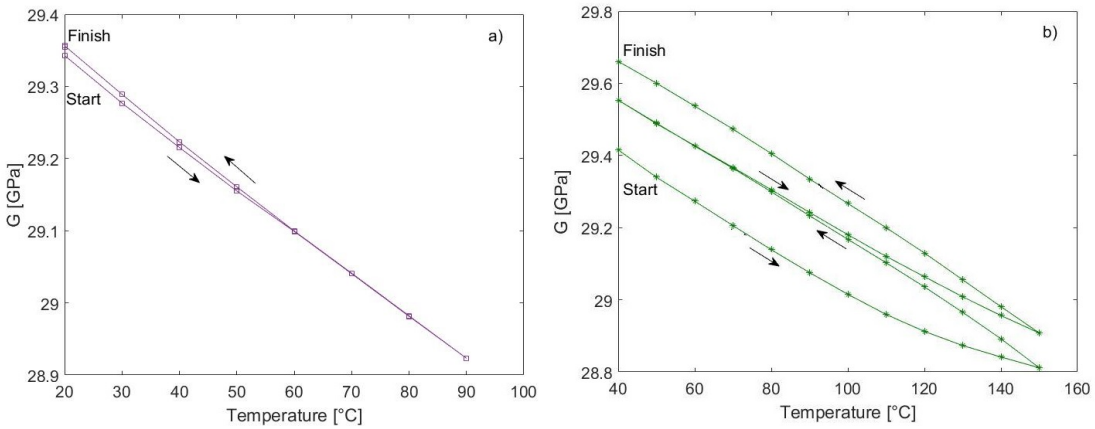

Figure 3: Temperature evolution of *G* a) of sample C during ageing to 90 °C, b) of sample D during ageing to 150 °C.

In order to examine the kinetics of β→ω_{iso} phase transformation, the isothermal ageing at 300 °C and temperature cycling between 240 °C at 300 °C were performed on samples A and B. The comparison of temperature and time evolution of shear modulus *G* is shown in figure 4. The growth of shear modulus during initial heating (fig. 4a) is the same for both samples. As expected, the overall increase of elasticity after heat treatment is slightly higher in case of isothermally aged sample B (which rose by 17.4 GPa) in comparison with non-isothermally aged sample A (which rose by 14.3 GPa). That is predictable because

higher temperature induces more intensive diffusion-controlled growth of the ω_{iso} particles. Concerning the temperature cycling of sample A, the most intensive rise of shear modulus takes place during the first cycle. The following cycles vary in the total increase of the shear modulus that becomes smaller with the number of the cycle; there is a visible gradual saturation in the following cycles. Fig. 4b shows the evolution of shear modulus with time during isothermal ageing at 300 °C of sample B and during cycling part of ageing of specimen A. The time dependence of shear modulus of sample B during isothermal ageing confirms the above described result during temperature cycling. The most intensive increase of shear modulus takes place during the beginning of isothermal ageing, afterwards it changes much slower. This behaviour is caused by the rejection of β stabilizing elements out of the ω_{iso} particles, which leads to the saturation of the surrounding β matrix by these elements and therefore the growth of ω_{iso} particles gets slower.

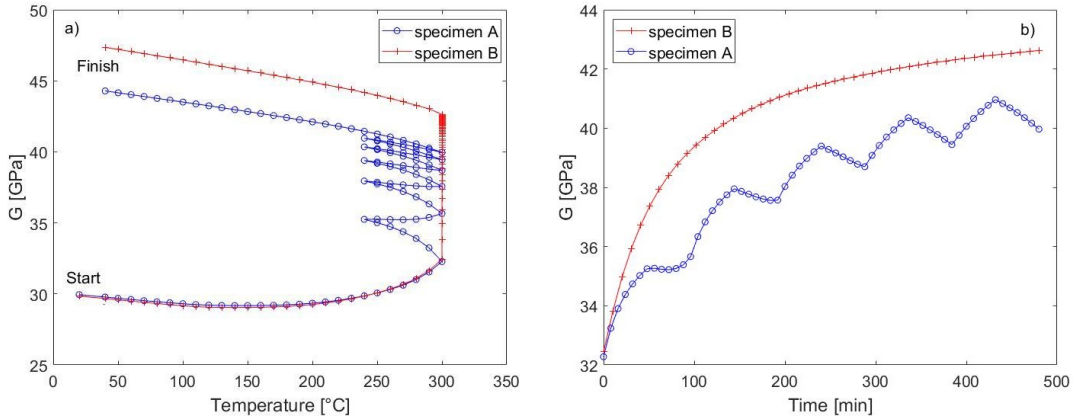

Figure 4: Temperature a) and time b) dependence of shear modulus *G* during non-isothermal (sample A) and isothermal (sample B) ageing.

4. Conclusion

The evolution of elasticity of polycrystalline specimens of TIMETAL LCB during isothermal and non-isothermal ageing was investigated using ultrasonic methods. The change in shear modulus is directly related to formation and growth of ω_{iso} phase, which starts, as our experiments indicate, between 150 and 180 °C. Formation of this new phase causes the stiffening of the β-matrix, which is observed as an increase of shear modulus with time and temperature. The overall increase of elasticity after heat treatment is about 18 % higher in case of isothermally aged sample B in comparison with non-isothermally aged sample A. Both isothermal and non-isothermal ageing prove a visible gradual saturation in ω_{iso} growth. The rejection of β stabilizing elements out of the ω_{iso} particles leads to the saturation of the surrounding β matrix by these elements, and therefore the growth of ω_{iso} particles gets slower. It was proved that ultrasonic methods are suitable tool for in-situ investigation of microstructural processes in metastable β-Ti alloys.

5. Acknowledgement

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6. References

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