

# Experimental report

17/06/2022

**Proposal:** 5-24-628

**Council:** 4/2019

**Title:** Phase transformation kinetics in metastable beta-Ti alloys studied by in situ neutron diffraction

**Research area:** Materials

**This proposal is a new proposal**

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**Samples:** Ti-15Mo  
Ti-6.8Mo-4.5Fe-1.5Al  
Ti-5Al-5Mo-5V-3Cr

Instrument	Requested days	Allocated days	From	To
D20	3	2	20/01/2020	22/01/2020

## Abstract:

Metastable beta-Ti alloys contain a sufficient amount of so called beta-stabilizing elements to retain the high-temperature beta phase upon quenching. Due to the metastable nature of the retained beta phase, a number of phase transformations can occur as the alloy reaches its thermodynamic equilibrium. The physical mechanisms of these transformations are quite complex, and some of their aspects have not yet been satisfactorily explained. The proposed project aims to study the kinetics of phase transformations occurring in three selected metastable beta titanium alloys during linear heating. In particular, the formation of metastable particles of omega and alpha'' phases and their subsequent transformation to thermodynamically stable alpha phase will be investigated. For this in situ experiment, the high-flux neutron diffraction instrument D20 at ILL is an ideal tool.

## Phase transformation kinetics in metastable beta-Ti alloys studied by in situ neutron diffraction

The aim of the research was to investigate phase transformation kinetics during linear heating in several metastable  $\beta$  titanium alloys: LCB, Ti-5553 and Beta-CEZ. The experiment was conducted at D20 using 1.54 Å neutrons. The initial conditions of the samples (rods with the diameter of 10 mm and the length of approximately 40 mm) were prepared by solution treatment above the  $\beta$ -transus of the given alloy followed by quenching in water. Two heating rates were employed, 5 °C/min and 1.4 °C/min. The maximum reached temperature was 850 °C – 950 °C, so that the  $\beta$ -transus of the given alloy was exceeded.

Examples of the temperature evolution of measured diffraction patterns are shown in figure 1. In the LCB alloy (left panel in Fig. 1), sharp peaks belonging to the  $\beta$  matrix are clearly visible at the beginning of heating (i.e. top of the image). Furthermore, weaker and wider peaks arising from athermal  $\omega$  particles formed during quenching can be observed. The  $\omega$  maxima sharpen and gain intensity with increasing temperature up to approximately 500 °C, where an abrupt dissolution of the  $\omega$  phase can be seen. From 500 °C, the evolution of thermodynamically stable  $\alpha$  phase is observed. Above the  $\beta$ -transus (approx. 790 °C), the material is composed of the  $\beta$  phase only. In addition to diffraction maxima arising from the materials itself, we detected additional peaks which do not change much during heating/cooling. These peaks arise from the thermocouple which accidentally intersected the beam.

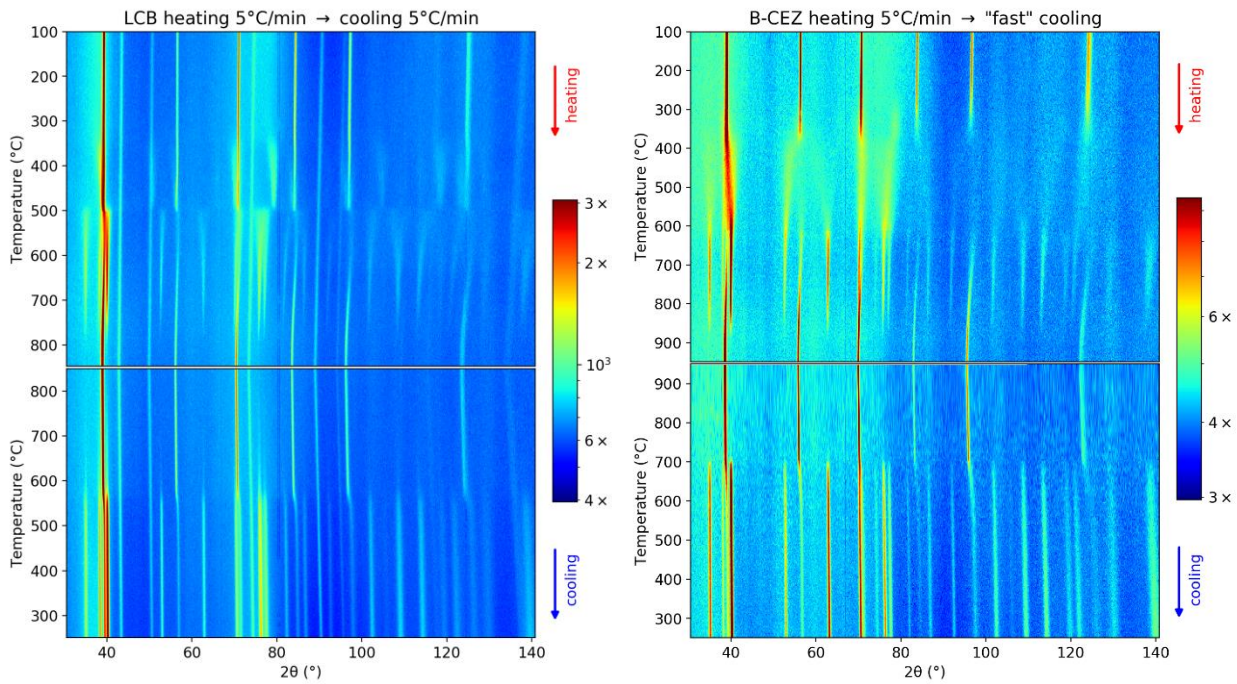


Figure 1: Evolution of neutron diffraction patterns with temperature during heating and subsequent cooling for LCB (left) and Beta-CEZ (right).

In the case of Beta-CEZ (right panel in Fig. 1),  $\omega$  maxima are relatively weaker in the initial stages of heating. There is no abrupt  $\omega$  dissolution but rather, the transformation  $\beta + \omega \rightarrow \beta + \alpha$  is more continuous. The  $\beta$ -transus was observed at approximately 890 °C.

The obtained neutron diffraction data were fitted in the FullProf software, which allowed us to identify the transformation sequences for all studied alloys, obtain temperature evolution of volume fractions and lattice parameters of individual phases.

The experiment provided us with invaluable data which contribute to our ongoing research of phase transformations and microstructure changes in titanium alloys. Neutron diffraction is complementary to our laboratory techniques and thanks to this experiment, we were able to obtain statistically relevant diffraction data from a large volume of polycrystalline samples.