



	<b>Experiment title:</b> <b>Precipitation mechanisms in near-beta titanium alloys. Influence of the thermal path and of chemical composition of the parent phase</b>	<b>Experiment number:</b> MA 865
<b>Beamline:</b> ID15b	<b>Date of experiment:</b> from: 17/02/2010 to: 23/02/2010	<b>Date of report:</b> 31/08/2010
<b>Shifts:</b> 18	<b>Local contact(s):</b> Jérôme Andrieux	<i>Received at ESRF</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> Elisabeth Aeby-Gautier SI2M IJL UMR CNRS 7198 NANCY UNIVERSITY Moukrane Dehmas SI2M IJL UMR CNRS 7198 NANCY UNIVERSITY Guillaume Geandier LETAM CNRS FRE 3143 METZ UNIVERSITY Amico Settefrati SI2M IJL UMR CNRS 7198 NANCY UNIVERSITY Mick��l Mourot SI2M IJL UMR CNRS 7198 NANCY UNIVERSITY		

## Report:

Our objectives were to realize in situ characterizations of precipitation in near beta titanium alloys during different thermal paths in order i) to increase our knowledge on the precipitation mechanisms at the lower temperatures of these alloys either during direct cooling or during aging, ii) to analyze the effect of the chemical composition of the beta-phase on the transformation process.

We focused our attention on precipitation in titanium alloys at the lower temperatures (300°C – 600°C), in order to precise the transformation sequences by analyzing the successive phases and to quantify the evolutions of the phase amounts and their cell parameters. These data give interesting information on the diffusive character (or not) of the transformation, necessary to know in order to model the transformation kinetics, as well as to characterize the development on internal stresses during the precipitation progress.

The experimental set up and the analysis of data are similar to the one described in [1,2]. The alloy studied is the near  $\beta$  titanium alloy Ti-5553. The nominal composition in weight percent is Ti-5Al-5Mo-5V-3Cr. The particularity of near beta titanium alloys is to be able to retain a full  $\beta$ -metastable structure after rapid cooling from the high temperature  $\beta$  phase field, avoiding  $\alpha$  precipitation. The  $\beta$ -metastable decomposition has been studied for different transformation paths, either during direct cooling or during further heating and aging in the  $\alpha+\beta$  temperature range. It must be emphasized that the whole thermal path determines the morphology and distribution of the  $\alpha$  precipitates, and thus their properties. Previous results were done in our laboratory using in situ electrical resistivity allowing to precise the transformation temperature ranges, and thus to define the best conditions that have to be applied on the experimental setup. In the following we develop some obtained results that have already been presented at PTM 2010 in Avignon (Solid Solid phase transformations) and are submitted for publication.

Specimens were previously heat treated at 890°C for 30min, i.e. at a temperature above the  $\beta$  transus temperature (847°C) and quenched to room temperature. The metastable  $\beta$ -phase was then heated on the experimental set up. Samples were then aged at 675°C for two hours using three different heating rates. Figure 1 shows the evolution of (I, 2 $\theta$ ) diffraction patterns for the three different heating rates. We demonstrate that the transformation sequence is function of the heating rate. For heating at 0.14°C/s (fig. 1a) the transformation sequence observed previously by Bruneseaux et al., is confirmed i.e. at first formation of hexagonal  $\omega$  phase

(between 350°C and 420°C) followed by formation of  $\alpha''$ , above 420°C and formation of  $\alpha$  above 540°C. The formation of  $\alpha''$  is poorly mentioned in the literature and is demonstrated here. For the higher heating rate,  $\omega_{iso}$  does not precipitate anymore and the first phase formed is the orthorhombic  $\alpha''$ .  $\alpha$  is observed at temperatures above 610°C. With a heating rate of about 1°C/s,  $\omega$  does not precipitate anymore and the first phase formed is the orthorhombic  $\alpha''$ . The hexagonal structure is observed at temperatures above 610°C. If higher heating rate is applied (fig. 1c), neither  $\omega$  nor  $\alpha''$  precipitation is observed. The presence of  $\alpha$  phase is evidenced at temperatures above 585°C. The transformation essentially occurs during the isothermal ageing at 675°C.

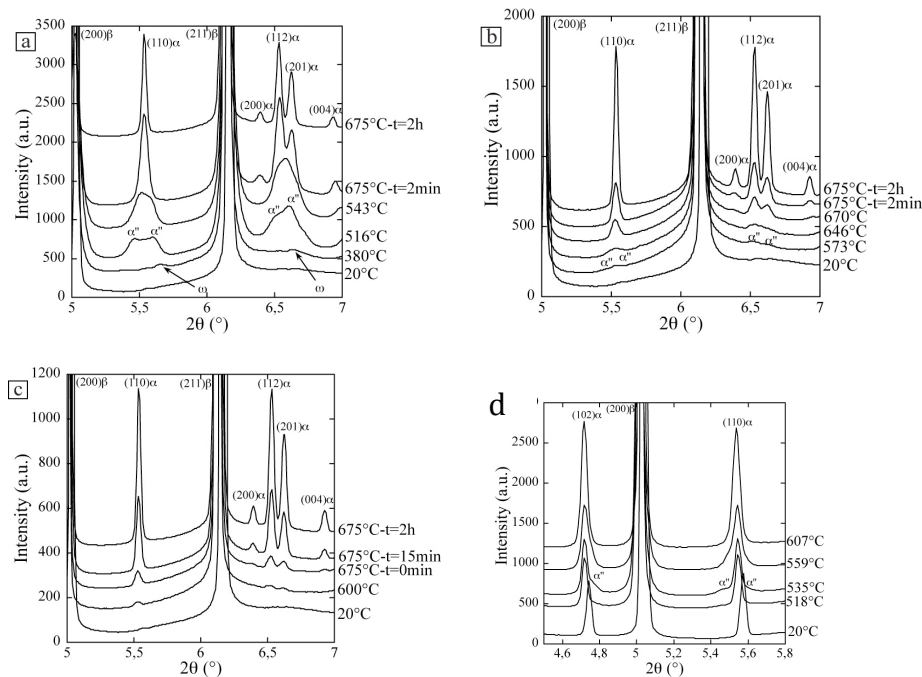


Fig. 1: Evolutions of the diffraction patterns obtained during heating to 675°C and during the isothermal step at this temperature for a 100% beta metastable initial state and heating rates of about (a) 0.14°C/s, (b) 1.4°C/s and (c) 3°C/s; (d) for a 85% beta metastable initial state and heating rates of about 0.14°C/s.

In addition, the decomposition of beta metastable phase was studied after a solution treatment in the high  $\alpha+\beta$  temperature range. This solution treatment leads to 85wt% of beta metastable phase, enriched in beta stabilizer elements as compared to 100%  $\beta$  metastable phase. During heating at 0.14°C/s (fig. 1d),  $\omega$  precipitation does not occur any more and we have a direct precipitation of  $\alpha''$  during heating.

The comparison of behavior observed for Ti 17 and Ti5553 shows that the critical heating rate to form  $\omega$  phase is dependent on the alloy composition.

[1] F. Bruneseaux, G. Geandier, E. Gautier, M. Dehmas and P. Boulet in: Ti-2007 Science and Technology, edited by M. Niinomi, S. Akiyama, M. Hagiwara, M. Ikeda and K. Maruyama, Kyoto, Vol. 3 (2007), pp. 563-566.

[2] F. Bruneseaux, E. Aeby-Gautier, G. Geandier, J. Da Costa Teixeira, B. Appolaire, P. Weisbecker, A. Mauro In situ characterizations of phase transformations kinetics in the Ti17 titanium alloy by electrical resistivity and high temperature synchrotron X-ray diffraction Materials Science & Engineering A 476 (2008) 60-68.

**Paper presented at PTM 2010 and submitted in Solid State phenomena:** Precipitation in near  $\beta$  titanium alloys on aging: Influence of heating rate and chemical composition of the metastable phase A. Settefrati, E. Gautier, M. Dehmas, G. Geandier, B. Appolaire, S. Audion, J. Delfosse.