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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Formation and growth of particles of alpha and omega phases in beta-Ti single crystals measured by fast in-situ reciprocal space mapping during heating	**Experiment number**: MA-3122
Beamline: ID11	**Date of experiment**: from: 20. 10. 2016 to: 25. 10. 2016	**Date of report**: 1. 3. 2017
Shifts: 15	**Local contact(s)**: Andrea Bernasconi	*Received at ESRF:*

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Report:

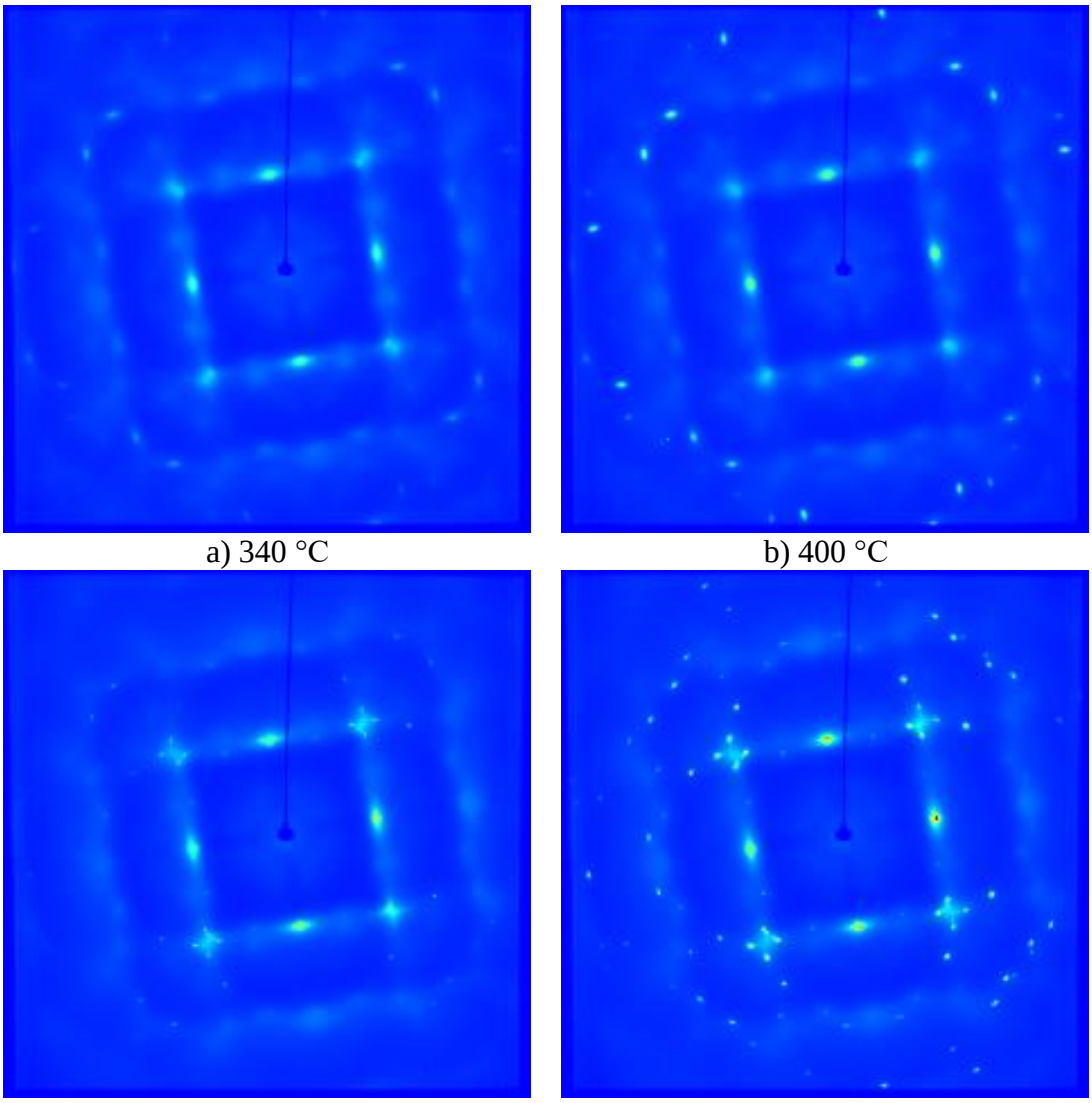
This research was conducted on a representative metastable β titanium alloy, Ti-15wt.%Mo. Metastable β titanium alloys are Ti alloys containing a sufficient amount of elements which stabilize the high-temperature β phase (body-centred cubic) to lower temperatures. More precisely, martensitic transformation to the low-temperature α phase (hexagonal close-packed) is suppressed below room temperature. In a certain composition range of this type of titanium alloys, small particles (a few nm in size) of metastable ω phase form in the alloy. ω particles have a significant impact on mechanical properties of the alloy (they increase its hardness) as well as on subsequent phase transformations occurring in the material (they serve as nucleation sites for thermodynamically stable α phase) [1]. ω particles are coherent with the β matrix and there are 4 possible crystallographic orientations of the ω lattice with respect to the β structure [2].

In our previous works [3-5] we studied the growth of ω particles by X-ray diffraction and in-situ small-angle X-ray scattering. We have demonstrated that the particles are self-arranged in a weakly ordered three-dimensional cubic array along <100> axes in the parent β phase and the driving force of the ordering is the elastic strain caused by local elastic strains due to an inhomogeneous distribution of β-stabilizing Mo atoms.

In this experiment, the mechanisms of formation and growth of particles of ω and α phase were studied employing reciprocal space mapping technique. The investigation was performed on Ti-15wt.%Mo single crystals grown in an optical floating zone furnace [6]. We investigated the evolution of ω and α particles in situ during linear heating and isothermal ageing in a dedicated microtomography furnace which could be evacuated up to 10^{-6} mbar. The experiment was done at ID11. Using a 2D detector, we were able to monitor diffraction spots arising from the parent β matrix and ω and α spots simultaneously.

Our initial objective was to detect maximally one β peak and one or two ω/α spots and from their evolution analyse the kinetics of secondary particles (ω and α) evolution. Due to these experimental requirements, we started the measurement in experimental hutch 3 (EH3) at ID11. Unfortunately, due to severe technical problems (safety door to EH3 fell out of its hinges), this initial aim could not be fulfilled. Nevertheless, we were able to continue our experiment with modified parameters in EH1. EH1 experimental setup did not

allow us to move the detector far enough from the sample and therefore, we observed a greater number of β, ω and α diffraction spots. Examples of 2D detector images acquired during linear heating to 800 °C are shown in Fig. 1. The diffraction patterns were obtained in transmission geometry with $[100]_\beta$ direction parallel to the primary beam. Fig. 1a) shows diffraction pattern at a relatively low temperature, at which the material consists of β matrix and very small ω particles formed during quenching of the alloy. In Fig. 1b), sharpening of ω spots can be observed. In this temperature range, ω particles grow by diffusion controlled mechanism, ejecting β stabilizing elements (Mo) into the surrounding β matrix. Fig. 1c) shows a pattern at a temperature at which all ω particles dissolved and α phase particles started to precipitate. Finally, Fig. 1d) shows well-developed strong α spots, suggesting relatively high volume fraction of the α phase.



a) 340 °C b) 400 °C

c) 620 °C d) 760 °C

Fig. 1 – Examples of diffraction patterns obtained during linear heating from room temperature to 800 °C, heating rate of 5 °C/min. a) Characteristic $(100)_\beta$ pattern with very weak ω spots; b) with increasing temperature, ω diffraction spots are becoming more intense; c) above a certain temperature, ω spots disappear and weak α reflections form; d) at high temperatures, strong and sharp α spots are observed.

[1] F. Prima et al., Scripta Materialia **54**, 645 (2006).
[2] D. De Fontaine, Acta Metall. **18**, 275 (1970).
[3] J. Šmilauerová et al., Acta Mater. **61**, 6635 (2013).
[4] J. Šmilauerová et al., Acta Mater. **81**, 71 (2014).
[5] J. Šmilauerová et al., Acta Mater. **100**, 126 (2015).
[6] J. Šmilauerová et al., J. Cryst. Growth **405**, 92 (2014).