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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.

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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:** Anomalous diffraction and DAFS from particles of alpha and omega phases in beta-Ti(Mo) single crystals	**Experiment number**: MA2605
Beamline: BM02	**Date of experiment**: from: 8.7.2015 to: 14.7.2015	**Date of report**: 5.8.2015
Shifts: 18	**Local contact(s)**: Hubert Renevier	*Received at ESRF:*

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Report:

Metastable β titanium alloys are important materials for various construction purposes, as well as for biomedical applications. Metastable β titanium alloys contain enough β stabilizing elements to suppress martensitic transformation from body-centered cubic β phase to hexagonal close-packed α phase upon quenching to room temperature. However, due to the metastable nature of the β phase, various other phases can form during ageing at elevated temperatures. In a certain composition range, nm-sized particles of metastable ω phase form upon quenching in the β matrix by a diffusionless displacive transformation [1]. When exposed to elevated temperatures, ω particles grow by a diffusion-assisted mechanism. The ω phase particles play a significant role in alloy hardening, as well as in subsequent phase transformations, i.e. ω-assisted nucleation of thermodynamically stable α phase [2].

In our previous works [3-5] we studied the growth of the ω nanoparticles 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.

The task of the beamtime was to determine directly the local concentration of Mo in Ti(Mo) alloys by anomalous x-ray diffraction (AXRD) and DAFS. The experiments have been performed on a series of Ti - 8.1 Mo (in at. %) single crystals grown from the melt in an optical furnace (see [6] for technological details). Using a two-dimensional detector we measured the reciprocal-space distribution of the diffracted intensity for various energies around the MoK absorption edge (20.0 keV); the measurements have been carried out both in the 006 maximum of the body-centered cubic β-Ti phase and in the maximum 44-82 of the hexagonal ω phase. In addition, we have measured the energy dependence of the MoKα fluorescence signal.

Examples of experimental results are presented in Fig. 1. Panel (a) shows the intensity distribution in the detector plane around the 006 β-Ti maximum (the sharp peak in the center of the figure). It is obvious that the ω nanoparticles give rise to diffuse x-ray scattering; we have shown in [4] that the scattered intensity can be

described by the well-known Huang model. Panel (c) shows the 44-82 maximum of the ω phase; the shape of the maximum depends mainly on the shape of the ω nanoparticles. We have chosen several regions of interest (ROI) in the detector plane (denoted by white and black boxes in panels (a) and (c)) and collected the total intensities in the ROIs as functions of the photon energy (the AXRD curves in panels (b) and (d) for diffractions 006β and 44-82ω, respectively).

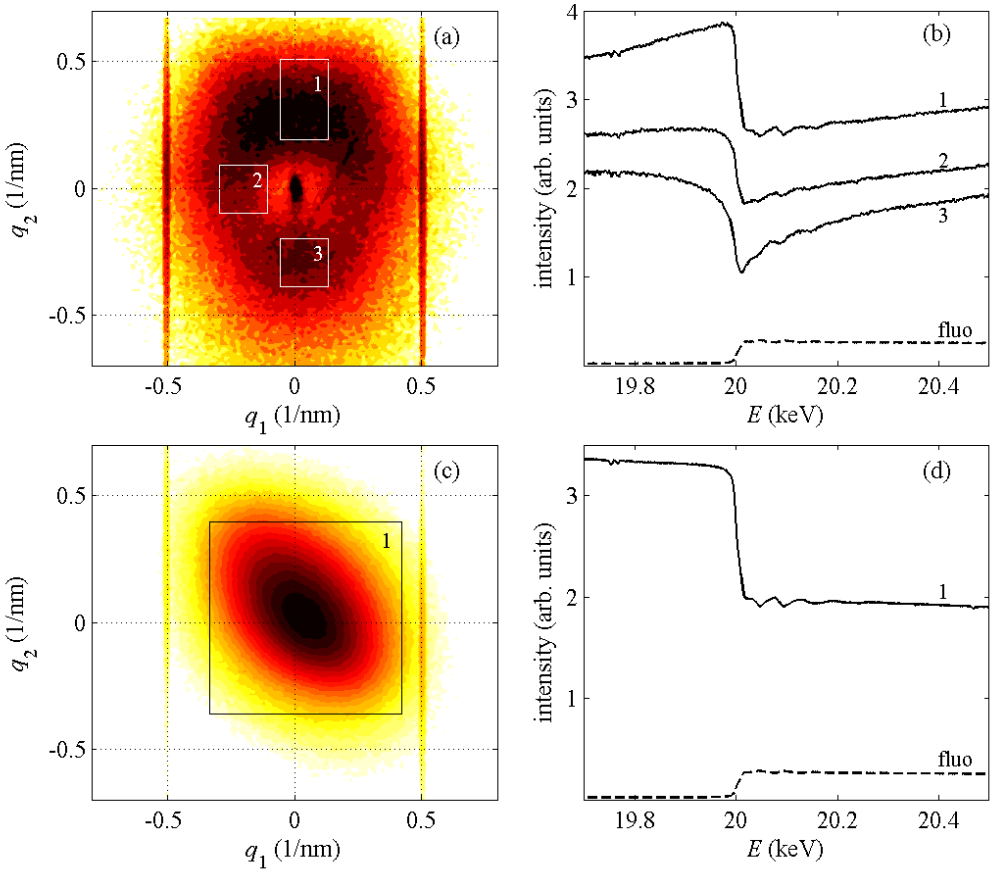

Fig. 1. Reciprocal-space maps in diffractions 006β (a) and 44-82ω (c) and the energy dependences (panels (b) and (d)) of the intensities collected in the ROIs indicated in panels (a) and (c); sample Ti+Mo(8%at) after annealing at 370°C for 512 h; the curves are shifted vertically for clarity. The energy dependence of MoKα fluorescence is denoted by dashed lines.

From Fig. 1(b) it is obvious that the AXRD curves of different ROIs are different. This fact can be ascribed to different Mo concentrations in the iso-strain volumes around a ω nanoparticle, which contribute to the particular ROIs. From a self-consistent simulation of the Huang scattering from the particles based on the calculation of local elastic strain fields for a given Mo distribution, combined with the simulation of the AXRD effect it will be possible do determine the local distribution of Mo around a particle. This could confirm our hypothesis about strain mediated self-ordering of the nanoparticles. The AXRD curve of the maximum 44-82ω in Fig. 1(d) is affected mainly by absorption in the β-Ti(Mo) matrix. Nevertheless, from a detailed simulation it will be possible to determine the Mo concentration in the ω nanoparticles and confirm the model, in which the Mo atoms are expelled from the growing nanoparticles [5].
The AXRD curves both in the β and ω maxima exhibit well-pronounced DAFS oscillations, from which it will be possible to determine the lattice positions of Mo atoms in both phases and the local distortion of first and second coordination spheres around a Mo atom.

[1] D. De Fontaine, Acta Metall. **18**, 275 (1970).
[2] F. Prima et al., Scripta Materialia **54**, 645 (2006).
[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. accepted (2015).
[6] J. Šmilauerová et al., J. Cryst. Growth **405**, 92 (2014).