EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** The evolution of deformed microstructure of Al during annealing: a generalisation of 3DXRD	**Experiment number**: MA 3920
Beamline: ID11	**Date of experiment**: from: 18/04/2018 to: 23/04/2018	**Date of report**: August 14th, 2020 *Received at ESRF:*
Shifts: 15	**Local contact(s)**: Marta Majkut, Jonathan Wright	

Names and affiliations of applicants (* indicates experimentalists):

H.F. Poulsen[1,*], C. Detlefs[2,*], M. Kutsal[1,2,*], Y. Zhang[3]

[1] Department of Physics, Technical University of Denmark, 2800 Kgs. Lyngby, Denmark

[2] European Synchrotron Radiation Facility, 71 avenue des Martyrs, CS40220, 38043 Grenoble Cedex 9, France

[3] Department of Mechanical Engineering, Technical University of Denmark, 2800 Kgs. Lyngby, Denmark

Report:

In this beamtime, we carried out the first implementation of a new high resolution modality of 3DXRD microscopy[1], HR-3DXRD on 50% recrystallized Al 1050 and eutectic high entropy alloy (HEA). The experiments were carried out at 35 keV, selected with Si 111 bent Laue double crystal monohromator ($\Delta E/E \sim 10^{-3}$). With the help of local contact M. Majkut, the experimental setup was prepared by aligning the condenser optics and the detector configuration. The monochromatic incident beam was collimated by KB mirrors by overfocusing in horizontal direction and focusing on the sample in vertical direction. Such alignment optimized the attainable incident flux in the given energy range. The diffraction experiments were carried out with a FReLoN camera (Kodak chip) with a native pixel size of 24 µm. The effective pixel size of the camera was selected with the available x4 and x10 optical objectives and a fixed x3.1 eye piece to 1.94 and 0.77 µm. For this experiments, we've analyzed two samples: (1) Commercially pure 50% recyrstallized Al1050 alloy having large recrystallized grains (~10-50 µm) and small deformed grains (~0.7-1 µm) [2–4],and (2) a novel eutectic HEA alloy, $AlCoCrFeNi_{2.1}$, with an average grain size of ~0.4 µm [5,6]. The sample to detector distance was chosen as 7.3 mm.

The HR-3DXRD method is proposed to achieve a higher resolution by optimizing the sample to detector distance and pixel size of the detector. By placing the detector in between the near-field and far-field regimes and using a high resolution imaging detector, Debye-Scherrer rings are captured partially in detector's field of view. Thus, HR-3DXRD can be realized via translating the detector in its y-z plane (assuming x-axis for X-rays) and performing a full 3DXRD at different azimuthal positions. With the described camera configuration, entire azimuthal range can be observed in four camera positions, i.e. "panels". In Figure 1 shows a comparison of a classical ff-3DXRD pattern and four HR-3DXRD panels captured in this experiment. For the latter, it should be noted that a full 3DXRD scan was performed at each panel position. For all samples both classical ff-3DXRD and HR-3DXRD scans were collected.

Figure 2 shows a single panel from HR-3DXRD scan of Al1050 sample. In the figure, it can be clearly seen that diffraction signal is obscured by an increased backgroud. This is a common scattering feature in deformed materials that is known to be caused from high concentration of low angle dislocation boundaries [7,8]. For this sample, it was concluded that the annealing state of the materials is inadequte for the high resolution detector setup to differentiate diffraction signals efficiently from the unwanted signal.

For the second sample of HEA alloy, full HR-3DXRD scans were acquired. Scanning a single panel took ~2 hours, thus total scanning time was ~8 hours. In the first trials, it was seen that during the scans, the detector system was drifting away from its aligned positions. Remedy is found by selecting single diffraction spots in each panel position and use them for referencing with height scans. With this approach 2 full datasets were acquired. Implementation of analysis for HR-3DXRD method is still ongoing.

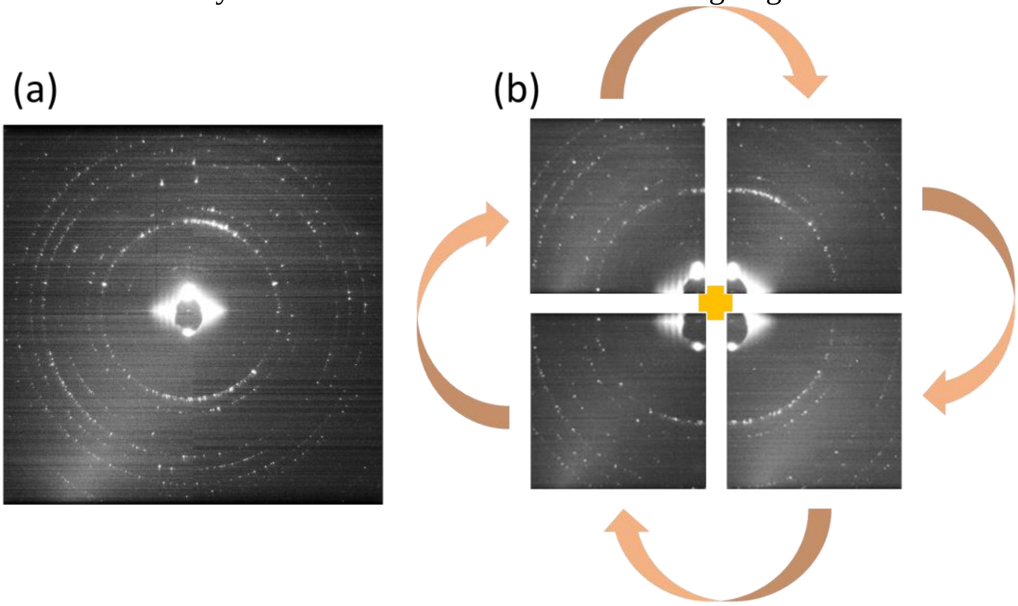

Figure 1: A comparison of FoV for (a) classical ff-3DXRD and (b) HR-3DXRD techniques. Due to small pixel size, the latter configuration covers the Debye-Scherrer rings with four panels in azimuthal direction. The images correspond to same slice in the tomographic rotation.

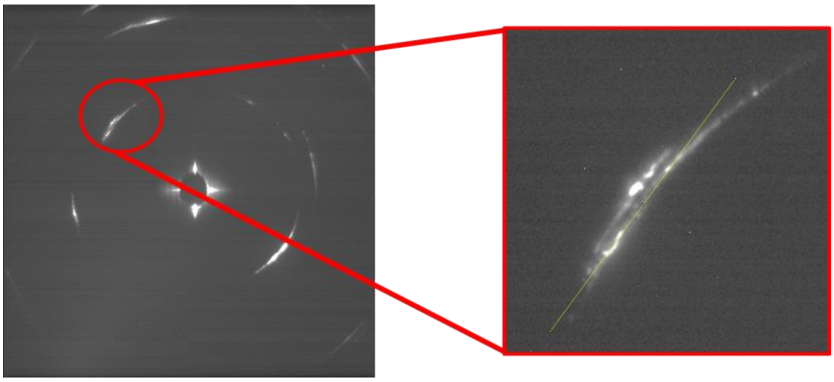

Figure 2: A frame from ff-3DXRD scan (left) and its close-up from HR-3DXRD scan (right). The diffraction signal from small grains/domains are obscured dut to the high density of low angle boundaries.

References

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