EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** Imaging Sub-Grain Deformation Structure in Ni by Micro-Beam Topography	**Experiment number**: MA874
Beamline:	**Date of experiment**: from: 01/10/2009 to: 07/10/2009	**Date of report**: 20/02/2010
Shifts:	**Local contact(s)**: Alexander Rack, Remi Tucoulou Tachoueres	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Brian Abbey, Department of Engineering Science University of Oxford Parks Road Oxford OX1 3PJ Great Britain*

Mr Felix Hofmann, Department of Engineering Science University of Oxford Parks Road Oxford OX1 3PJ Great Britain*

Prof. Alexander Korsunsky, Department of Engineering Science University of Oxford Parks Road Oxford OX1 3PJ Great Britain*

Report:

A) Introduction

The fundamental mechanisms governing the deformation of polycrytalline metals is an issue of prime importance in engineering science. During deformation new dislocation boundaries form, propagate and nucleate resulting in significant changes to the strength and ductility of metallic materials. Techniques which enable dislocation formation and movement to be tracked 'in-situ' are key to understanding how materials can be made stronger and more durable. There is now a substantial body of evidence showing that as single crystals or individual grains within a polycrystal deform, they fragment forming a new more complex sub-grain structure. This structure is composed of relatively dislocation-free regions or 'cells', seperated by sub-grain boudaries ('walls') where the dislocations are pinned during deformations. In recent years new micro-beam X-ray methods such as 3D XRD [1] or differential apeture microscopy (DAXM) [2] have been able to detect the presence of this dislocation cell/wall arrangment. Research has shown that diffraction peaks from defomed single crystals are characterised by sharp peaks produced by coherent scatter from the low dislocation density interior of the cells and a diffuse scatter from the dislocation rich sub-grain boundaries. Although the presence of the sub-grain structure has been confirmed using X-ray techniques, methods for actually obtaining a real space image of the structure as well as mapping the dislocation density have been lacking.

In the present study the aim was to test a new technique for imaging and charactersing the sub-grain dislocation structure in highly deformed Ni polycrystal. This technique, which we here refer to as scanning X-ray microbeam topography is a combination of standard X-ray topography and microbeam XRD. Several

suggestions for X-ray topography of highly-warped crystals have been made in the literature [3,4], however these do not apply to very severe distorsion and lattice misorientation and are difficult to implement practically at a synchrotron source.

In previous work we found that a monochromatic beam does not provide a sufficient spread of energies ($\Delta E/E \sim 10^{-4}$) to be able to capture all of the lattice rotations within the grain making a complete map of the deformed grain unpactical in a reasonable amount of time. White beam meanwhile would be ideal for imaging, however extracting quatitative information such as strain from the diffraction data then becomes extremely challenging. 'Pink' beam however with an energy spread (typically at least a few 100 eV) provides the ideal compromise between quantitative interpretation and speed of measurment. If in addition a higly focused/collimated micron sized beam is used to probe the sample, the orientation the spread of the lattice rotations probed by the beam should be small enough that the resulting topograph may be interpreted in terms of real space measurment of the sample. The availability of focused pink beam and of a high-resolution detector (FReLoN) at beamline ID22 at the ESRF allowed this idea to be fully realised and tested.

B) Results

During 18 shifts of beamtime allocated to this experiment we were able to collect transmission microbeam topography data from a single grain within a Ni polycrystal in two different defomormation states. Due to the non-standard setup for the experiment a substantial amount of time (3-4 days) was spent on alignment and setting up of the equipment. The first aim of the experiment was to demostrate the improvement offered by the new technique compared to 'classical' X-ray topography where normally a large box beam is used to image the sample. Figure 1 shows the orientation map (obtained via electron backscatter detection –EBSD) and scanning transmission X-ray microscopy (STXM) image for the region of the sample which was studied. It is important to note that since we have combined STXM and diffraction data, we know to within 1 μm exactly where we are scanning on the sample. Shown in Fig. 2 is a topograph obtained from the Ni grain using a 100 x 100 μm parallel beam and a topograph obtained from within the grain using a ~1 x 1 μm focused beam.

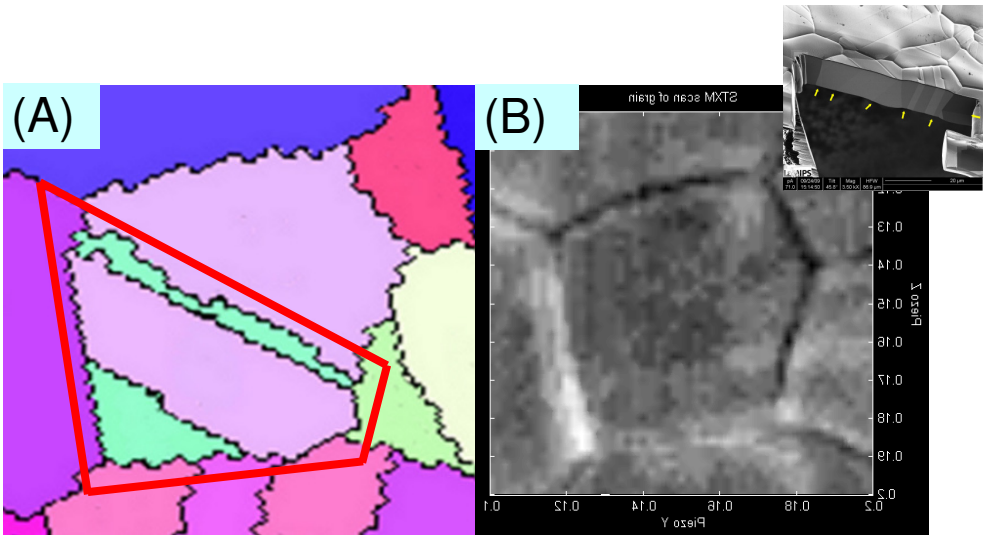

Fig.1 (A) EBSD orientation map of grain showing twin. The red line delineates the grain which we were able to map. (B) Scanning transmission image from ID22 of the same area collected at the same time as the diffraction data. Inset shows a cross-sectional SEM image from a representative portion of the sample confirming that there is normally only one grain through thickness.

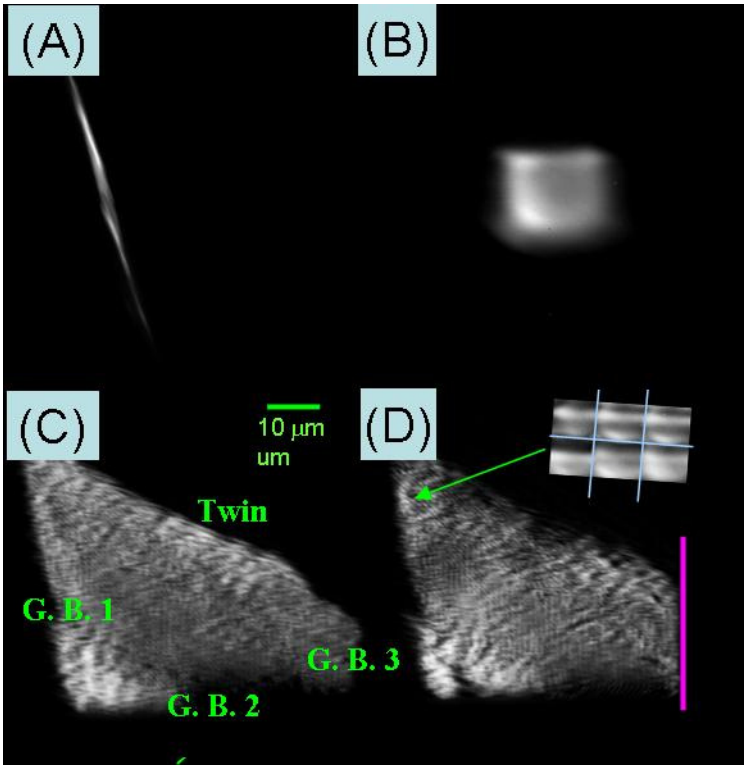

Fig2. (A) 'Classical' X-ray diffraction topograph of the (220) reflection from whole grain using a 100 x 100 μm 'box beam', the elongation is due to significant lattice misorientation within the grain. (B) Topograph from ~1 x 1 μm region within the grain, the uniform intensity indicates that this is probably an image of the incident beam diffracted from a dislocation-free region of the grain. (C) Composite image from ~5000 topographs similar to those shown in (B), note that over the area probed by the focused beam there is only significant spreading of the reflection close to the grain boundary. The twin boundary and grain boundaries 1, 2 and 3 are labelled. (D) Image of the same grain after further deformation made up of ~3000 topographs, the pink line indicates the end of the scan.

Results show clearly that the topograph from the whole grain cannot be interpreted in terms of a real space image, however using the scanning technique a composite topographic image of the grain may be recovered providing the beam is small compared to the gradient of the lattice rotations within the crystal. The composite topographs from the scanning method provide a wealth of information compared to the box beam topograph in Fig. 2A or the scanning transmission X-ray intensity image in Fig. 1B. There is indication of the presence of dislocation boundaries running through the topographs and of strain at the twin boundary, there also appears to be evidence for the beginning of a crack at the corner of the grain in Fig. 2D. These qualatative observations can be interpreted through analysis of the individual topographs shown in Fig. 3.

Figs. 3A and 3B show the variation in θ, (half the scattering angle between the incident and diffracted beams) whilst 3C and 3D display the change in the value of χ, defined here as the angle between the scattering plane an the vertical plane at the detector. In theory the derivative of the lattice rotation should reveal the presence of dislocation boundaries. Figs 3E,F and 3G,H are plots of the magnitude of the gradient of rotation in θ and in χ. The pile-up of dislocations at the grain boundary and along lines within the grain are visible. Since the grain orientation was later determined using microbeam Laue we can identify the active slip sytems associated with the {111} planes. Based on this data the dislocation boundaries match two of the four possible slip directions calculated for this grain. In addition to the dislocation data we also scanned the energy during the 2d scans and thus were able to obtain an estimate of strain acccross the grain boundary (this data is not presented here). Finally we are devloping models capable of simulating the dislocation pile-up within the grain which will help further our fundamental understanding of how the Ni polycrystal deforms.

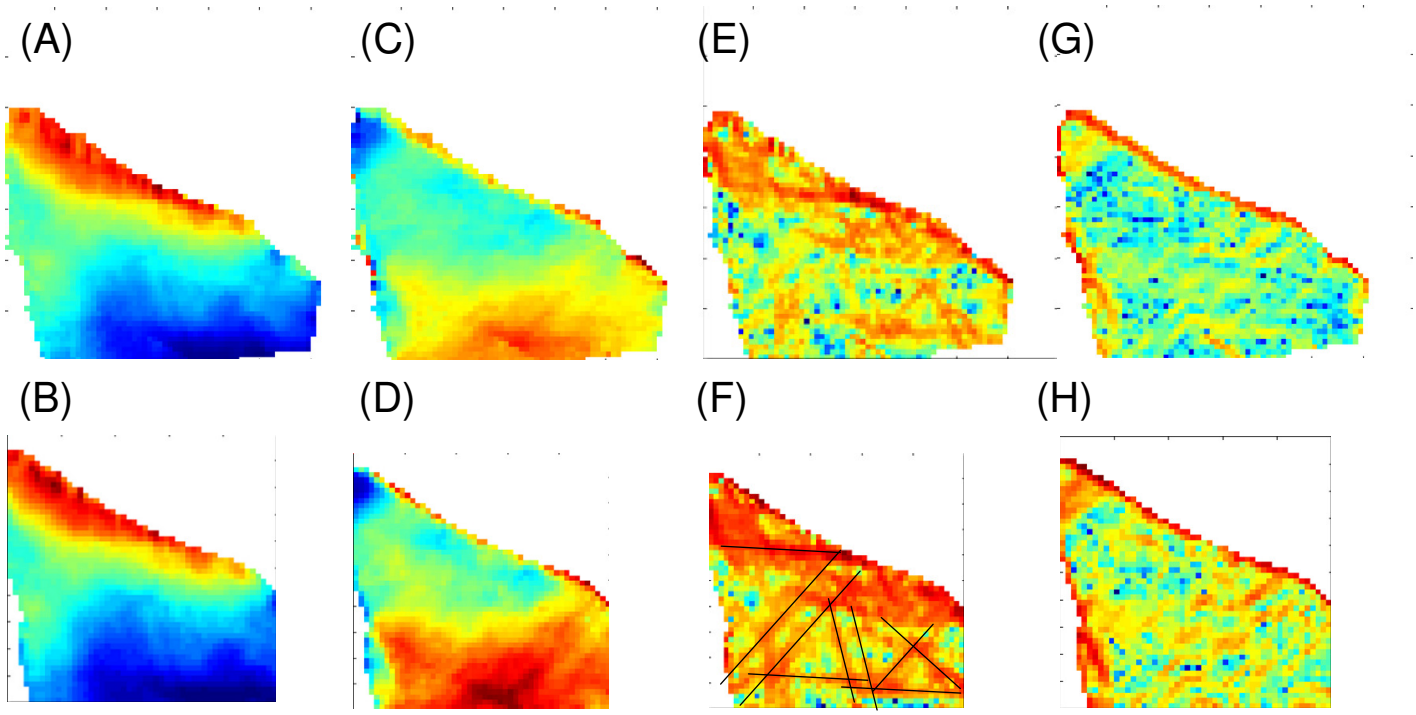

Fig3. (A), (C), (E) and (G) show the variation in θ (half the scattering angle), the angle between the scattering plane and the vertical (y) axis in the detector plane (χ), the gradient of θ and the gradient of χ respectively for the grain in the initial deformation state. (B), (D), (F) and (H) are the corresponding values for the grain after further deformation. Note that all data are presented at the same pixel resolution at which it was collected (i.e. with *no* interpolation).

C) Conclusions

The experiment had the following outcomes:

- A new scanning technique for making topographic measurements of highly deformed polycrystalline materials was tested.
- It was shown that this method can be used to obtain a diffraction image of the grain when standard topography cannot.
- Combined real and reciprocal space imaging techniques allowed a single grain to be mapped at sub-micron resolution simultaneously giving both absorption and diffraction constrast.
- The data clearly shows 2d maps of dislocation boundaries within the deformed Ni grain and their build up after applying load.
- This data can be combined with micro-beam Laue measurements to indentify the active slip systems within the grain.
- The experiment provides the fundamental data necessary to further develop models to predict the crystal plasticity of Ni polycrystal.

This work is currently being prepared for publication for presentation to the materials science and engineering community.

D) References

[1] Fu et al. *Non-destructive mapping of grains in three dimensions*. ***Scripta Materialia*** 49, 1093-1096 (2003).

[2] Larson et al. *Three-dimensional X-ray structural microscopy with submicrometre resolution.* ***Nature*** 415**,** 887-890 (2002).

[3] Mellaer et al. *Feedback-Controlled X-Ray Diffraction Topography*. **Phys. Stat. Sol.** (a) 3, 687 (1970).

[4] Lang and Makepiece, *Reticulography: a simple and sensitive technique for mapping misorientations in single crystals*. **Journal of Synchrotron Radiation** 3, 313-315 (1996).