EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Long Term Project Report : Interim/Final

Summary Page

1. **Beamtime Used**

Please give a short summary of progress for each scheduling period for which beamtime has been allocated/used :

Scheduling period	Beamline(s) Used	Shifts Used	Summary of results obtained
2011 /II	ID11	15+6	The interim nanoscope set-up has been commissioned. It allows 3D grain mapping at the level of 100 nm. Data analysis on a 3D map of the dislocation cells within a highly deformed Aluminium sample is in progress. A proposal for completing the Nanostage set-up with a dedicated optics mount and a dedicated goniometer has been forwarded to ESRF management. The ESRF shut down did not allow for beamtime on other branhes of the project, but for pragmatic reasons two days were used to complete data taking on an external project MA-1213 .

2. **Resources Provided by User team (financial, personnel, technical...):**

Outstationing of personnel progress as forseen in contract. In addition the 3D detector at ID11 was refurbished with a new set of (structured scintiallator) screens in Dec 2011. The R&D behind the screens come from Risø.

3. **Technical and Scientific Milestones Achieved** (in relation to the milestones identified in the original proposal):

Year 1
Interim nanoscope commissioned

Year 2

Year 3

4. **List of publications directly resulting from beamtime used for this Long Term Project:**

None yet.

Annual report for LTP 1317

ESRF	**Experiment title:** Next generation 3DXRD	**Experiment number:** MA-1317
Beamline: ID11	**Date of experiment:** from: 1/9 2011 to: 31/12 2011	**Date of report:** 1/2/2012
Shifts:	**Local contact(s):** A. King, G. Vaughan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

H.F. Poulsen[1]*, S. Schmidt[1]*, X. Huang[2], E.M. Lauridsen[3]*, G. Winther[4], W. Pantleon[4], W. Ludwig*[5], J.-Y. Buffiere[5], A. Snigirev*[6]

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This report highlights the research from the first four months within LTP MA-1317.

This LTP is the continuation of a long standing collaboration between the staff at beamline ID11, a group in Denmark and one at INSA-Lyon on the development and proliferation of 3D grain mapping and grain dynamics methods. Starting in 1997 pioneering optics for hard x-rays were developed and first feasibility experiments demonstrated by the Risø group. This lead to the commissioning of the 3DXRD microscope at ESRF in 2000, an instrument funded by and built at Risø. During the years the technique rapidly developed, the user group expanded and with time the ID11 staff took over more of the external user guidance. In 2005 it was decided to completely refurbish the beamline and install a new 3DXRD instrument in a new hutch. This instrument was commissioned in 2011. In 2007 the INSA-Lyon based group jointly with Manchester University pioneered Diffraction Contrast tomography (DCT), a powerful alternative to 3DXRD. In conjunction with a number of other technical improvements during the last couple of years – e.g. on detectors and algorithms - a diverse and mature user community has been created.

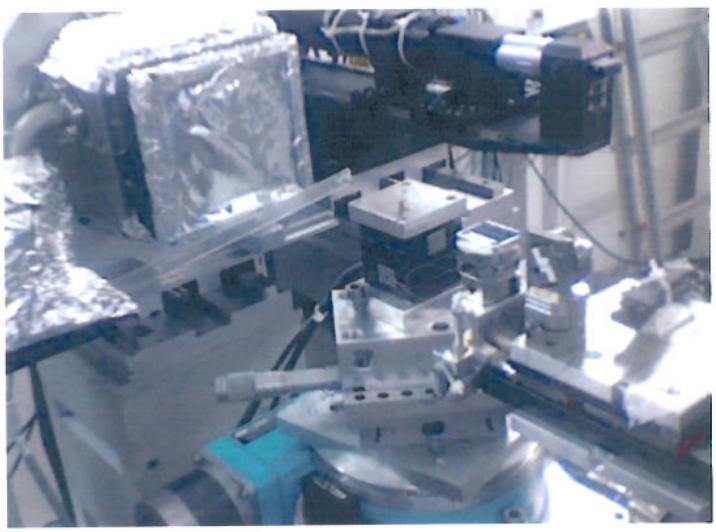
The methods however have two basic shortcomings: a) the spatial resolution is limited by detector technology to at best 500 nm and b) strain variations within grains cannot be imaged.
The overall objective behind the current LTP is to provide solutions to these two outstanding issues. As such progress within the LTP is intimately tied to the strategy and technical progress of the ID11 beamline.

A. Developing a diffraction based Transmission X-ray Microscope.

In Nov 2011 it was announced that one of the proposers – Henning Friis Poulsen - received an **ERC Advanced Grant**. The proposal has the title "diffraction based Transmission X-ray Microscopy". The grant will strengthen this part of the LTP and most likely lead to the **out-stationing of an additional post doc at ESRF**, in addition to those already promised.

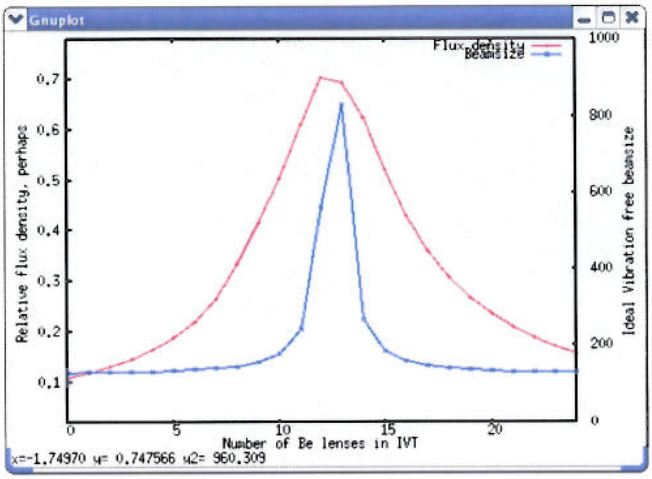
Commissioning of the nanoscope

The main focus within the period has been work on the Nanoscope at the end of the EH3 hutch. The Nanoscope is currently intended to be an interim "hard x-ray optics test bench" with the aim of demonstrating scanning diffraction microscopy in the energy range above 25 keV and a resolution of order 100 nm. The strategy has been to build a stable permanent sample/optics platform and a detector arm - in part by components delivered by Risø. On top of the optics/sample platform an *ad hoc* compound refractive lens (CRL) mount and an *ad hoc* sample tower has been built from existing components, the aim being to better understand the current limitations on spatial resolution, in order to construct in the immediate future an instrument capable of performing in the desired range.



The set-up is illustrated above. 1D Si lenses are used. These have small shape errors and also have the advantage in comparison to 2D lenses that a larger demagnification can be used in the horizontal plane, compensating for the anisotropic source size. The use of coupled 1D lenses also allows the elimination of any astigmatism. This allows a more or less isotropic focus size to be obtained. To obtain a sufficient yield a fluorescence detector with a large acceptance angle is mounted close to the sample (not shown).

- *Optimisation of flux:* At the expense of broadening of the nano-focused beam the flux can be increased by inserting an in-vacuum transfocator (IVT) upstream from the nano-lenses. Theoretical estimates (by J. Wright) are shown to in the figure to the right for a beam energy of 30 keV. As function of number of Be lenses in IVT the size of the nano-beam is shown in blue color and the relative flux is shown in red color. It is seen that, e.g. by inserting 11 lenses the beam size is increased from 130 nm to 241 nm whereas the flux has gained a factor 7. The same trend has been observed in actual experiments at the nanoscope, see below.



- *Vibration test of nanoscope.*
A good mechanical stability of the nanoscope is essential in order to produce a high quality nano beam. A vibration spectra measured on top of the sample/optics platform ("table") at the nanoscope, see the figure to the right, revealed an excellent performance below 70 Hz. The studies also showed that amplification of existing vibrations as well as creation of new frequencies could easily happen when combining positioning components on top of the table. Consequently, a custom integrated sample mount is desirable in the final nanoscope setup. For more information, see Appendix B.

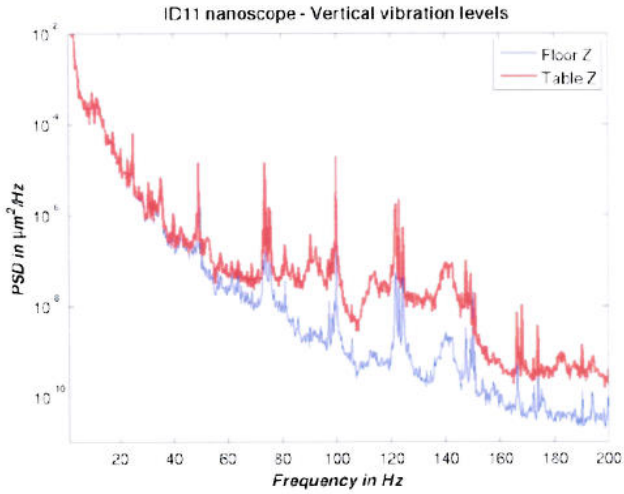
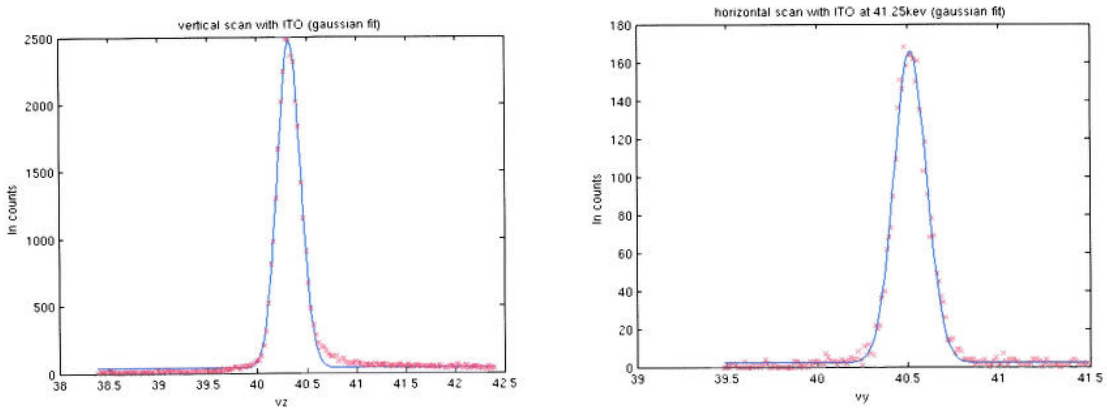


Figure X: Vibration spectra at the nanoscope. Red color: on the table. Blue color: on the floor in EH3 at ID-11.

- *Beam profiles and beam stability tests.*



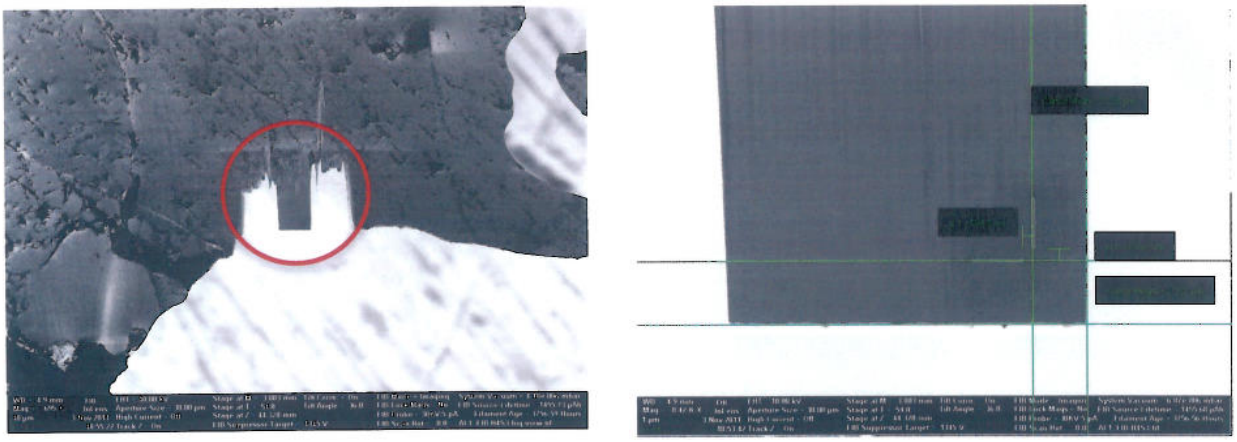
Beamprofiles obtained at 41.25 keV is shown above (vertical to the left, horizontal to the right). They are seen to be nearly Gaussian with FWHM of 271 nm and 216 nm, respectively. A monotonous drift was observed during several nights with an average drift velocity of 1.16 nm/minute (vertical) and 0.61 nm/minute (horizontal).

The various contributions to the beam profiles shown above are estimated as (numbers in nm):

	Source size	Diff. limit	sample	Bandwidth	Vibrations	Sum	Observed
Vert	94	169	120	50	140	272	271
Hor	108	57	120	50	140	227	216

First Nanoscope Experiment: 17-22 December 2011

A monochromatic beam of energy of 42 keV was provided by the double Laue monochro-mator. The focal lengths of the vertical and horizontal Si nano-lenses were 110 mm and 58 mm, respectively, thus situating the common focal point, i.e. the sample position, at an amble distance to the nano-lenses. The lenses used the setup had 50 μm holes in a configuration with 1662 lenses vertically and 3153 lenses horizontally (i.e. the setting "Set-2-Nano-50-02"). In order to increase the flux an in-vacuum transfocator (IVT) with 16 Be lenses was inserted upstream from the nanoscope. This resulted in a seven times increase in flux at the expense of broadening of the beam. The resulting beam size was 386 nm horizontally and 301 nm vertically.

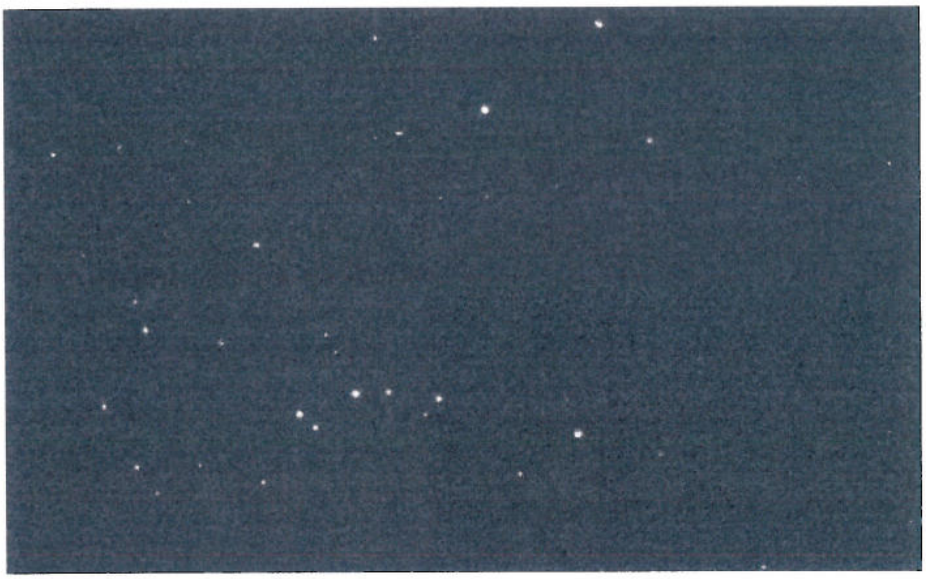


The sample material was highly deformed Al, produced by the so-called ECAE process. It has a grain size of ~ 500 nm, an almost random texture and a very high fraction of high angle boundaries (70% of the grain boundaries are mis-oriented by more than 15 degrees). A 20.4 μm × 8μm (height) x 13 μm square block was manufactured by FIB'bing. This is marked by red in the figure above to the left and shown as a zoom to the right. An L-shaped marker of Pt – with a thickness of 293nm - was placed near the edge of the block – this served as a fluorescence marker.

The w-rotation table in this interim set-up is associated with a wobble that prohibits integration of intensities by "rocking" as conventionally performed in x-ray diffraction. Instead 2D mapping was performed at a series of w positions, with a step of 0.11 degrees – sufficiently smaller than the typical mosaic spread of the diffraction spots. The following ω intervals were covered: [-60; -33.93], [0; 5.72], [20; 29.9] and [40; 48.69]. For each ω-step a grid pattern with 45 steps along the y-direction and 18 steps along the z-direction were recorded by translating the sample using the piezo stages. The step size along y and z was 100 nm, thus covering 4.5 μm by 1.8 μm.

The positions of the Pt markers were measured prior to each grid scan using a fast zapscan algorithm enabling measurement of the florescence signal during continues movement of the piezo stages. The precision of the marker positions was 10-20 nm. As the makers were deposited on the surface of the sample their measured positions were not coinciding with the sample reference positions. Adjustments were made to compensate for systematic shifts.

The diffraction data was acquired with a Frelon2k detector binned in 1024 by 512 pixels to optimize sensitivity and readout time. The exposure time was 0.3 s per frame. Use of frame transfer mode allowed dead time to be negligible. Sharp distinct spots were observed, as seen in the figure to the right. Notably, spot overlap is negligible.



A preliminary evaluation of the data has been carried out on the ω interval [-60; -33.93]. First, the position and intensity of the individual spots were identified using the program *peaksearch,* which is part of ImageD11. Next, for each ω the diffraction spots were merged into unique spots having the same position in some of the 45x18 images in the grid using the program *NProbe*. Afterwards, unique spots in neighboring ω's are merged and g-vectors are calculated along with weighted mean positions of the piezo stages (corrected for Pt marker offsets). The g-vectors were then indexed by the program *GrainSpotter*. The precision of the orientations is estimated to be better than 0.1 degrees. On average 10 g-vectors are assigned to each grain in good agreement with simulations.

The data analysis is ongoing; the first goal is to produce a centre of mass map of the grains in the illuminated volume. Afterwards, the map will be extended to include the morphology of the individual grains. The fully reconstructed volume will correspond to a gauge volume of approximately 1.8 x 4 x 4 μm^3 in the middle of the sample, comprising approximately 200 grains.

Conclusions on Nanoscope

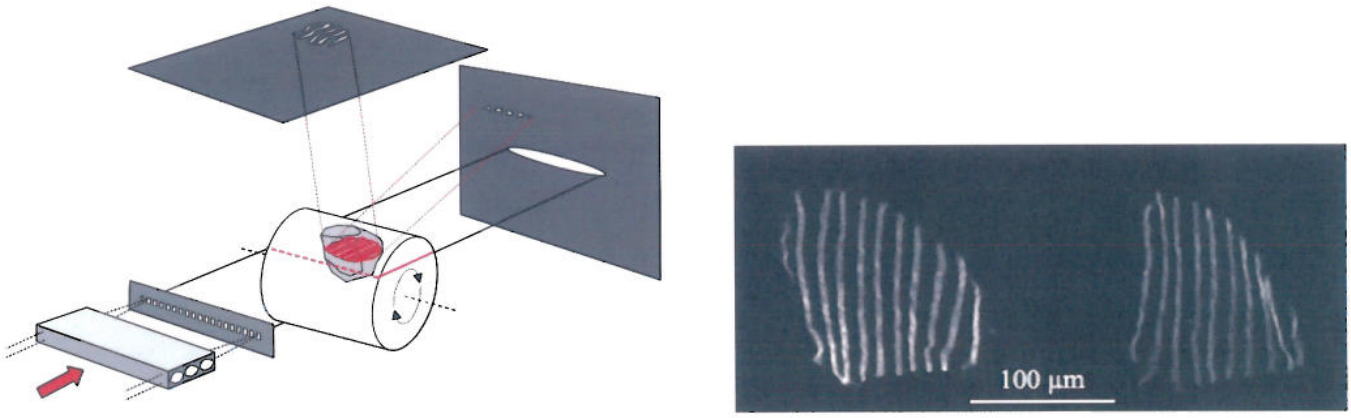
- Pending the outcome of the data analysis, the interim set-up seemingly allows 3D grain mapping at the level of 100 nm. However, the lack of a dedicated rotation stage with a wobble of less than 100 nm implies that the 3D mapping procedure is prohibitively slow for most applications. Likewise the spatial resolution of the set-up is fundamentally limited by the vibrations in the *ad hoc* optics and sample towers – we estimate the vibrations to be of the order 140 nm at the moment. A proposal for completing the Nanostage set-up with a dedicated optics mount and a dedicated goniometer based on a nano-spindle concept has been forwarded to ESRF management.

- Operating at higher energies of 40-50 keV has the advantage that the effective aperture is larger, and the diffraction limit smaller. Furthermore the peak flux from the monochromator at the beamline is at about 40 keV. The scattering efficiency of the sample is sufficient that 3D mapping of 500 nm Al grains is not limited by flux.

Bright-field and Dark field microscopy

Due to the limited period, no experiments performed at ID11.

B. Developing imaging methods for intra-grain strain mapping.



. Vertical acquisition geometry for optimized spatial resolution and strain sensitivity. Data from feasibility experiment MI1026 - ID18F. PhD projects of L. Nervo and N. Vigano, (ANR projects Crystal, MicroNaSel; W. Ludwig, ID11).

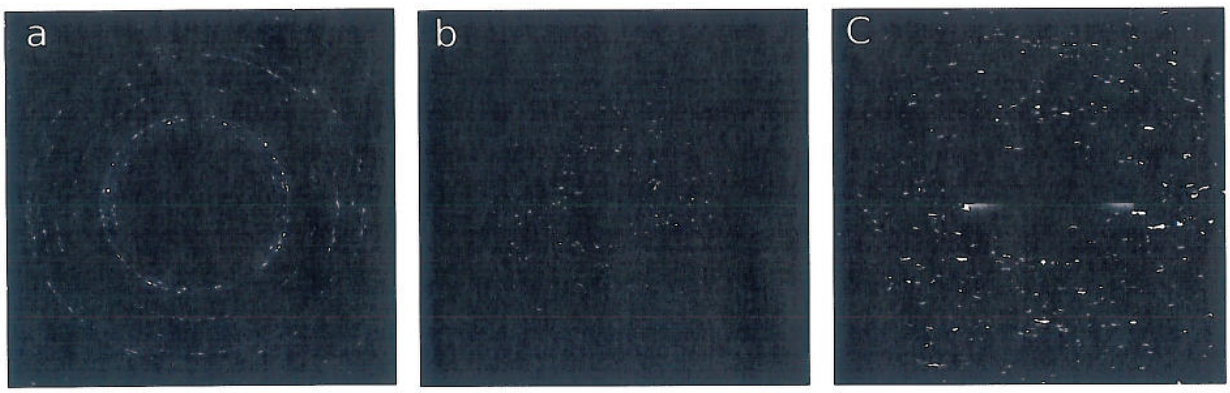
Working at energies below 20 keV, it is possible to acquire X-ray section topographs at (2θ) diffraction angles close to 90° (vertical diffraction plane). By structuring the incoming and/or diffracted beams (fig. X) one can measure local variations of the diffraction angles with high spatial and angular resolution, as required for characterization of intra-granular elastic strains. First feasibility experiments (MI1026 & IHR at ID18F) are highly encouraging and should be pursued in the frame of the current long term proposal. However, these experiments are best performed with a horizontal rotation axis at energies below 20 keV - a spectral range which would require refurbishment of the Bragg-Bragg monochromator at ID11. A request to continue this part of the LTP technique development at a different beamline has been submitted to the ESRF director of research.

C. Scientific use

As outlined in proposal such experiments are planed for a later stage.

Mapping of 3D twin morphology in Zr

In collaboration with Mark Daymond from Queen's University in Canada two days of beamtime was spent to map the 3D morphology of deformation twins in a 2.5% deformed Zr sample. The stress state in the individual parents and twins in the same specimen had previously been mapped (MA-1213, ID11 June 2011) at several load steps from the undeformed twin-free state, through the initiation and growth of deformation twins during in situ tensile loading. The experiment was only the second use of the 3D detector after the refurbishing of EH3 and the data quality was really good. Once reconstructed the 3D grain map will be used to study the influence of neighbouring grains on twin inception and propagation, and the results will be used to improve the Crystal Plasticity Finite Element models developed at Queen's that currently build on the 2D experimental evidence from EBSD.



Diffraction data from the Frelon detector (a) and the two screens of the 3D detector (b and c) showing the increased spatial resolution when going from a to c.