Session II, Monday, June 20

L7

LINE PROFILE ANALYSIS AND ROCKING CURVE EVALUATION OF 3D DIFFRACTION DATA

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Diffraction patterns from ~100 individual grains of a solutionized and quenched metastable -Ti alloy were obtained by high energy synchrotron diffraction during in-situ tensile deformation experiments. The diffraction patterns of select grains were analyzed per an established single-crystal line profile analysis technique to assess the dislocation density evolution on individual slip systems. Further, a new technique to estimate the geometrically necessary dislocation (GND) density from rocking curves is introduced. The results provide a powerful complement to previously published comparisons between measured and crystal plasticity simulated internal elastic strains (and stresses). In particular, they reveal there is no preference for 1/2 Burgers vector dislocations to reside on a particular glide plane, since they have similar densities on {110} and {112} planes. In addition, an explanation for the observation of strain softening in some of the grains is hypothesized as form of "plastic buckling". A select number of strain softening grains exhibit higher lattice curvature (GND density) than other grains, indicating that the grains have "broken up" into smaller domains which are deforming in distinct ways from one another, and more easily than they would have together.

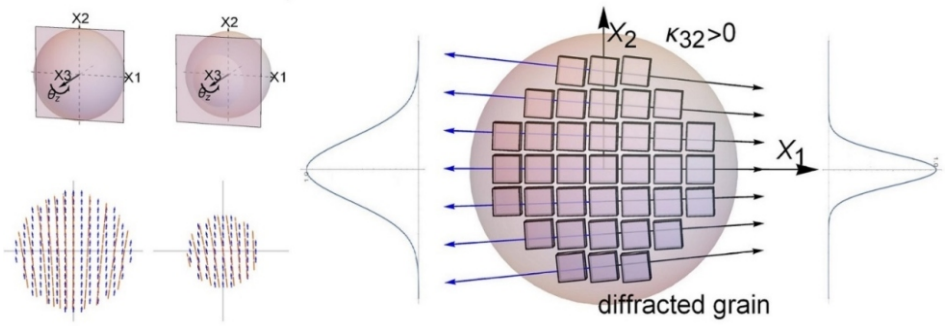

Figure 1. Rocking curve broadening vs grain curvature

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LABORATORY AND SYNCHROTRON ROCKING CURVE IMAGING FOR CRYSTAL LATTICE MISORIENTATION MAPPING

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X-ray diffraction topography (diffractometry) is a traditional method of visualising crystal structure perfectness on a film with a high spatial resolution. However, angular misorientation inspection requires multiple exposures for several diffraction angles around the Bragg peak by means of a manual film exchange, which is a bit cumbersome. This limitation has been overcome by utilisation of digital 2D detectors which were available at synchrotron imaging beamlines with high flux and parallel beam, thus the rocking curve imaging (RCI) technique has been developed. Recently, RCI was transferred from synchrotron to laboratory set-ups.

Nowadays, RCI is an X-ray diffraction technique which combines full-field X-ray digital topography and Bragg-diffraction rocking curve recording. A large (almost) parallel monochromatic beam irradiates a crystalline sample with a misorientation distribution characterized by local tilt angles. Series of digital topographs are measured by a two-dimensional detector at different sample orientations from which peak characteristics of millions of local Bragg peaks from each series are extracted. The field of view and lateral resolution is given by the camera size, its pixel size and the Bragg angle, while the angular resolution is given by the rocking curve width being typically much smaller than the misorientation angles of the studied crystal. Simultaneous high spatial resolution provided by the two-dimensional detector and high angular resolution (0.001°) allows to quantify crystalline structure perfectness over large sample area which scales with the area of the detector. Therefore the rocking curve imaging is an imaging method with faster recording compared to usual laboratory scanning area diffractometry which requests measurement of the rocking curve at each surface point.

Synchrotron RCI [1,2] profits from large parallel beam, high flux and small detector pixel size down to one micrometre. For small misorientations of the crystal lattice, detector can have any distance from the sample, while larger misorientations due to inherent focusing and defocusing of the diffracted (micro)beams require a dedicated reconstruction procedure.

Laboratory RCI [3] with a slightly diverging beam requires small misorientation angles and very small sample to detector distance, thus a home-made extension for a commercial diffractometer is necessary. Current two-dimensional detectors available at laboratory diffractometers have typical spatial resolution downto 0.1 mm which make it possible to analyze a large sample area at once.

On several examples, we will demonstrate the RCI technique for a characterisation of several large-area semiconductor wafers, such as silicon, silicon carbide, gallium nitride or overgrown silicon-germanium microstructures.

1. P. Mikulík, D. Lübbert, D. Korytár, P. Pernot, T. Baumbach, *Journal of Physics D: Appl. Phys.* **36**, 2003, A74.
2. D. Lübbert, C. Ferrari, P. Mikulík, P. Pernot, L. Helfen, N. Verdi, D. Korytár, T. Baumbach, *Journal of Applied Crystallography* **38**, 2005, 91.
3. M. Meduňa, O. Caha, E. Choumas, F. Bressan, H. von Känel, *Journal of Applied Crystallography* **54**, 2021, 1071.

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RADON TRANSFORMATION IN RECIPROCAL SPACE

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Mapping intensity in reciprocal space around certain reciprocal lattice points is mostly realized in two-dimensions within the (Q_xQ_z) plane which is usually sufficient for any analysis of crystal lattice strain, mosaicity, structural quality, internal defect distribution or surface morphology and structure, depending on the scattering technique used. Nevertheless, in certain cases mapping of the scattered intensity in 3D space, out of the scattering plane e.g. along the Q_y axis, is more and more demanded as well. Scanning reciprocal space in 3D however requires a well collimated X-ray beam in all directions, so that such experiments are realized mainly at synchrotron sources in order to have a sufficiently intense and parallel beam. Practically any 2D pixel detector is also extremely suitable for timely effective collection of reciprocal space maps (RSMs).

In this work we realize a collection of 3D RSMs using standard laboratory equipment with only a partially-collimated beam using a typical linear focus X-ray tube. We use a Rigaku SmartLab diffractometer equipped with a 2D HyPix area pixel detector for recording series of symmetric diffraction RSMs on microstructured semiconductor samples. The 2D detector is used in a linear mode for fast collection of typical 2D Q_xQ_z RSMs where the sample is scanned at many different azimuths with respect to the diffraction vector as an axis of rotation. The 3D intensity distribution in reciprocal space is then reconstructed by means of a Radon transform procedure, typically used for many decades in real space computer tomography (CT) [1]. The details of the technique used here in reciprocal space and experimental details realized can be found in recently presented work [2], including the details of the samples.

The method of applying the Radon transform for the reconstruction of RSMs is presented on Ge, GaAs and SiGe microcrystals epitaxially grown on patterned Si substrates [3]. Series of synchrotron experiments using a nanofocused beam, where 3D RSMs have already been measured on many samples, were previously presented in several of our publications [4,5,6], however in this study we compare similar laboratory measurements with the previously performed synchrotron experiments [2].

The Q_xQ_z RSMs obtained at various azimuths are first decomposed into sinograms for all Q_z positions, see an example for fixed Q_z in the left panel of figure 1. This example is built from RSMs where certain (004) lateral diffraction satellites appear and their Q_x position changes as the sample is rotated along . After application of the inverse Radon transformation on this sinogam map, we can obtain the spatial distribution of these maxima within the Q_xQ_y plane perpendicular to the axis of rotation which shows four-fold symmetry, see the right panel of figure 1. Let us note that these satellites originate from a superlattice covering the faceted SiGe microcrystals. Comparing the new laboratory data [2] with previous synchrotron measurements [6], we get a very good agreement of the 3D spatial distribution of intensity.

1. P. Suetens, in Fundamentals of Medical Imaging, Cambridge University Press, 2009, pp. 35–41.

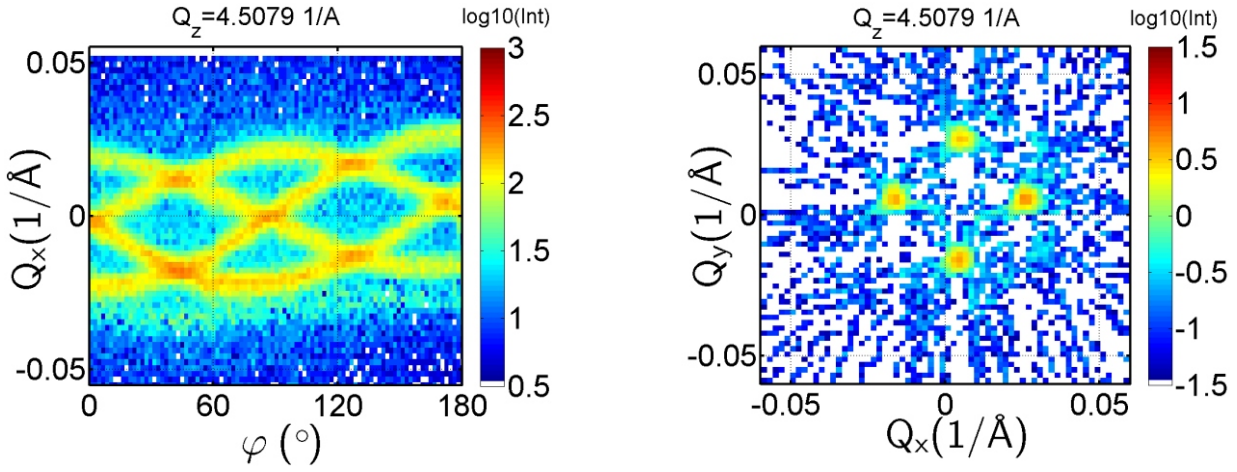

Figure 1. (left) Sinogram built from slices of typical Q_xQ_z RSMs for fixed Q_z position close to SiGe (004) recorded at a series of azimuthal rotations of the sample containing four-fold symmetric diffraction superlattice satellites. (right) Reconstruction of the Q_xQ_y map in reciprocal space using the inverse Radon transform applied on the sinogram in the left panel can be understood as a given slice for fixed Q_z through the 3D RSM. The four-fold structure of the four maxima is evident.

2. M. Meduňa, F. Isa, F. Brennan, H. von Känel, J. Appl. Cryst. 55, (2022) in print.
3. C.V. Falub, H. von Känel, F. Isa, R. Bergamaschini, A. Marzegalli, D. Chrastina, G. Isella, E. Müller, P. Niedermann, L. Miglio, Science, 335, (2012), 1330.
4. C. V. Falub, M. Meduňa, D. Chrastina, F. Isa, A. Marzegalli, T. Kreiliger, A.G. Taboada, G. Isella, L. Miglio, A. Dommann, and H. von Känel, Scientific Reports 3 (2013), 2276.
5. M. Meduňa, C.V. Falub, F. Isa, A. Marzegalli, D. Chrastina, G. Isella, L. Miglio, A. Dommann, and H. von Känel, J. Appl. Cryst, 49, (2016), 976.
6. M. Meduňa, C.V. Falub, F. Isa, D. Chrastina, T. Kreiliger, G. Isella, H. von Känel, J. Appl. Cryst., 47, (2014), 2030.

We acknowledge the staff of the ID01 beamline at the ESRF, Grenoble.

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IMAGE CORRECTIONS FOR POWDER DIFFRACTION WITH THICK SENSOR DETECTORS

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Flat hybrid pixel detectors present the most common instruments for recording X-ray intensities in scattering experiments at photon light facilities as well as in X-ray laboratories. This holds in particular for conventional crystallographic experiments. In order to optimize quantum efficiency of the detection process the ratio of detector sensor thickness and pixel size is often set quite high. A narrow X-ray beam entering such a detector at an oblique angle is absorbed in multiple consecutive pixels. This is causing an effective shift of the detected signal known as the "parallax" effect [1, 2]. Beside this the absorption of X-ray beam in the detector sensor is more complete. The latter is called an "oblique incidence effect" [2]. Appropriate corrections are well established in software for single crystal diffraction data processing [1]. Marlton et al. [2] introduced the parallax effect correction for pair distribution function measurements. The idea is used to improve effective camera resolution in synchrotron and medical imaging [3]. In case of diffraction experiments with a flat powder sample in parallel beam geometry angular resolution is dominated by geometrical effects. In addition, for samples with crystallite size >1 um grain statistics is often not-ideal and azimuthally integrated diffraction profiles are not well defined. Diffraction spots may present blurred signal due to the parallax and oblique incidence effects as shown in Figure 1. Point spread function (PSF) over the detector area was estimated by ray-tracing the detector and the know PSF is deconvoluted from the measured signal in the next step. Results of such diffraction image processing are presented (Figure 1). Different methods for positionally variant deblurring with known PSF were used: direct inversion with regularization, Richardson-Lucy deconvolution [4, 5]

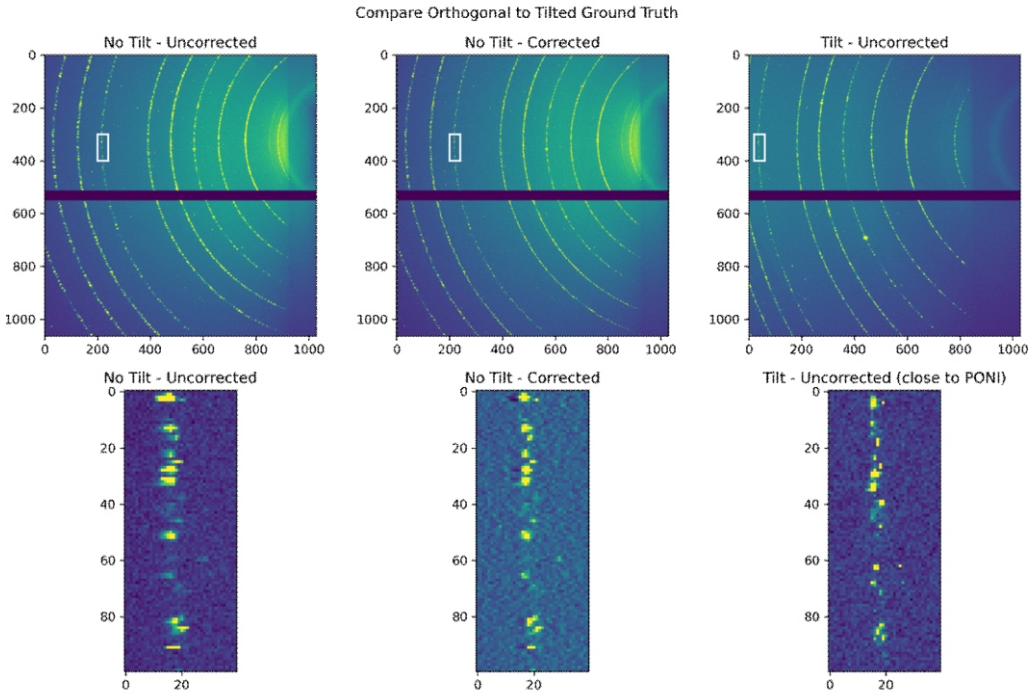

Figure 1. Positionally variant deblurring of X-ray diffraction images with known detector PSF.

and Deep learning approach [6]. Pros and cons of different methods are described, and applicability of the method is briefly discussed.

1. W. Kabsch, *X-ray Detector Software, XDS version: March 15,* (2007), https://xds.mr.mpg.de/html_doc/Release_Notes.html (visited May 20, 2022)
2. F. Marlton, O. Ivashko, M.V. Zimmerman, O. Gutowski, A.C. Dippel, M.R.V. Jørgensen, *J. ApplCryst.*, **52**, (2019), 1072-1076. doi: 10.1107/S1600576719011580
3. P. Shikhaliev, O. Fox, N. Tartoni, *IEEE Nuclear Science Symposium and Medical Imaging Conference (NSS/MIC)*, (2020), 1-3. doi: 10.1109/NSS/MIC42677.2020.9507906
4. W. Richardson, *J. Opt. Soc. Am.*, **62**, (1972), 55-59. doi: 10.1364/JOSA.62.000055
5. L.B. Lucy, *Astronomical Journal*, **79**, (1974), 745. doi: 10.1086/111605
6. J. Mustaniemi, J. Kannala, S. Särkkä, J. Matas. J. Heikkila, *IEEE Winter Conference on Applications of Computer Vision (WACV)*, (2019), 1914-1922. doi: 10.1109/WACV.2019.00208

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25TH ASSEMBLY AND CONGRESS OF THE IUCr IN PRAGUE, HISTORY, EXPERIENCE, STATISTICS

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