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# Observation of slip bands in the bulk of polycrystals using a combination of DCT and Topotomography

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**Abstract.** 3D diffraction imaging techniques like X-ray diffraction contrast tomography and Topotomography enable the observation of slip bands at the onset of plastic deformation in polycrystalline materials. Using the previously introduced 6D DCT reconstruction framework local changes in crystal orientation adjacent to these bands can be detected. The combination of both scanning modalities into a joint reconstruction problem with potentially superior spatial resolution and increased angular sensitivity is under investigation.

## 1. Observation of incipient plasticity in bulk polycrystals

Recent advances in the field of non-destructive bulk characterization by means of high energy X-ray diffraction techniques, as well as progress in image based simulation of polycrystalline microstructures (e.g. phase field, crystal plasticity finite element) enable one to one comparisons between experimentally observed and simulated material behaviour. The increase in X-ray flux due to the upgrade of the major synchrotron storage rings will enable 4D time-lapse observations of microstructures reacting to external stimuli like strain, temperature and corrosive environments.

In the current work we focus on the 3D characterization of early stages of plastic deformation in bulk of metallic polycrystals. The localization of plastic deformation inside individual crystals leads to the formation of slip bands. Slip bands have been studied extensively at the sample surface and thin foils using electron microscopies (SEM, TEM, EBSD). Polychromatic scanning micro diffraction techniques and their three-dimensional extension have recently been shown to be able to resolve minute orientation and strain gradients in the vicinity of slip bands [1] located in the bulk of the material. Slip bands in bulk crystals can also be observed by full-field X-ray diffraction imaging techniques like X-ray topography [2]. In this case gradients in orientation and strain give rise to topographic image contrast. However, the quantitative analysis of these contrasts in terms of absolute values of lattice rotation and strain generally requires series of diffraction images over the full range of the rocking curve and multiple reflections to be measured. It is not clear if 3D extensions of monochromatic beam, full-field X-ray diffraction imaging techniques like X-ray diffraction contrast tomography (DCT) [3] and Topotomography (TT) [4] can provide the spatial resolution and sensitivity to quantitatively characterize slip bands in terms of local lattice rotations.

In a recent study we have shown that the combination of DCT and TT can be used for studying the onset of plastic deformation in the bulk of polycrystalline microstructures [5] in a qualitative way, i.e.

enabling the *detection* of slip bands by inspection of image contrasts in the acquired projection images. Here we report on first attempts to reconstruct small intra-granular misorientations by means of the 6D DCT reconstruction algorithm [6][7] and discuss the potential benefit as well as remaining technical problems which prevent us from running such kind of 6D reconstructions from the joint set of DCT and TT projection images.

## 2. Material and Methods

Miniature tensile samples with a gauge section of  $\sim 600 \mu\text{m}$ , compatible with the Nanox load frame [8] have been prepared by electrical discharge machining from a plate of recrystallized Ti-7Al alloy (see [9] for details on exact composition and thermo-mechanical processing conditions of the material). A micro-speckle pattern was applied to one of the sample surfaces and the sample was characterized by SEM-DIC and EBSD before and after tensile deformation 0.4 % plastic strain (corresponding results will be reported elsewhere). The sample was then transferred to ID11 where a DCT scan with a detector pixel size of  $1.4 \mu\text{m}$  has been recorded prior to scanning a series of grains by means of topotomography using a second detector system with  $0.65 \mu\text{m}$  pixel size. The DCT scan comprised 3600 images (0.1 degree rotation increment, 1s exposure time) over a full sample rotation. The subsequent TT scans covered a base-tilt range of  $\pm 0.8$  degrees with respect to the nominal Bragg angle. For each of the 90 omega rotation positions, a continuous basetilt scan covering the full width of the crystal rocking curve (32 images in steps of 0.05 degrees) was recorded. These TT scans were acquired using a focused beam ( $200 \times 200 \mu\text{m}$ ) in order to increase the flux density in the central part of the image. The grains of interest were translated onto the center of rotation of the diffractometer. The required sample translations and goniometer tilt angles to align the grain / reflection of interest were calculated by a Matlab script, using the indexing output of the DCT scan.

## 3. Results

### 3.1. 3D - DCT reconstruction and X-ray Topographs

Figure 1 shows a 2D slice through the 3D DCT reconstruction of the sample, color-coded according to the inverse polefigure along the sample Z (vertical) direction. A total of 620 grains have been indexed in the illuminated sample volume ( $0.7 \times 0.5 \times 0.5 \text{ mm}$ ). The depicted reconstruction is based on the 3D reconstruction algorithm (i.e. each grain described by its average orientation) and no dilation post-processing has been applied to it.

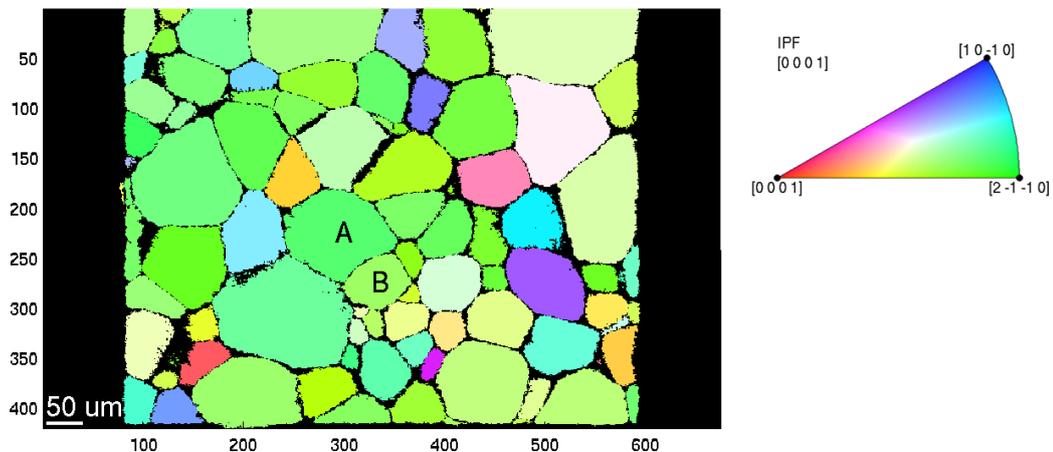


Figure 1: 2D slice ( $x$ - $z$  plane) through the DCT grain volume and corresponding IPF colour key.

Visual inspection of some of the diffraction spots of the 0.4% pre-deformed sample revealed band-like contrasts as illustrated in Figure 2. Figure 2a shows the  $[0\ 1\ -1\ 0]$  diffraction topograph of the grain labeled ‘A’ in Figure 1 and has been acquired during the DCT scan. The topograph shows a series band-like contrasts which appear to be co-linear to the trace of the diffracting lattice plane. As a guide to the eye, the red line materializes the intersection of the diffracting lattice plane with the detector.

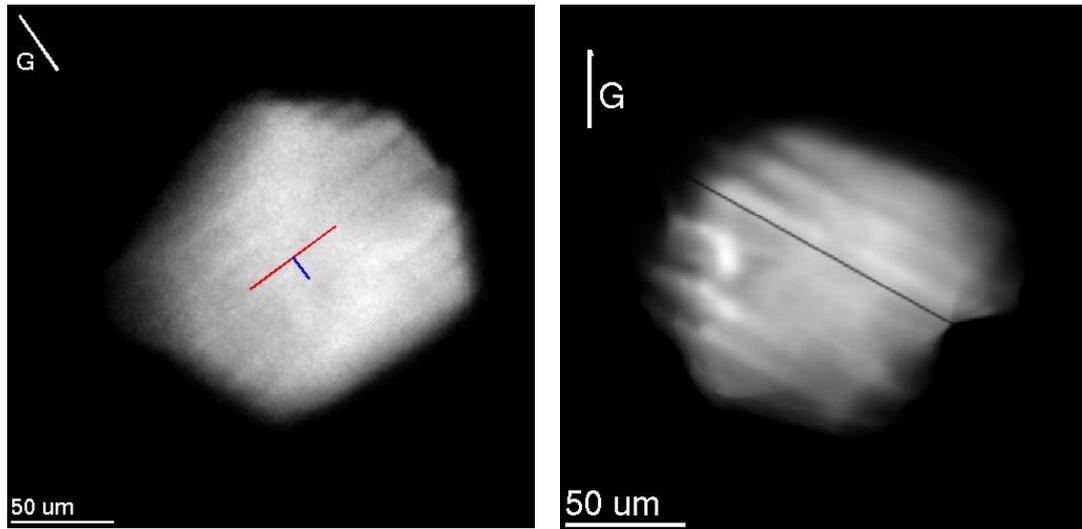


Figure 2: a)  $[0\ 1\ -1\ 0]$  diffraction topograph of the grain labeled A in Figure 1, as acquired during the DCT scan. The observed band-like diffraction contrast appears to be aligned with the trace of the diffracting lattice plane. b)  $[1\ 1\ -2\ 0]$  diffraction topograph of the same grain as acquired during the topotomography scan. The selected omega rotation angle corresponds to the position where the  $[0\ 1\ -1\ 0]$  lattice plane (highlighted in black) is observed in edge-on configuration.

Figure 2b shows the  $[1\ 1\ -2\ 0]$  diffraction spot of the same grain, as observed during the topotomographic acquisition. The projection shown has been acquired at the omega rotation position where the  $(0\ 1\ -1\ 0)$  lattice plane, (materialized as a black line) is observed in edge-on configuration (i.e. the diffracted beam is propagating along some direction contained in the  $(0\ 1\ -1\ 0)$  lattice plane).

Making the assumption that the observed intensity variations are related to orientation contrast (i.e. focusing and defocusing effects as a consequence of local variations in effective misorientation adjacent to slip bands) and ignoring the contribution of lattice strain, we have applied the 6D –DCT reconstruction algorithm [6], [10] to the neighboring grains A and B, highlighted in Figure 1. Figure 3 shows a 2D slice of the local intra-granular misorientation, derived from the 3D orientation fields inside the reconstructed grains. The top grain (A) clearly shows band-like variations in crystal orientation which are aligned with the  $(0\ 1\ -1\ 0)$  (prismatic) lattice plane (materialized by the black line). The amplitude of these orientation modulation is of order of 0.2 degrees.

The orientation field in grains A and B has been reconstructed independently. Interestingly, there seems to exist some correlation in local misorientation along the grain boundary (see for instance the boundary region highlighted by the white arrow in Figure 3), which in turn suggests that some mutual interaction in plastic deformation and strain localization between these neighboring grains has taken place and can be detected by means of DCT acquisitions.

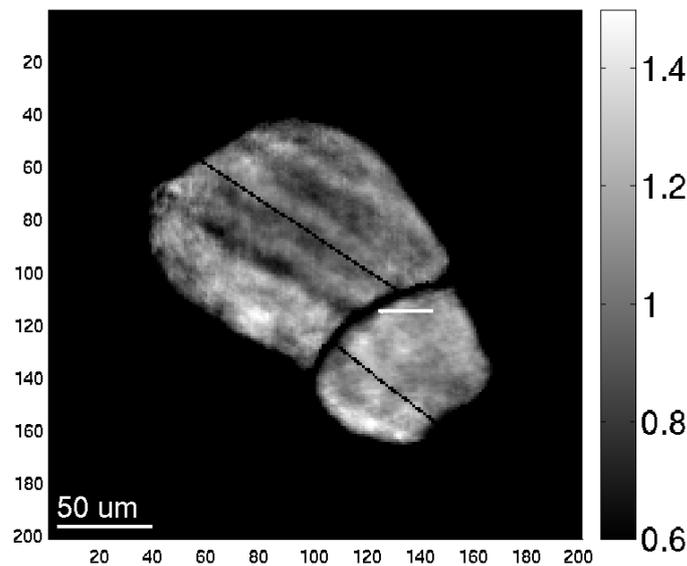
#### 4. Discussion

The orientation field reconstructed by the 6D algorithm shows band-like variations which are spatially correlated with the location of the observed slip bands in the projection topographs. However, in an isolated single crystal, activation of a single slip system would result in a simple shear of the crystal

and no misorientation between adjacent crystal blocks is expected. A plausible explanation for the observed orientation contrast would be that strain localization in the bulk of a polycrystalline aggregate leads to a locally defected crystal structure (escape of the dislocations as in the case of a free-standing crystal is impeded by the grain neighbourhood and dislocations accumulate in the regions of strain localization / primary slip bands). It may therefore be speculated that these bands act like obstacles for dislocations activated on other slip systems, which in turn may accumulate and lead to local lattice rotations in vicinity of the primary slip bands. Nevertheless, further studies are required to confirm the proposed scenario and to further elucidate the mechanisms giving rise to the observed topographic image contrasts.

Although the reconstructed orientation field shows amplitudes comparable to the values obtained in other observations [1], cross-validation with other measurement techniques will be required. First attempts to reconstruct the 6D orientation field from the topotomographic acquisition alone resulted in a rugged outline of the grain (not shown here). This could be due to the fact that topotomographic acquisition geometry lacks sensitivity for crystal rotations around the scattering vector. It may also indicate that the kinematic diffraction model may be not adequate and / or that the level of elastic strains may be non- negligible compared to the lattice rotations.

The lacking sensitivity around the scattering vector can be relatively easily compensated by formulating a joint reconstruction problem including projection data from DCT and (multiple) TT acquisitions. The mutual alignment of these projections and corrections for diffractometer error motion are technical problems to be solved for successful application of such joint reconstructions to the current data (work in progress).



*Figure 3: Intragranular misorientation in grains A and B as determined from 6D-reconstruction using a fine orientation sampling grid with 0.025 degree stepsize. The top grain shows band-like misorientation structures aligned with the  $(0\ 1\ -1\ 0)$  slip plane (highlighted in black). Correlation of the misorientation field along the grain boundary seems to indicate interaction of the plastic deformation mechanisms in both grains.*

## 5. Conclusions

Full-field 3D diffraction imaging techniques like diffraction contrast tomography and topotomography enable the visualization of slip bands formed during early stages of plastic deformation of metals.

Details of the image contrast observed in the diffraction topographs remain to be elucidated. Assuming orientation contrast as the dominant contrast mechanism, the application of the 6D reconstruction framework yields an orientation field exhibiting band-like intragranular misorientations with an amplitude of 0.2 degrees inside the reconstructed grain. It can be expected that the use of a high resolution detector system and small angular increments employed in Topotomography will ultimately yield in improved spatial resolution and orientation sensitivity, especially when DCT and (multiple TT) scans are combined into a joint reconstruction problem. In practise technical problems like diffractometer error motion and mutual scan alignment have to be overcome. We anticipate this type of joint DCT + TT reconstructions will become a valuable technique for in-situ studies of the formation and propagation of slip bands in the bulk of polycrystalline aggregates. The proposed combination of techniques may be used to provide guidance for experiments employing higher spatial resolution scanning microdiffraction and/or X-ray dark-field microscopy studies on the same sample.

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