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Strain tensor evaluation in polycrystalline materials by scanning high energy X-ray diffraction

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3rd generation high-energy synchrotron sources made possible during the last 20 years the development of several diffraction-imaging methods, which led to meaningful physical insights into the real structure and dynamics of bulk polycrystalline materials. A not exhaustive list could include the work on stress corrosion cracking (King et al., 2008), predictions of crystal plasticity (Pokharel et al., 2014), stress variation in copper through-Si vias (Levien et al., 2015), martensitic transformation (Sedmák et al., 2016) or on long-range symmetry breaking in embedded ferroelectrics (Simons et al., 2018). The pioneering work of Poulsen (2004) paved the way for the majority of the techniques using monochromatic radiation, commonly called Three Dimensional X-Ray Diffraction (3DXRD) or High Energy Diffraction Microscopy (HEDM) (Suter et al., 2006). Since then, a wide variety of techniques have been developed, which are mostly classified based on the distance between the detector and the specimen in a) far-field or b) near-field techniques. However, from the point of view of the final reconstruction describing the spatial distribution of the crystallographic orientation (implicitly the grain shape) and strain/stress, a classification based on their spatial and angular resolutions seems to be more appropriate. According to this *two-resolution criterion* all existing techniques can be included in one of the following three categories:

a) *High-spatial and low-angular resolution methods* use a near-field setup with a high resolution detector (effective pixel size of about 1 μm) closely placed downstream the specimen at a distance of a few millimeters. Their angular resolution is relatively low ($\sim 0.1^\circ$), but they can resolve the structure at micrometer/sub-micrometer length scale over millimeter sample sizes. Grain and intra-grain orientation is obtained via scans done either with a broad (Ludwig, 2008) or a planar beam (Li et al., 2013), the beam size being usually adapted to the grain size to avoid peak overlap.

b) *Low-spatial and high-angular resolution methods* involve a low resolution detector with pixel size of about 50 to 200 μm placed in the far-field (~ 0.2 to 1 m as a function of the pixel size and beam energy). Since diffraction peak positions can be determined with sub-pixel accuracy (Borbély et al., 2014) this setup has high angular resolution ($< 0.01^\circ$) that allows the elastic strain to be determined, however, the grain shape remains unknown. Measurements are usually performed with a broad beam.

c) Finally, *high-spatial and high-angular resolution methods* enable characterization of both the crystallographic orientation and strain inside single grains at sub-micrometer length scale, even for deformed materials. It is expected that these “holy grail” methods will provide the missing local experimental evidence for understanding unsolved problems in materials science such as polycrystal plasticity, recrystallization or damage initiation. There are already two methods achieving high-spatial and high-angular resolution, the Differential-Aperture X-ray Microscopy (DAXM, Larson et al. 2002) and Dark Field X-ray Microscopy (DFXM, Simons et al. 2015). The first uses a polychromatic pencil beam with sub-micron cross section for identifying the local orientation from the resulting Laue pattern and an additional energy scan (Chung & Ice, 1999) for strain determination. DFXM uses monochromatic radiation and compound refractive lenses in the diffracted beam, which magnify a small diffractive volume of the analyzed grain/subgrain.

To improve the spatial resolution of the *low-spatial & high-angular resolution* methods Hayashi et al., (2015) proposed a “scanning 3DXRD” approach, using a monochromatic pencil beam, where the spatial resolution could be controlled by the beam size. Combining a lateral scanning of the sample and tomographic approach (rotation around an axis perpendicular to the beam) the authors were able to reconstruct the variation of the crystallographic orientation inside single grains. Very recently, the method was also applied for determining local strain and stress tensors (Hektor et al., 2019, Hayshi

et al., 2019). These developments, however, considered a simplified reconstruction procedure based on the assumption that the crystallographic structure in a given voxel can be refined from the subset of diffraction peaks generated by the beams that intersect its volume. Evidently, this assumption, which could be referred to as “single crystal reconstruction” (SCR), does not obey the tomographic principle, when the recorded signal is an average response over the X-ray beam path.

The paper of Henningsson et al., (2020) in this journal issue suggests two solutions to the tomographic problem of lattice strain, both considering all voxels in the beam path. The first called “polycrystal reconstruction” (PCR) is based on the minimization of the Euclidean distance between the experimental and simulated peak centers measured on the detector. The simulation approximates the scattered intensity by assuming that the contribution of a given voxel to the measured peak is proportional to the fraction of the voxel illuminated by the beam. The second solution is inspired by the common algebraic reconstruction (ART) method, as used, for example, by Alpers et al. (2006) to reconstruct grain shapes from 3DXRD data, extended to reconstruct tensor fields of smoothly varying intragranular strain. Both the PCR and ART methods were checked against simulated data and performed better than the simple SCR method. Finally, the strain state in a columnar tin grain was determined from experimental data measured at the ID11 beam line of the ESRF using a pencil beam cross with section of $0.25\ \mu\text{m} \times 0.25\ \mu\text{m}$. The reconstructions obtained with all three methods (SCR, PCR & ART) evidence the existence of a strain gradient in the grain.

By establishing the correct tomographic approach, the paper of Henningsson et al., (2020) upgraded “scanning 3DXRD” to a viable alternative to DAXM and DFXM. Since it only involves lateral sample scanning and rotation around a single axis the technique can be easily implemented in practice, which helps its wide use. However, to gain more confidence in the results, checking of real reconstructions against other techniques (such as near-field HEDM, similarly to Renversade et al., (2016) would be valuable. Today, scan duration for representative volumes is somewhat long (12h for scanning a volume of $37^3\ \mu\text{m}^3$ with a beam size of $1\ \mu\text{m} \times 1\ \mu\text{m}$ in steps of $1\ \mu\text{m}$, Hayasi et al., 2019), however, with the advent of 4th generation synchrotron sources and fast detectors the scanning time can be reduced by one order of magnitude, which already allows in situ studies to be performed during common beam time duration.

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