Scanning precession electron diffraction tilt series for orientation analysis

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Identifying the orientation of crystalline phases at the nanometer scale is relevant for understanding materials properties. Here we will demonstrate that collecting and processing scanning precession electron diffraction (SPED) datasets collected at a few different specimen tilts can improve the accuracy of template-based orientation mapping [1,2]. In addition, the tilt series allows complete determination of the relation between the specimen crystallographic setting and the goniometer axes. This insight, combined with the orientation map, can be used in a convenient semi-automatic approach to predict the tilts required to reach a target specimen orientation for further structure analysis.

SPED of different polycrystalline systems (Si, Ag, and oxides) were recorded in a JEOL JEM2100F with a NanoMegas DigiStar precession system and a Quantum Detectors MerlinEM direct electron detector. Scans were taken over areas up to 15 x 15 µm containing 10's of grains using a nominal precession angle of 1˚. Tilts series using one or two axes contained 3-5 tilts in the range 0 - 20˚ from the initial flat specimen position. For data analysis and visualization, we used primarily the open-source python library pyxem [3].

The indexed frames, taken at different tilts, were compared, after manually aligning the frames, using the set tilt as expected misorientation. Together with considering as well the best 5 to 25 normalized cross correlations between the experimental patterns and the simulated pattern bank, orientation-dependent misindexations can be identified and the orientation estimate refined. Compared to the standard approach of collecting SPED using only a single specimen tilt and the best correlation scores for each pattern, the tilt series approach reduces indexation variations within grains. This gives a more uniform representation of the grains in the final orientation maps.

The accuracy of the refined orientation analysis can be determined with a known orientation relation, here for example Σ3 twins in face-centered cubic and diamond crystal systems. The misorientation deviation between the measured and expected misorientation between twin domains is used as the metric [4]. The found accuracy is below the used precession angle.

The refined orientation mapping based on a small tilt series has a further practical use. From a single axis tilt series, the position of the two perpendicular tilt axes can be determined. As the tilt series is small and over a limited angular range, sufficient probe positions must be used to accurately determine the tilt axis position. Misaligned between frames and areas with overlap, such as grain boundary areas, were excluded through thresholding. A second tilt axis series or grains correctly indexed in different frames can be used to verify the found axes positions. Using the determined orientation of a grain together with the deduced axes positions, the tilts to reach a target zone for a grain can be predicted. Based on tests on different TEMs and holders, the target zone was within 2˚. In the tests the specimen was placed at approximately the same rotation relative to the holder axes. However, should the specimen be differently placed compared to where the orientation was mapped out, an additional transformation matrix can be included in the navigation tool to recalculate the target tilts for the given specimen placing. This correction is based on a manual estimation of misorientation from the tilts to the actual target zone, diffraction (using the Laue circle), or imaging (assuming in-plane rotation).

To conclude, template matching based on multiple SPED scans at a few varying specimen tilts improves the accuracy and the final orientation visualization. In addition, the approach is used to make a practical navigator tool that widens to use of template matching results for subsequent lattice imaging and further crystallographic analysis. With the advancements in automatic scan controls, faster detectors and optimized transparent open-source routines, the benefits gained will more than compensate for the drawbacks of acquiring and processing multiple scans.

Keywords:

Texture, diffraction, open-source, template-matching, ACOM

Reference:

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