Inclined scanning 3DXRD microscopy for the high-resolution 3D grain orientation mapping

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Keywords: orientation microscopy, 3DXRD, inclined scanning 3DXRD

Understanding the microstructure of polycrystalline material is essential to accurately predict and optimize the materials’ properties. A comprehensive understanding of grain size, grain boundaries, and defect distribution can help predict and optimize the performance, as well as prevent the malfunction of material. Studies have demonstrated that the mechanical strength of polycrystalline metals increases as grain size decreases to a critical value. This is due to the grain boundaries acting as barriers to dislocation propagation and fatigue crack growth, known as the Hall-Petch relation. Other research on the effect of misorientation angle on grain boundary energy has found that under specific angles and axes, the grains form a coincident site. At a low degree of coincidence, grain boundary energy is lower, resulting in improved physical and chemical properties. Those cases give insight into the importance of understanding microstructure in terms of controlling mechanical, and chemical properties.

In recent decades, methods based on x-ray diffraction have proven successful in obtaining spatial maps of the grain orientation within polycrystalline materials. The development of orientation microscopy techniques, including differential aperture x-ray microscopy (DAXM), diffraction contrast tomography (DCT), three-dimensional x-ray diffraction microscopy (3DXRD), and high-energy diffraction microscopy (HEDM), has been accelerated with the help of high-brilliance synchrotron x-rays. Among these methods, scanning 3DXRD microscopy [1, 2] is particularly useful for thick specimens. The scanning 3DXRD uses a point-focused or pencil beam to illuminate a sample, and the diffraction spots are captured through a 3D scan that involves sample rotation. The 3D grain maps are created through voxel-by-voxel multigrain indexing, with each voxel representing a regular grid in three dimensions. The use of a conical slit in the detection of diffracted beams reduces the overlap of diffraction spots by selectively shielding diffracted beams from many grains, except for those from grains in the area of interest. As a result, the scanning 3DXRD is applicable even for thick samples with numerous grains.

A straightforward approach to improve the spatial resolution in scanning 3DXRD is to use brighter x-ray sources, which can increase the intensity of the diffracted x-rays and enhances the signal-to-noise ratio, making it easier to distinguish between different diffraction peaks in the diffraction spots. Furthermore, optimizing the x-ray optics and reducing the detector pixel size can also contribute to the resolution enhancement. Those two approaches enable to enhance the quality of diffraction patterns in the diffraction pattern measurement process, while advanced data analysis techniques achieving high resolution have yet to be explored in the field of scanning 3DXRD microscopy. Here we present a new method, called the inclined scanning 3DXRD, which enhances the spatial resolution by subdividing a voxel into small sub-voxel pieces. This method involves inclining the sample rotation axis similar to x-ray laminography, thereby obtaining geometrical functions for sub-voxel analysis in all sample coordinates. A synchrotron x-ray-based inclined scanning 3DXRD experiment on a polycrystalline α-Fe wire with a sample translation of 10 µm showed a grain microstructure with a sub-voxel resolution of 1.25 µm. The simulation results were in agreement with the experiment, suggesting that the proposed method can significantly improve the spatial resolution of the 3D grain orientation map.