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**3D grain orientation mapping of polycrystals on scales from 1 mm to 1 nm using 3D-XRD and TEM**

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Polycrystals are composed of millions of small crystals (grains). The orientations, shapes, sizes and relative arrangement of these grains are important in determining many material properties. Traditionally, it has only been possible to obtain information on these structural parameters by looking at a cut surface using two-dimensional characterization techniques, for example optical microscopy and electron microscopy. We have developed a set of techniques for three-dimensional (3D) grain orientation mapping of polycrystals and here we provide an overview of these techniques.

For millimetre size specimens the characterisation is based on diffraction by x-rays generated by a synchrotron source. The technique, Three-Dimensional X-Ray Diffraction (3DXRD) microscopy, allows several modes of operation, including grain morphology mapping and grain center mapping. In the grain morphology mapping mode, 3D grain maps with more than 1000 grains are made routinely, with a spatial resolution of 1-5 µm. In the grain center mapping mode, each grain is characterized with respect to center-of-mass position, volume, and its average orientation. At present the positional accuracy is ~2 µm.

Recently we have succeeded in developing a new technique, 3D Orientation Mapping in the Transmission Electron Microscope (3D-OMiTEM), for 3D grain orientation mapping in nanocrystalline materials that have grain sizes substantially smaller than the thickness of TEM foil specimens. The data collection is based on the concept of conical scanning dark field imaging; hence images are acquired over a wide range of beam tilts. In addition, to enable a 3D reconstruction of the grains, the acquisition procedure is repeated for many sample tilts. The reconstruction software is a variant of the algorithm originally developed for orientation mapping with 3DXRD.