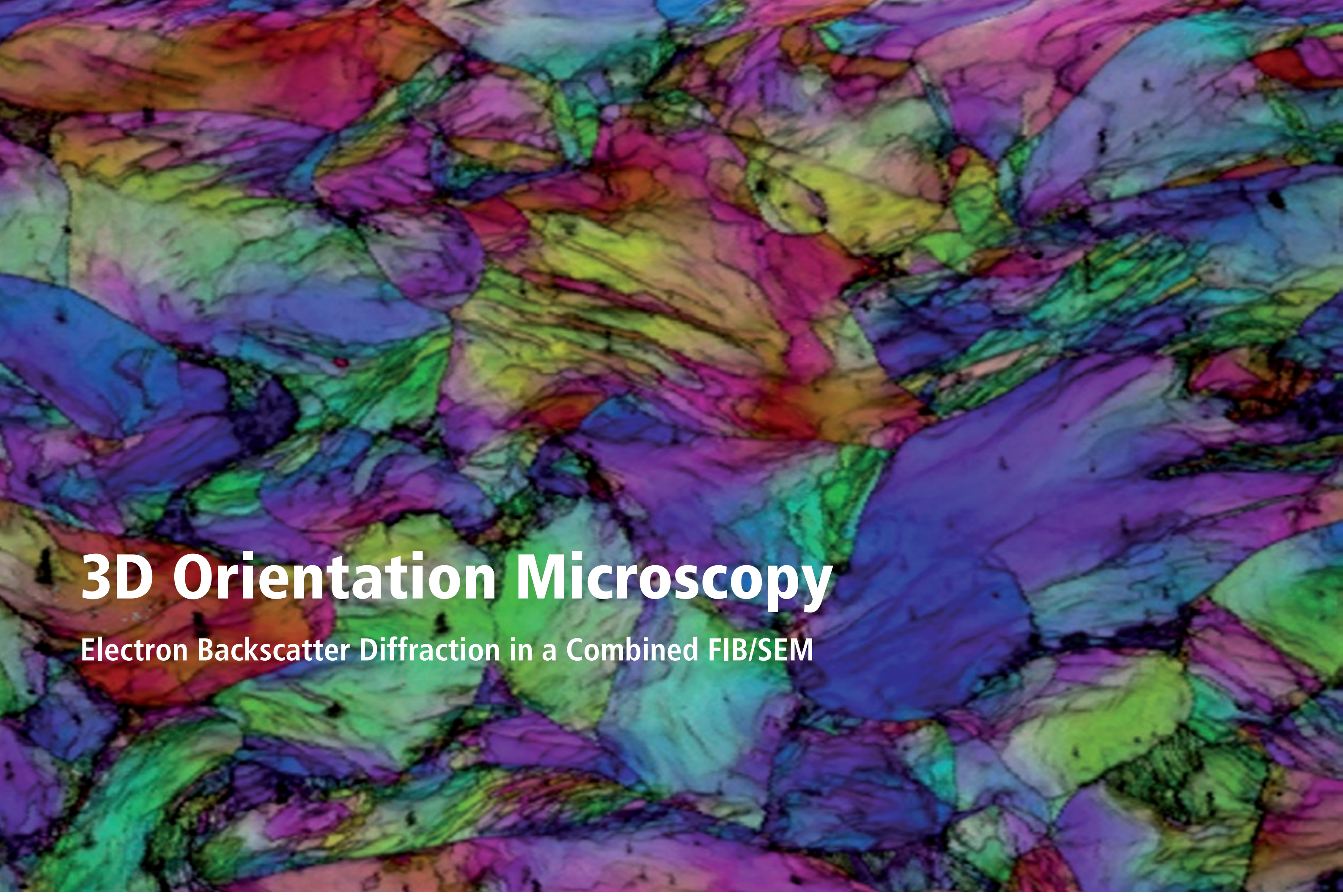

3D Orientation Microscopy

Electron Backscatter Diffraction in a Combined FIB/SEM

Combining electron backscatter diffraction (EBSD), a scanning electron microscope (SEM) and a focused ion beam (FIB) together into a single instrument enables three dimensional (3D) characterization of microstructure in crystalline materials. Combining these techniques together has enormous potential in materials science.


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Keywords:

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Electron Backscatter Diffraction

Traditionally, microstructure refers to features that are visually evident in an optical or electron microscope. However, many critical aspects of microstructure are not visually evident. For example, the crystallographic orientation of the constituent grains can not be observed in basic microscopic imaging. While some indirect evidence may be observed, other techniques are required to gain true quantitative information. If we consider the microstructure at the scale revealed in the SEM then the crystallographic orientation is best characterized using EBSD. An EBSD pattern is formed when a stationary electron beam is focused on a highly-tilted and properly-prepared sample in the SEM. Electrons are scattered as the incident electron beam interacts with the crystal lattice within the sample. Electrons satisfying physical laws governing the interaction of electrons with the crystal lattice are coherently diffracted out of the sample. The diffracted electrons form a pattern on a phosphor screen positioned near the sample. The pattern provides information about the structure of the crystal lattice within the interaction volume such as the orientation of the crystal lattice with respect to the sample reference frame. Orientation microscopy [1] is a fully automated technique where the patterns are imaged using a CCD camera and then analyzed to determine the corresponding orientation. The system controls the electron beam enabling the procedure to be repeated at each point on a scan grid superposed over the sample. By mapping the measured orientations to various color scales it is possible to visualize orientation aspects of the micro-

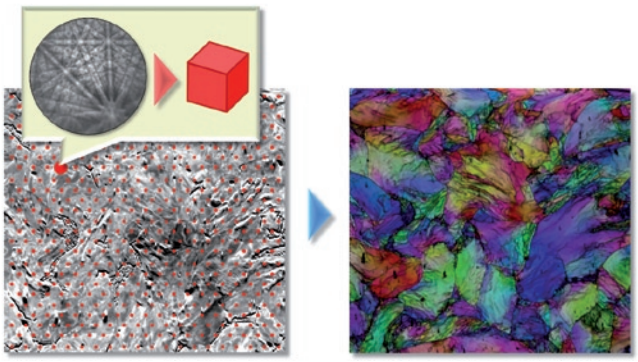

Fig. 1: A schematic showing how EBSD is used to obtain orientation from a crystalline material and an example of an orientation map generated from the data.

structure. Orientation microscopy has proven to be a valuable tool for characterizing crystalline materials as evidenced by the large number of papers published where the technique has been used in the research.

3D Data Acquistion

As with traditional metallographic techniques, EBSD is usually performed on 2D planes cut through a sample. 3D microstructural characterization can be achieved through serial sectioning. The technique simply comprises the cutting of material slices, recording of the structure of these slices and then reconstructing the 3D structure by stacking of the recorded images. Serial sectioning is applicable to a wide range of materials and material problems with the only serious disadvantage that it is destructive. For serial sectioning a large number of different methods can be imagined, e.g. mechanical cutting, grinding or polishing, chemical polishing, or laser or electrical discharge ablation. The main challenge associated with these methods is controlling the sectioning depth, obtaining flat and parallel surfaces and correctly redetecting and aligning the observation area. Also, these serial sectioning methods tend to be extremely laborious. A technique that avoids these problems is serial sectioning with an ion beam in a combined FIB-SEM. The impact of the ion beam onto the sample leads to localized removal of the target material and can therefore be used to mill parallel serial sections through the material a few nanometers in depth. Irradiating a material surface in grazing incidence allows preparation of smooth surfaces that show little damage [2]. Recent progress has led to a fully automated technique for alternately sectioning and subsequent scanning of the new section using EBSD [3–5].

Data Visualization

Software for analyzing and visualizing the wealth of information in the 3D EBSD data is still in its infancy. Basic functions based on the analysis of 2D EBSD data are being extended into 3D [6]. Visualization software is being built upon existing tools developed for general 3D rendering. Functions for rotating and slicing through the data cube as well as the ability to extract individual grains from the 3D data have been developed. Grain facets can be colored according to their orientation relative to the associated crystal lattice of the grain [7]. Figures 3 and 4 show examples from steel and nickel.

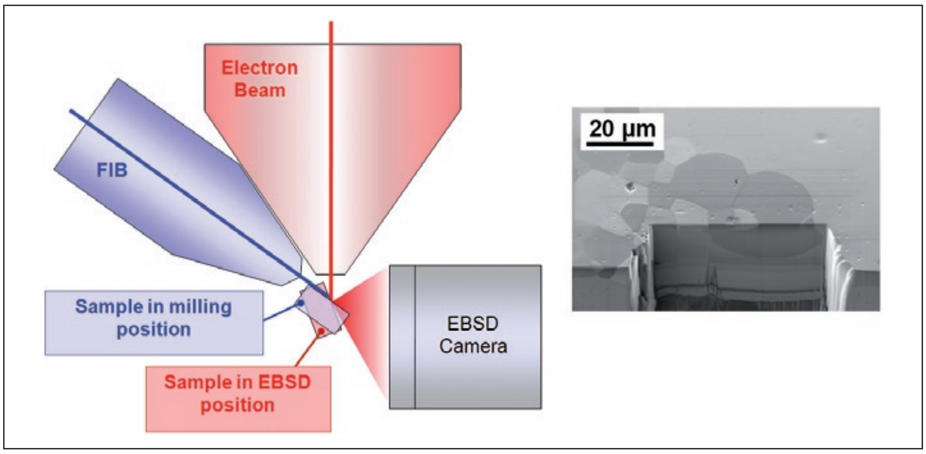

Fig. 2: Schematic of the instrument combining the EBSD, SEM and FIB techniques and micrograph of a milled section.

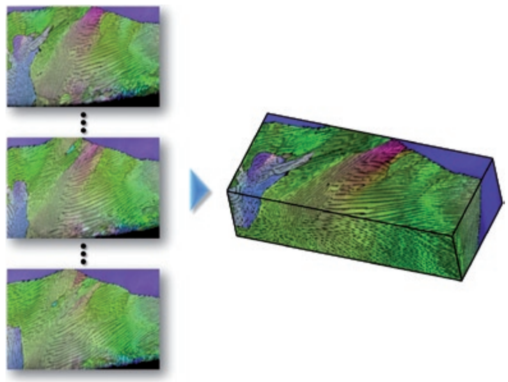

Fig. 3: A 3D EBSD scan from pearlite structure in a steel sample showing a 3D data cube reconstructed from EBSD scans on serial sections.

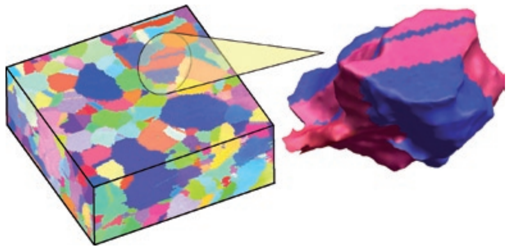

Fig. 4: A 3D EBSD scan from a nickel sample and a twinned grain extracted from the data and rendered in 3D.

Conclusions

A system for 3D orientation microscopy based on fully automated serial sectioning and EBSD based orientation microscopy has been developed in a FIB-SEM. The technique yields the crystal orientation at each voxel of the measured volume. A spatial resolution 50 x 50 x 50 nm^3 can be achieved. Volumes on the order of 50 x 50 x 50 μm^3 can be practically observed. The technique works on a wide variety of materials but some exceptions have been found, namely materials that adversely react to the Ga+-ion beam irradiation. The technique has been applied to several different materials and has yielded information that gives insight into microstructures that cannot be realized without the 3rd dimension.

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Further references are available from the authors.

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