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EBSD MICROSTRUCTURE EXAMINATION USING SEM

The electron backscatter diffraction (EBSD) technique – a powerful tool to study microstructures by SEM (scanning electron microscopy)

In the field of scanning electron microscopy (SEM) the electron backscatter diffraction (EBSD) technique has developed into a powerful tool for the crystallographic analysis of materials. In particular, the emergence of computer algorithms for the fully automated analysis of diffraction patterns have pushed the technique to develop into a new kind of scanning microscopy technique, known as “orientation imaging microscopy, OIM*” [1] or “crystal orientation microscopy/mapping, COM”. It uses computer algorithms for the automated analysis of the diffraction patterns. The COM technique is based on the consecutive acquisition of electron diffraction patterns obtained from every point of a scan grid on a flat surface of a steeply inclined sample in the SEM. The automatic analysis of these EBSD patterns yields, for every scan point, the crystallographic orientation and phase and a value indicating the quality of the diffraction pattern. From these data, the microstructure of the scanned area can be reconstructed. The resulting crystal orientation maps give a vast amount of information on the sample, including kind and distribution of different phases, size, form and defect condition of grains, kind and position of grain boundaries, local crystal orientation and misorientation distribution (texture) and others more. Furthermore, EBSD can be used to investigate the structure of crystals, i.e. lattice symmetry and lattice parameters. With dedicated software, even details of the atomic lattice [2] and residual stresses [3] may be determined. The EBSD technique allows observation of bulk samples, i.e. no thin foils as in the case of TEM are required, with an acceptably high spatial resolution of about 50 nm. Sample preparation usually is less complicated than TEM and consists of an accurate mechanical, chemical, or ion-assisted polishing with the aim of producing a flat and defect free surface. A comprehensive overview on the state of the art of the technique is given by the book of Schwartz et al. [4]. A very recent overview will be shortly published by [5].

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*The term “OIM” is nowadays used as registered trade mark of TSL/EDAX

Electron backscatter diffraction patterns which are obtained from bulk samples have lots of similarities with Kikuchi diffraction patterns obtained from thin foils in the transmission electron microscope (TEM). Figure 1 displays a typical pattern: It consists of bright bands (the so-called Kikuchi bands), on a relatively strong background.

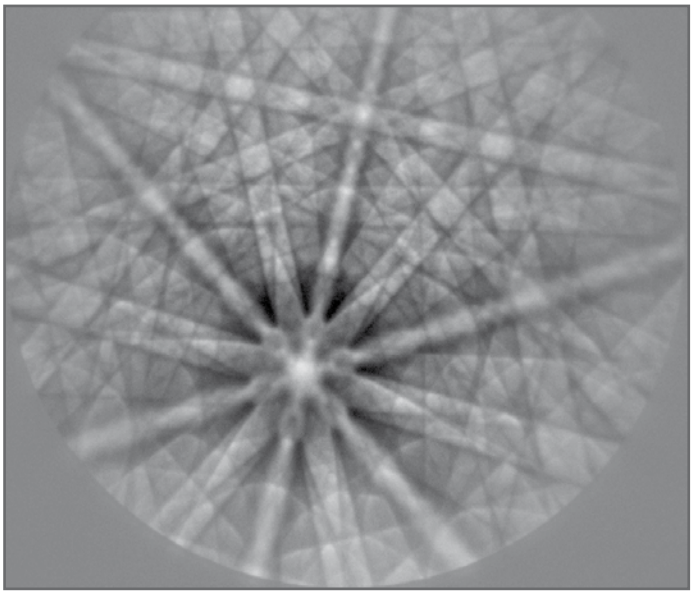


Figure 1: High-resolution EBSD pattern from an as-cast niobium sample (915 x 915 pixel). Exposure time 7 s, with background subtraction.

Each of the bands corresponds to a set of lattice planes in the crystal and from the angles between the bands and from their width the Miller indices of these lattice planes can be determined. Finally, from indexed bands the crystal orientation and phase can be calculated. The acquisition of patterns is performed using a detector which consists of a phosphor screen observed by a highly light-sensitive camera positioned in close distance to the sample (20 to 40 mm) as this is shown in Figure 2.

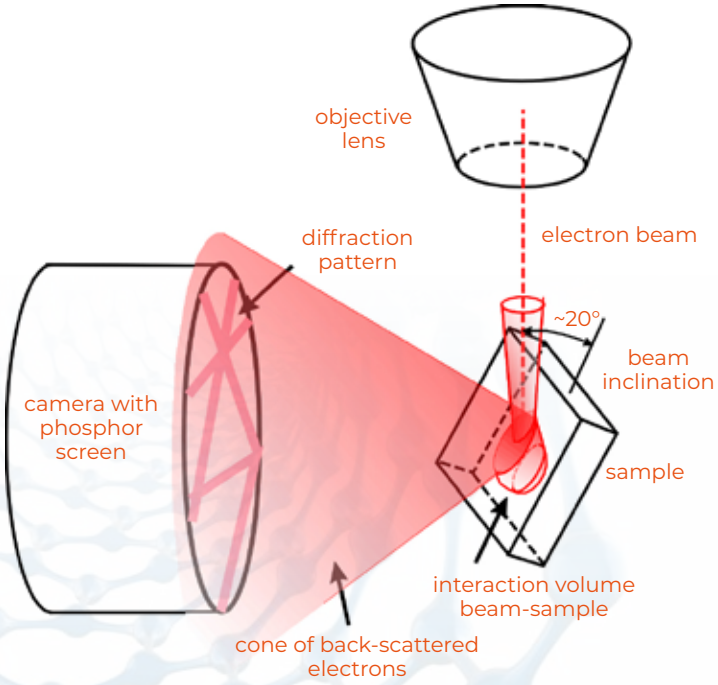
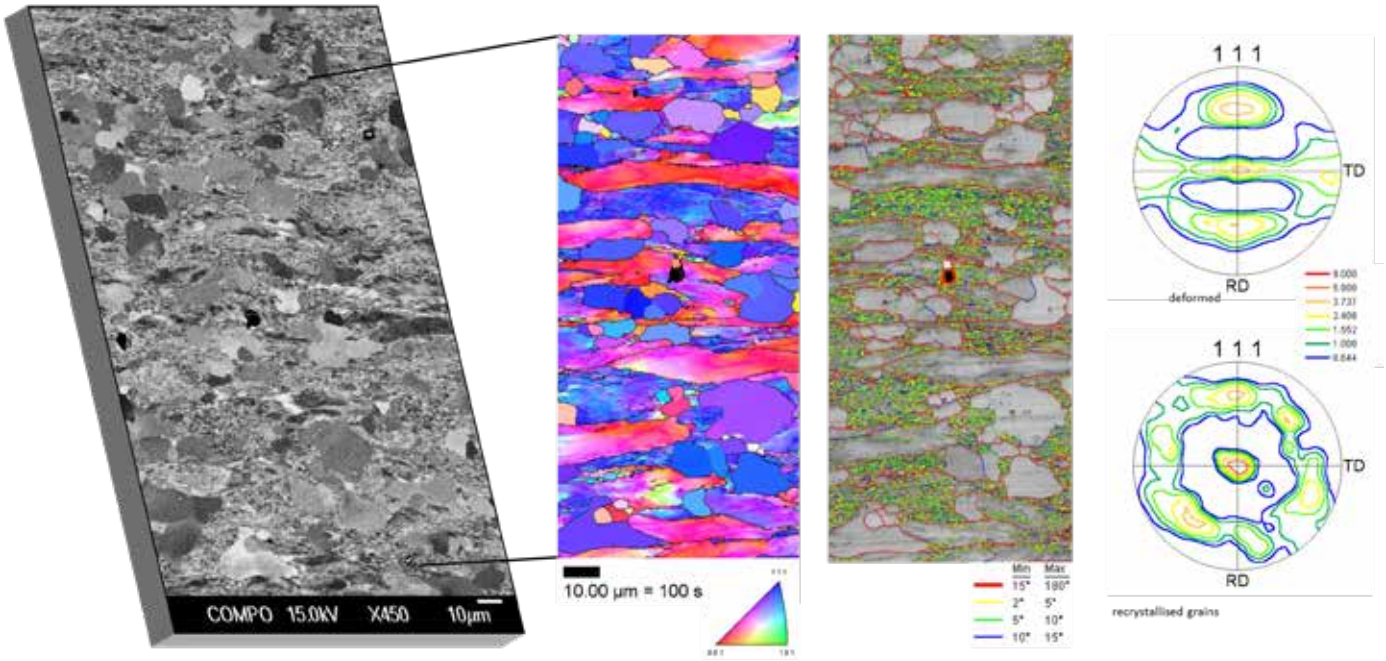


Figure 2: Setup of sample and detector within a scanning electron microscope (SEM).

The detection of band position and the subsequent analysis is done by fully automated and commercially available software.

For COM the electron beam is moved stepwise over the sample and at every position a pattern is acquired and analysed. Measurement and pattern analysis time is nowadays in the order of 50 to 200 patterns per second. A typical result obtained by COM is presented in Figure 3.



BSEI of a partially recrystallised IF steel

Inverse pole figure map

Grain boundary character

Pole figures

Figure 3: Example of ACOM on a microstructure of a partially recrystallised IF steel. a) Electron channelling contrast image obtained with the backscattered electron (BSE) detector from the untilted sample. b) Diffraction pattern quality map of the observed area. c) Crystal direction map indicating the crystal directions pointing parallel to the sample normal direction (ND). Grain boundaries with misorientation larger than 15° are marked as black lines. d) (111) pole figures of the deformed and recrystallised partition of the sample, showing that recrystallisation strongly supports crystal orientations with (111) || ND (centre of pole figure). (Courtesy of I. Thomas)

Figure 3 shows the microstructure of a partially recrystallised IF steel ([6]) as observed by electron channelling contrast and by COM. In the crystal orientation map similar colours stand for similar crystallographic directions pointing towards the sample normal. Grains with uniform orientations (i.e. recrystallised grains) appear in uniform colour while grains with internal orientation gradients show changing colours. In this way grain form and size, recrystallised and non-recrystallized areas, type of grain boundaries and the local texture (here displayed in form of pole figures separated for deformed and recrystallised areas) can be easily recognised and quantified.

The preparation is done very gentle for achieving a polished surface without deformation. A special mechanism for the generation of the vibrations yields optimal energy transmission onto the specimen. In combination with the robust design made by ATM Qness GmbH the vibratory motor assures silent operation.

As deformation and its associated layers underneath the surface distort the microstructure a preparation serving a polished surface without artefacts is of high importance. A preparation following a very gentle material removal achieves this ambitious aim. This can be achieved using a vibratory polishing device as shown in Figure 4.

Figure 4: Vibratory polishing device of ATM Qness GmbH: „Qpol Vibro“.

Following that kind of approach avoids almost any kind of deformation by using appropriate preparation settings. The material removal is done by combining fine-grained polishing suspension and polishing cloth together in a vibratory bowl. Consequently, the creation of artefacts is suppressed and in principle a polished surface without deformation can be examined. The results of such an examination are reliable and reproducible, which is of high importance for studies close to materialographic limits.

References

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