Sample Orientation for Electron Channeling Contrast Imaging

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Electron channeling contrast imaging (ECCI) is an SEM technique in which the variation of intensity of electrons backscattered from the sample surface allows visualization and quantification of crystalline defects such as dislocations, stacking faults, or grain boundaries [1]. ECCI makes use of the strong dependence of the backscatter electron intensity on the orientation of the crystal lattice planes with respect to the incident electron beam. Electron channeling contrast of defects is the strongest when sample is oriented close to the Bragg condition for a selected set of diffracting lattice planes. Small deviations from precise orientation leads to the decrease of the defect contrast [2]. Therefore accurate sample orientation with better than 0.1° precision is the key for successful defects characterization and metrology [3].

In order to achieve best visualization of defects in SEM it is essential to have information of the orientation of the region of interest with respect to the incident electron beam − an equivalent of a diffraction pattern, and be able to change this orientation in the controlled way. There are several ways to obtain crystallographic information from the sample. First and the most straightforward one is applied to the monocrystalline samples of large diameter ($>10^0$ mm). By simply scanning the large area of the sample relative beam orientation towards the sample is varied, producing an electron channeling pattern (ECP). This method does not require specialized devices or advanced scanning electron microscope optics settings. Moreover such a pattern can be easily obtained on samples with patterned structures on monocrystalline substrate. Typical example of application of this technique is illustrated in Figures 1a b, c, showing ECCI of threading dislocations in $Si_{0.3}Ge_{0.7}$ blanket layer. Images were recorded under controlled diffraction conditions using two opposite diffraction vectors. Inversion of the defects contrast is easily noticeable. In the case of the large area scanning ECP usually covers about 4 degrees that is reasonable for analysis of samples of known surface orientation, e.g. semiconductors wafers. In the case the ECCI technique should be applied for defect analysis in polycrystalline samples the site specific information is required. Common way to generate ECP from a localized region of interest is by rocking the primary beam through a pivot point located on the sample surface. This requires special microscope settings that enable rocking of the primary beam [4]. Beam rocking in the range of 10 to 20 degrees covers reasonable part of the reciprocal space to find and setup appropriate diffraction conditions. Typical ECP obtained using beam rocking on the $Si_{0.3}Ge_{0.7}$ blanket layer is shown on Figure 1d. Modern SEM and FIB/SEM systems can achieve routinely spatial resolution below 10 µm without special optics settings. The first step in the workflow is to find the grain of interest and optimize beam parameters for high resolution defect visualization [1]. Then, SEM is switched to ECP mode in order to determine the crystallographic orientation of the grain of interest. Using the acquired ECP, sample is then tilted to the desired crystallographic orientation making sure that the set of the selected crystallographic planes is in Bragg diffraction condition. Finally, SEM is switched back to imaging mode to acquire ECCI image of defects under selected diffraction conditions. An example of dislocations visualized under controlled diffraction conditions in a polycrystalline Fe-Cr alloy is shown in Figure 2. Alternatives to the beam rocking method employ other techniques, such as EBSD, for example [1], however with some limitation on the technique accuracy. The future developments for obtaining sample orientation does not require

change of the optical mode of the microscope from standard imaging to beam rocking, or complicated stage manipulations as when using the conventional EBSD camera. This novel approach is facilitated by using the tilt-free EBSD detector [5]. Such detector is mounted directly below the lens and its operation is based on high sensitivity pixelated semiconductor device. Example of the diffraction pattern acquired on the $Si_{0.3}Ge_{0.7}$ blanket layer is shown in Figure 1d. There are multiple advantages for this approach, one of them being the possibility of using the extended sample tilt range thus covering larger area in the reciprocal space for the extended analysis of the crystalline defects.

References
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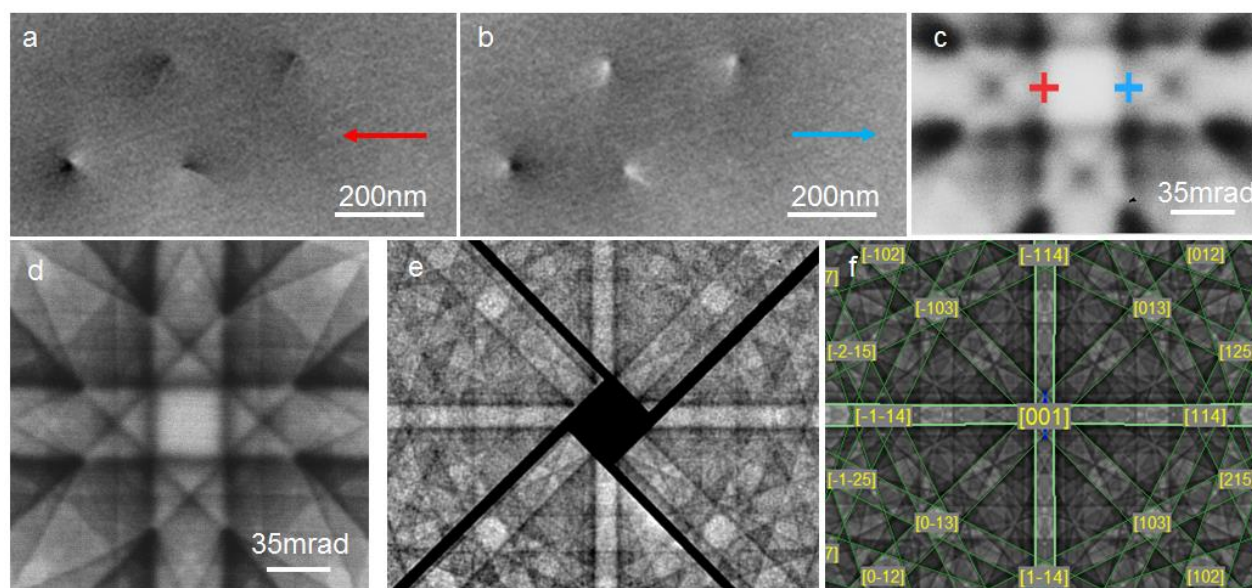[5] T. Vystavel et al, (2018), Microsc. Microanal., Vol 24(S1), pp 1126-1127

Figure 1. ECCI at the very same location of $Si_{0.3}Ge_{0.7}$ blanket layer a) at $g = (\overline{2}20)$, b) at $g = (220)$, c) corresponding ECP acquired by scanning over large area, red and blue cross corresponds to orientation used for ECCI, d) ECP obtained by beam rocking, e) EBSD pattern acquired by tilt-free EBSD technique, f) simulated tilt-free EBSD. pattern.
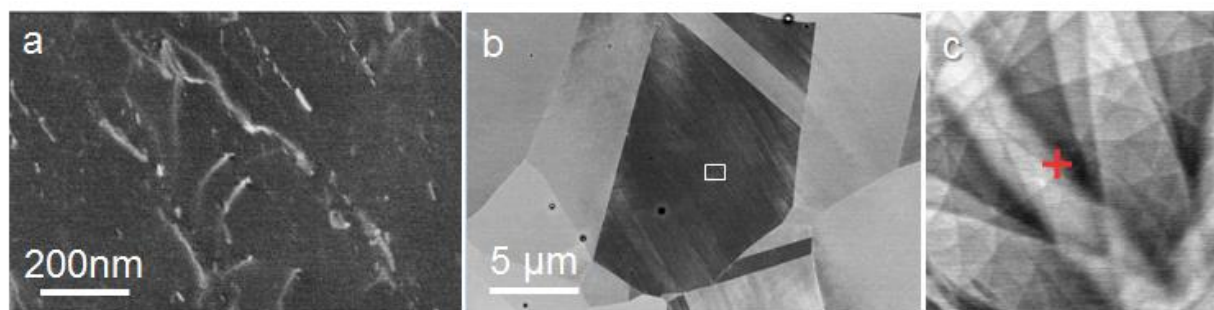

Figure 2. a) ECCI image of Fe-Cr alloy from inset shown on b) demonstrates dislocations contrast taken at g=200, ECCI conditions set by ECP c) via beam rocking acquired from middle grain from figure b).