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Combined Application of EBSD and ECCI Using a Versatile 5-Axes Goniometer in an SEM.

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Electron backscatter diffraction (EBSD) and electron channelling contrast imaging (ECCI) are two complementary diffraction techniques used to characterize microstructures of crystalline materials in a scanning electron microscope (SEM). EBSD allows the accurate measurement and mapping of crystal orientations and crystallographic phases. ECCI in contrast, is applied to observe individual lattice defects like dislocations, stacking faults and nano twins in the same manner as in TEM dark field images. Furthermore, ECCI can be used to image elastic strain fields with high accuracy. As in TEM optimum lattice defect contrast is obtained only if the sample is oriented in so-called two-beam conditions, when only one set of crystal lattice planes is in Bragg position with respect to the primary electron beam. We call the resulting technique 'ECCI under controlled diffraction conditions', cECCI. To obtain proper diffraction conditions one can either use electron channeling patterns (ECP) or one has to calculate the position from the orientation determined by EBSD. The first option is available only on very few modern microscopes and is strongly limited by the large spatial resolution of ECP. The second option is therefore the much more versatile selection. We use the computer program TOCA [1] to simulate, based on EBSD-measured orientations, ECPs for the position under observation. From the simulated patterns the correct tilt angles are determined and ECC can be observed [2]. Figure 1 shows the arrangement of the different techniques inside of the SEM. Since ECCI has a very high angular sensitivity (the backscattered electron intensity obtained from a given crystal position may change from maximum to minimum over 0.5° of lattice tilt) it is very important to control the tilt and rotation angles and observation position in a very accurate manner. To this end we constructed a small, highly accurate 5 axes sample stage with the following features (see figure 2 for a photo of the stage):

(i) It comprises x,y,z, eucentric tilt and rotation axes. The translation axes have an accuracy of 2 nm, the angular axes of 0.1°. The angular axes are encoded.
(ii) It enables operation under short working distance, down to approximately 7 mm.
(iii) It keeps the sample in perfect eucentric position, even at large tilt angles
(iv) It Reaches tilt angles of up to ±90° and provides unlimited rotation.

This stage has been mounted in a Zeiss field emission gun SEM (XB1530) with Gemini column which, due to its small beam convergence at high beam current, is excellently suited for this technique. The sample can be easily and accurately moved between the EBSD position (sample at 70° tilt) and ECCI position (sample at up to ±25° tilt) whereby keeping the observation position well in focus. Figure 3 shows an ECCI image of dislocations in a steel sample obtained in the indicated manner before and after straining the sample in an ex-situ experiment.

References:

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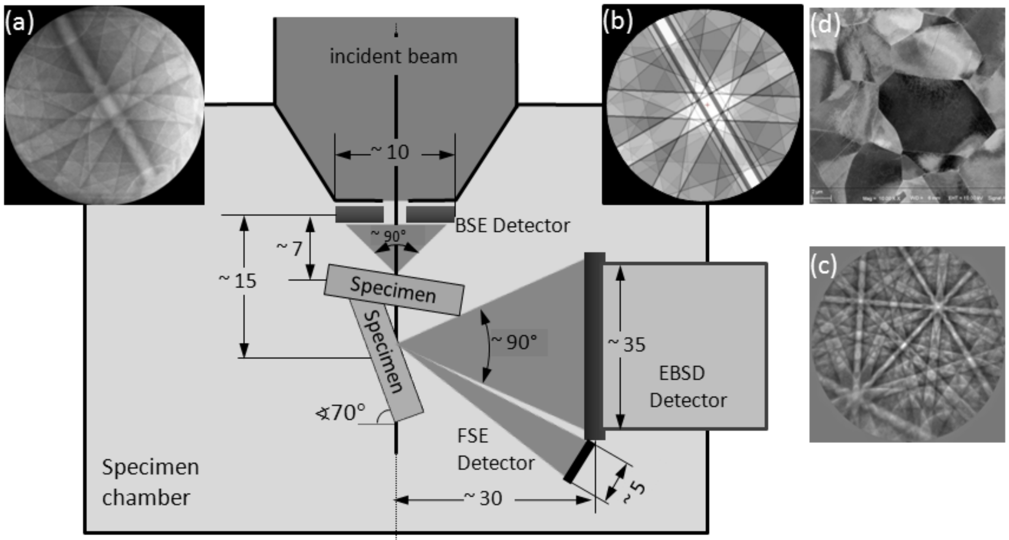

Figure 1. Overview on position of sample and detector arrangement required to perform combined EBSD-ECCI observations. Values indicate distances in mm. (a) Electron channeling patterns are replaced by simulated channeling patterns (b), which are calculated from EBSD patterns (c). After positioning the sample below the BSE detector, ECCI can be performed (d).

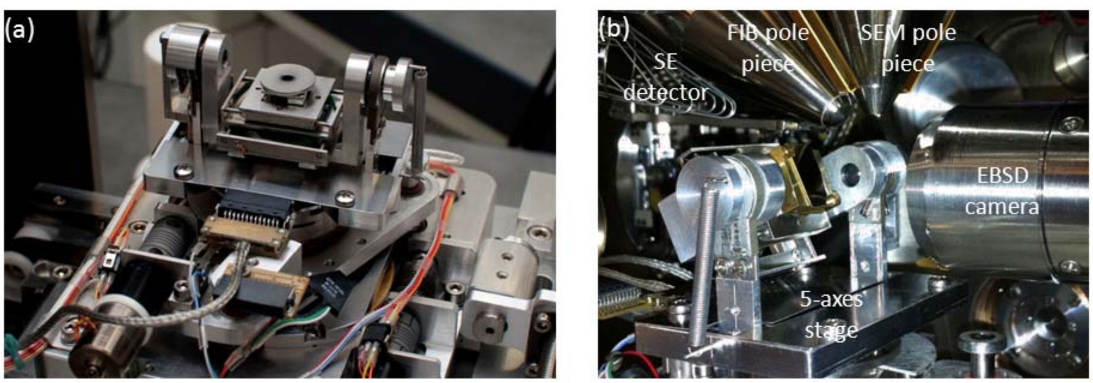

Figure 2. Two views of the new 5-axes sample goniometers. (a) Mounted on the SEM stage, (b) Position inside the SEM with the EBSD detector being inserted.

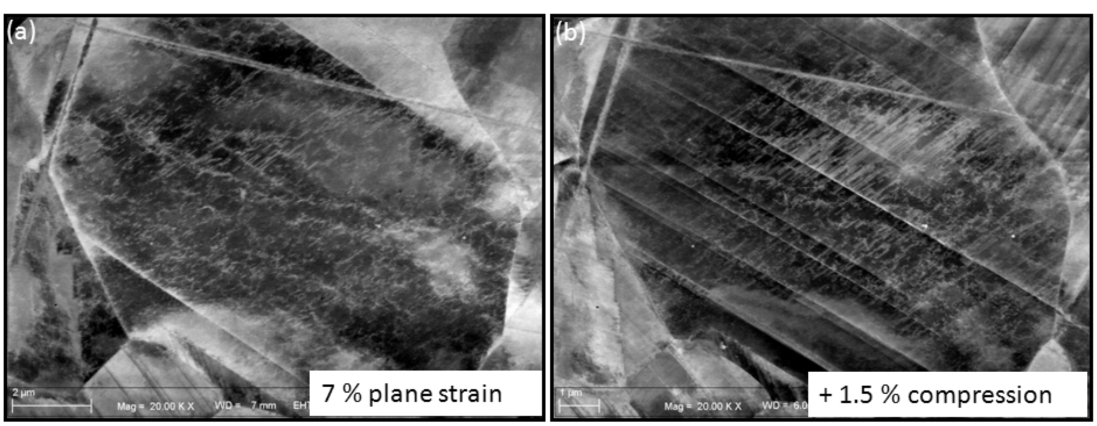

Figure 3. ECCI of a grain in a TWIP steel. (a) After 7 % of plane strain, (b) after an additional 1.5 % of simple compression (outside of the microscope). The formation of secondary twins and stacking faults is observed.

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