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Complementarity of On-Axis Transmission Kikuchi Diffraction and Forward Scatter Diffraction Imaging in SEM

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The recent Transmission Kikuchi Diffraction technique (TKD) produces orientation maps of the microstructure of materials at the nanometric scale in SEMs, via the automated recording and analysis of Kikuchi diffraction patterns generated in transmission on electron-transparent samples. Grains as small as 10 nm can be resolved in orientation maps [1,2], making the TKD technique much more suitable than EBSD for the study of fine microstructures. Two main geometries exist as of now, conventional TKD and on-axis TKD. While the spatial resolutions of the two geometries are expected to be similar, the latter was developed at LEM3 with the objective to decrease drastically the acquisition time in comparison to the former, via a scintillator sitting in the direction of highest transmitted intensity [3]. A factor 20 in the difference of acquisition speed was evidenced between the two geometries [4].

Besides a phosphor screen associated to a camera for the recording of the diffraction patterns, the on-axis TKD detector head OPTIMUS™ from Bruker also features a set of three diodes which can be used for imaging, namely for FSD imaging (Forward Scatter Diffraction). These diodes can be used to produce images in transmission, according to the same physical principles as with a STEM detector. They proved very useful and complementary to the TKD orientation mapping in many experiments.

First, FSD imaging can be used to focus the beam on the sample. Samples for TKD often lack features on the entrance surface, making the focus unreliable with secondary electrons. Besides, in the case of convergent probes, it is not even sure that focusing on the entrance surface optimizes the lateral resolution because the source volume for TKD is near the exit surface [5]. Maybe the focus should be made at the bottom of the sample instead. For these reasons, focusing via the FSD imaging appears very handy: when the FSD image is at its sharpest, it is likely that the lateral resolution for orientation mapping is at its best as well because the Kikuchi diffraction produces a large part of the contrast in FSD images. Also, it is interesting to note that FSD images display a better lateral resolution than TKD maps. The reason is that with a smaller range of acceptance angle than the scintillator, each diode scans a reduced fraction of the interaction volume leading to a higher lateral resolution (fig. 1).

Second, FSD images contain very useful information, often complementary to the TKD mapping. FSD images seemingly result from the projection of the entire lamella thickness, because they are sensitive, in addition to the Kikuchi diffraction, to the incoherent envelope as well as spots, which are not selective in depth, while TKD orientation maps tend to be very depth selective via the Kikuchi diffraction [5]. FSD images thus reveal any object regardless of in-depth position, like precipitates and particles (fig. 2). TKD orientation maps on the other hand typically display only a small fraction of precipitates and particles because these only rarely produce the predominant Kikuchi contribution in diffraction patterns, and even no contribution at all if they are too small or too far away from the back surface. In order to avoid missing microstructure features, TKD maps thus need to be associated to FSD images. Low angle boundaries revealed by orientation maps can also be correlated to dislocation walls visible in FSD images (fig. 3). In conclusion, it is advised to systematically take a high resolution FSD image of the same area as the orientation map, which often takes only a few seconds, for later interpretation.

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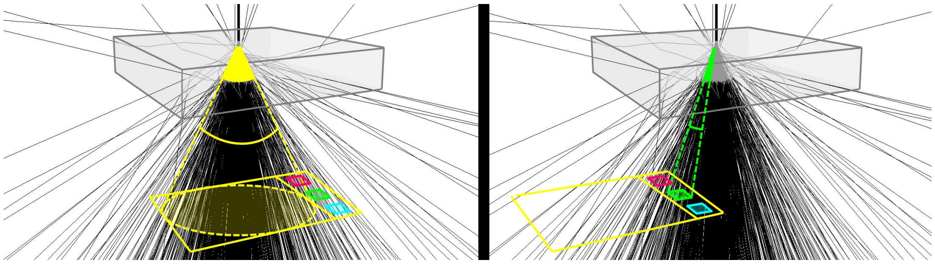

Figure 1. The three diodes (red, green, blue) have a narrow acceptance angle in comparison to the scintillator (yellow). Also, a green-magenta Z-contrast is produced (see fig. 2) because the green diode is at a shorter angular distances from the incident direction than the red and blue diodes (which in equal proportions produce the magenta color).

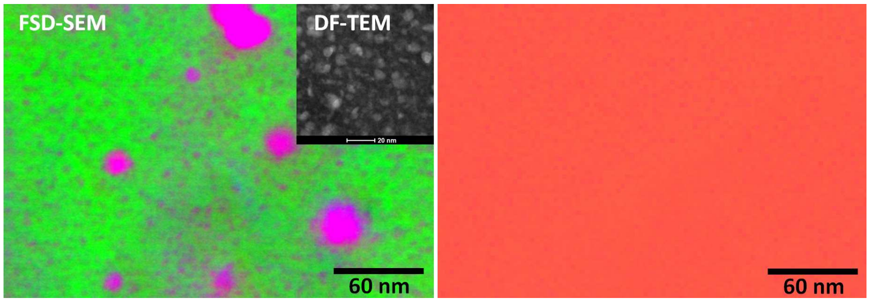

Figure 2. Z-contrast FSD image of Mg-Zn-Cu nano-precipitates and larger TiB_2 particles in an Al matrix and IPFZ orientation map of the same area showing only the Al matrix.

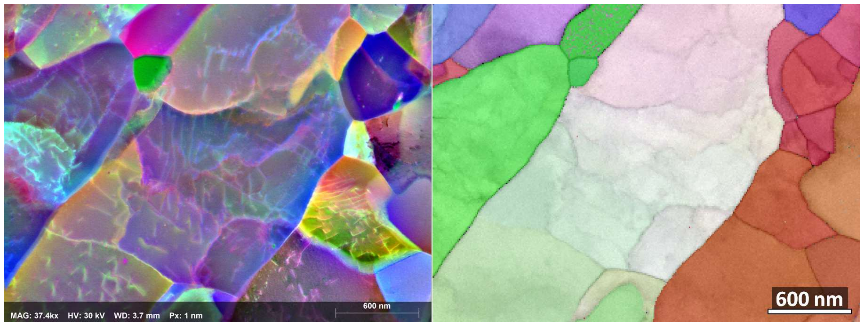

Figure 3. FSD image and IPFX orientation map superimposed on the quality pattern map on ferrite. The diodes are very sensitive to the variations of orientation and defects and with high lateral resolution.