Quantitative Diffraction Methods in the Scanning Electron Microscope

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The availability of aberration correction, the large interest in 2D materials and other nanostructures, as well as improved sample preparation yielding very thin samples have driven the TEM development to go to beam energies that have traditionally been in the realm of the much simpler (and cheaper) scanning electron microscopes. But not all experiments on these very thin samples require a high-end low-voltage TEM. In this work we present some quantitative diffraction experiments, carried out in a standard Zeiss GeminiSEM 500 equipped with a camera for recording transmission electron diffraction patterns.

We have, for example, implemented quantitative diffraction of Graphene. Large angle rocking beam electron diffraction (LARBED) is used for the 3D reconstruction of the crystal structure of complex crystals [1]. A tilt series was performed (Fig.1). Since Graphene is a 2D material, being thin in real space manifests as an extension in reciprocal space. In Fig.1 we can see the extended relrods in the kz-direction. Results on other materials systems will be shown using this technique.

Orientation mapping of Graphene was also performed. A 50x50pix image, each pixel corresponding to a diffraction map was acquired at 30kV, 20ms exposure, at 183pA, with a probe diameter ~8nm. The resulting 4DSEM data stack was fed into the ACOM software supplied with the ASTAR system [2] for mapping the crystallographic orientation, with Graphite as the reference crystal file. The result (Fig.2) is the identification of two crystallographic orientations through the Quantifoil grid. The orientation in the z-axis shows only (001), coherent with a single sheet of graphene perpendicular to the beam. Secondly, a larger area was scanned using the same methods (Fig.3). A reliability map was multiplied by the y-axis orientation map to assess the reliability of retrieved orientations. The red orientation is meaningless because the reliability is close to zero (broken graphene). We have shown orientation mapping of 2D materials on a large scale at 30kV can be facilitated in the SEM.

References

[1] C. T. Koch, Ultramicroscopy 111, 828 (2011).

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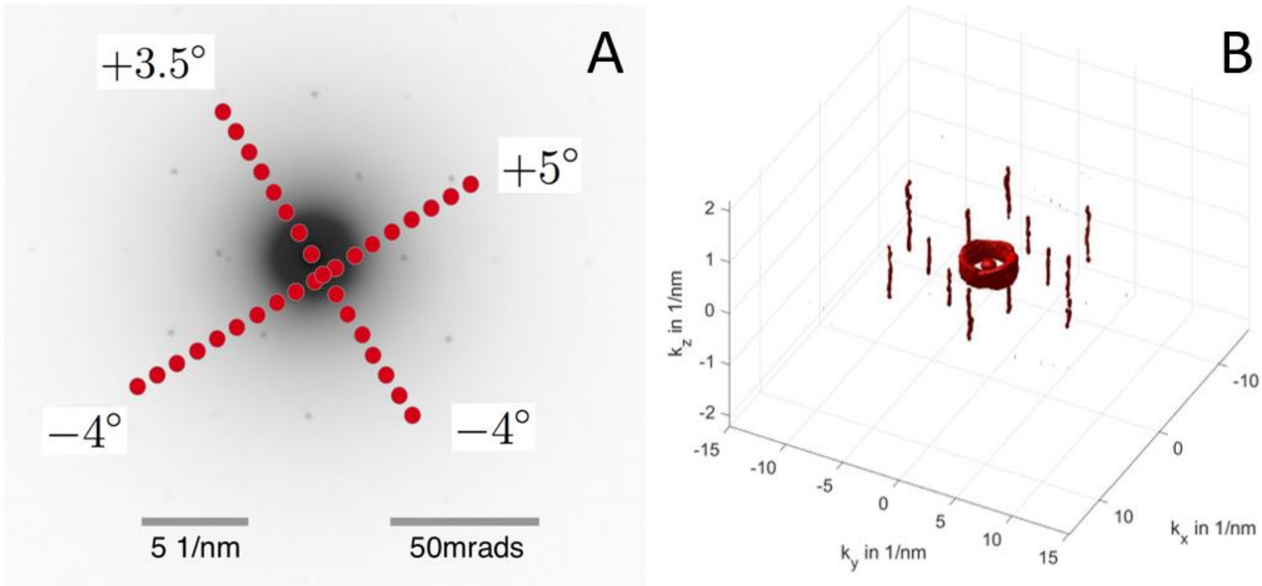

Fig.1: (A) Tilt map of the reconstruction overlaid on a graphene diffraction pattern obtained in the SEM at 30kV. The stage and sample were tilted in 0.5° steps, each red dot corresponding to a diffraction pattern at a separate tilt angle. (B) Reconstructed 3D diffraction space. Graphene is a 2D material, causing extended relrods in the kz direction.

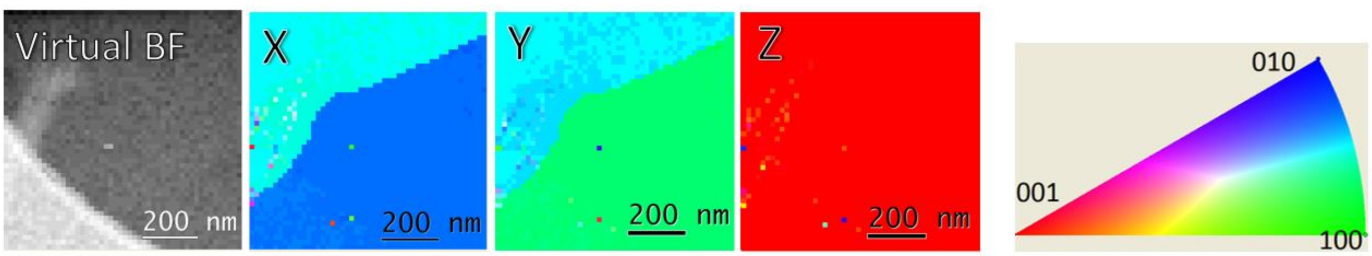

Fig.2: ASTAR crystallographic orientation mapping of Graphene on Quantifoil (50x50pix). VBF shows the Quantifoil grid (bottom left). In the X and Y crystal orientation directions, two identifiable orientations are seen. Z being the (001), is homogeneous. Right: Stereographic colormap showing crystallographic orientations.

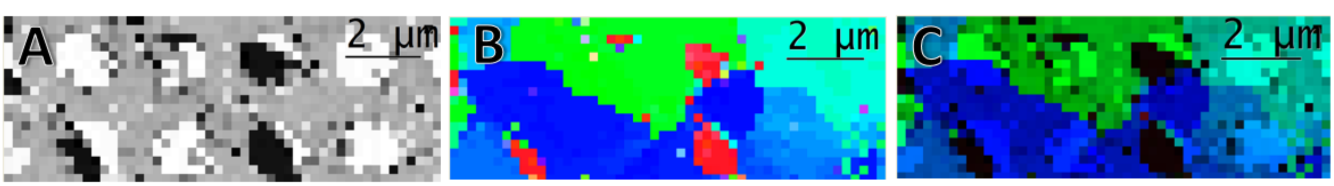

Fig.3: (A) Reliability map showing confidence in the correlation. (B) The same region identified for orientations in the Y direction. Colormap is the same as Fig.2. (C) Overlay of (A) and (B) Black regions show holes in the graphene where the confidence in orientation is low and should not be considered.