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Low Energy Nano Diffraction (LEND) – Bringing true Diffraction to SEM

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In recent years, a push towards low energies has been made in electron microscopy. On the one hand, this can help reduce damage of beam sensitive samples and on the other hand, low energies may strongly increase contrast in low-Z specimen. While this is an ongoing development on the TEM side with newer machines going lower and lower in voltage, SEM already operates at low energies by default. What SEM lacks in resolution (although already impressive at 0.3 nm) the technique makes up for in versatility. The large chamber for instance enables us not only to analyze specimen ‘as-is’, it also enables the integration of additional tools and *in situ* environments. While SEM already provides some STEM capabilities, a true diffraction mode is still missing. This additional mode would be a substantial improvement of the technique, further blurring the lines between SEM and TEM.

In this work, we present an approach to record diffraction patterns in SEM at energies of 0.5-30keV. We will show the principal setup and capabilites of the technique as well as application examples. Our setup is non-invasive, cost-effective, easy to use and it can be retrofitted to virtually any SEM. In essence the setup consists of a custom holder, a fluorescent screen and a chamber mounted camera that is directed towards the fluorescent screen (a 3D drawing of the major parts is shown in Figure 1 a). When the electron beam of the SEM is set onto a specific position of a thin sample, a diffraction pattern is visible on the screen (Far field). Due to the small convergence angle of SEM (typically <few mrad) a spot like diffraction pattern is obtained for crystalline samples very similar to what is known from TEM (see Figure 1 b: silicon oriented in the <100> zone axis). Scanning over a polycrystalline sample while recording the LEND pattern results in an average diffraction pattern of the scanned (selected) area as shown in Figure 1c for the example of platinum on graphite. The magnification is purely geometrical without the use of further lenses. The geometric distortion of the patterns given by the camera position is corrected for by a calibrated perspective transform algorithm.

An example for the application of LEND is shown in Figure 1 d. A suspended graphene membrane is analyzed in the SEM. By recording LEND patterns at dedicated spots on the membrane the relative rotation and layer number can be determined and mapped (can be expanded to full 4D STEM). Besides the analysis of intrinsic materials properties, diffraction can also be used to understand and optimize the contrast of STEM in SEM. This has already been exploited for the direct manipulation of individual dislocations in bilayer graphene[1] but can also be beneficial for the optimization of contrast obtained from organic blends as shown in Figure 1e.

The ability to record diffraction patterns concurrently with standard SEM signals (SE/BSE) is extremely useful for the investigation of dynamic processes. For instance, during the process of solid state dewetting[2], several mechanisms are intertwined such as texture formation and void formation, which can only be understood by having access to both real and reciprocal space. In Figure 2 such a heating experiment is shown in combination with the LEND setup. A thin gold layer (15 nm) is deposited onto a heating chip (b) which is placed in a custom holder (a). In the as-deposited state, the layer is fine grained and shows a random texture (c&d, acquired at the same time). During heating the dewetting as well as the crystallographic re-orientation can be tracked (e&f) and plotted. In the early stages of dewetting a texture formation is observed (vanishing of the 111-peak in Figure 2 g) which slightly precedes the visible change in area coverage obtained from the images (comparison in Figure 2 h).

The presented method enables recording of diffraction patterns in SEM, which proves extremely valuable for both soft and hard matter. The direct link between real and reciprocal space information in combination with in situ techniques enables novel insights into dynamic materials processes.

References:
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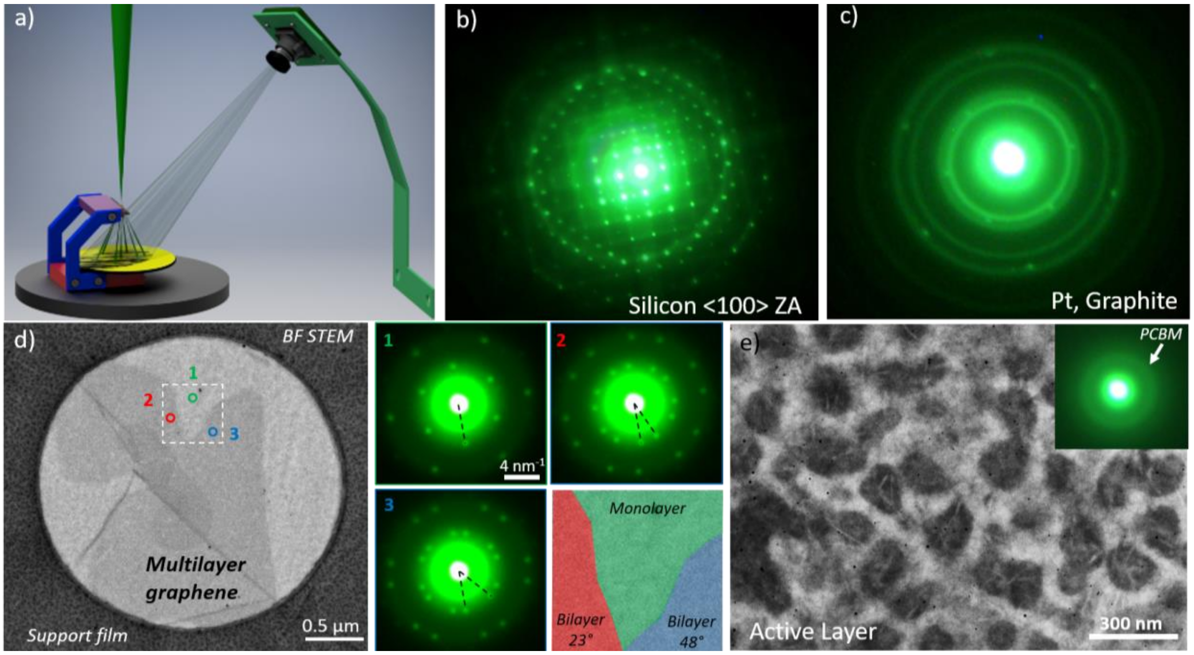

Figure 1. a) Setup of LEND. b), c) examples of LEND patterns. d) Application of LEND to analyze multilayer graphene at 20 kV. e) STEM-image of an organic blend optimized by SEM diffraction.

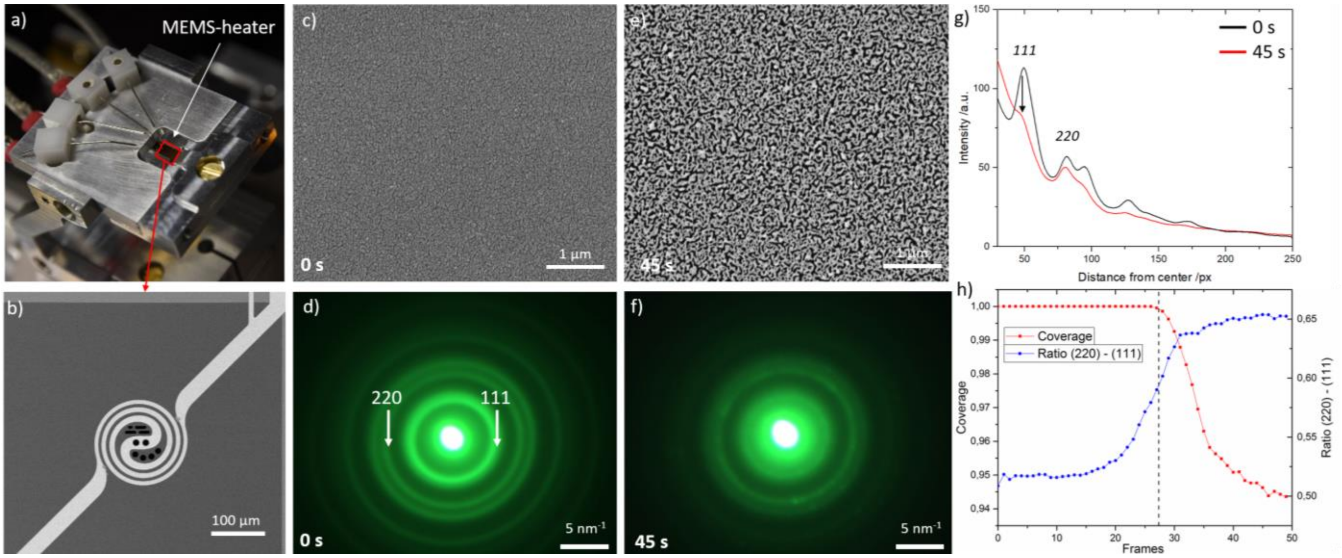

Figure 2. a) Custom-built stage for MEMS heating chips (b). c) as-prepared gold thin-film showing a random texture (d). During heating the film coarsens (e) while at the same time a texture is formed (f). Comparing diffraction (g) and image data the interplay of the different processes can be unraveled (h).