Diffraction Based Strain Mapping in Electron Microscopy

Implementation in STEM and Modified Transmission-SEM

Strain is a critical parameter for numerous novel material systems and can also yield information about local chemistry or defects. Several electron microscopy (EM) based techniques exist to measure this important quantity on the nanoscale, but the highest accuracy is achieved using diffraction based methods. Here, we show how precession diffraction in the scanning transmission EM (STEM) can be used to map strain and how to modify a commercial scanning EM (SEM) to perform diffraction based strain mapping.

Introduction

Strain is a technologically increasingly relevant quantity to be determined on the nanoscale for a range of materials. In electronics, strain is introduced to enhance the performance of devices [1] and in some topological insulator systems (e.g. HgTe) strain is used to open up a bulk bandgap and thus enable the exotic properties of these materials [2]. These are just two examples of novel material systems where strain plays a fundamental role. Furthermore, the local lattice spacings can also be used to determine nanoscopic stochiometry [3] or infer defect densities.

Transmission electron microscopy (TEM) has proven to be a powerful platform for nanoscopic strain measurements. A range of techniques has been developed to measure strain, including imaging [4] and holography [5] based techniques, with different advantages and disadvantages. Diffraction based methods, where changes in lattice distances of a material lead to varying Bragg angles, are very powerful to do so. Scanning diffraction in the scanning TEM (STEM) is also often called 4D STEM or momentum-resolved STEM because it produces a diffraction pattern (two momentum dimensions) for each scan position (two spatial dimensions). These data sets are typically huge (many GB of data) but also contain a wealth of information, which is why 4D STEM is currently intensively researched [6]. In the following we show how precession 4D STEM can be used to precisely map the strain state of a
topological insulator device and how 4D STEM can be implemented in a commercial scanning electron microscope (SEM) with a few modifications. Subsequently, we demonstrate how this comparatively inexpensive and accessible instrument with added 4D STEM capabilities can be used to measure strain in a semiconductor structure.

**Strain Mapping Using Scanning Diffraction**

Information about the strain of a sample region is contained in the Bragg angles of a diffraction pattern of this area.

By rastering a small electron probe over the sample and acquiring a diffraction pattern at each position, the strain can be mapped with a spatial resolution similar to the probe size. Originally, a small (a few nm in diameter) collimated, i.e. nearly parallel electron beam was used to obtain diffraction patterns with sharp Bragg spots [7]. However, it has been shown that not only the spatial resolution can be improved by using larger convergence angles (a few milliradians) but also the precision in the strain measurement [8]. This seems at first counter-intuitive as the sharp Bragg spots in the diffraction pattern turn into discs. However, calculating the auto-correlation function of the patterns leads to sharp peaks at the disc centers due to the periodicity of reciprocal space, and thus precise strain measurements.

**Enhancement by Beam Precession**

The high precision of scanning diffraction based strain mapping can be further enhanced by precessing the electron beam in a hollow cone above the sample and de-rocking it below it [9]. The resulting diffraction patterns are an average of the different incident beam directions, which enhances the amount of Bragg spots/discs that can be recorded and averages over many different dynamical scattering conditions. The second part is especially helpful for strain mapping with larger
convergence angles, as the multiple scattering of electrons leads to contrasts within the Bragg discs, which reduces the quality of the auto-correlation and thus the measurement precision.

Thus, adding precession to scanning diffraction experiments can lead to highest quality strain maps with below 2 nm spatial resolution and 0.02% precision [10]. This precision is close to X-ray crystallography, but with nm spatial resolution.

**Strain Mapping of a Topological Insulator**

We demonstrate the power of this technique by mapping the low but vital strain distribution of a HgTe/CdTe topological insulator (TI) system exhibiting an 8 nm HgTe quantum well. A high-resolution high-angle annular dark field image of the structure can be seen in figure 1(a) and depicts the quite sharp interfaces and absence of crystal defects. The HgTe only works as a TI when it is strained so that the bulk material exhibits a band gap and is thus insulating in contrast to its surfaces (here: edges). The necessary strain is introduced by growing the HgTe on CdTe substrate (sandwiched between HgCdTe buffer layers), but the exact strain distribution (sharpness of gradients) is important for such devices and had not been measured experimentally before.
The scanning diffraction experiment was performed using an FEI Titan Themis STEM operated at 200 kV with a convergence angle of 2.1 mrad and a beam current of 130 pA. Diffraction patterns were collected with 100 ms exposure on an FEI Ceta camera. A typical diffraction pattern can be seen in figure 1(b) with the white dashed circle depicting the precession angle. The resulting map of local lattice distances (in growth direction) with spatial resolution of 1.9 nm (actual probe size inside the sample) is shown in figure 1(c), where each pixel corresponds to a diffraction pattern as shown in (b). The determined precision, measured by the root-mean-square value deep in the substrate, is 0.03%. Below the map in figure 1(c) an experimental profile across the structure is shown in addition to profiles for a hypothetical perfect bulk structure and a finite-element simulation (using COMSOL), taking into account the sample thinning and also the effective probe size. It can be seen that the strain at the interfaces of the HgTe is not perfectly sharp, however, the values are small enough compared to the surface state wave function to guarantee the functionality of this device. Interestingly, the small gradient in the strain of the HgCdTe layers (less than 0.1%), which is an effect of thinning the sample for TEM investigation as shown by the simulation, is reproduced by the experiment. The bumps in the experimental curve seem to stem from ion beam preparation, where the Ga ions scattered at the interfaces of materials exhibiting different hardness. More detailed information about sample, experiment and results can be found in [11].

**Scanning Diffraction in a Modified SEM**

Commercial scanning electron microscopes (SEMs) are able to produce a fine electron probe and raster it in a controlled fashion across a sample, like STEMs. However, in most setups they collect mainly back-scattered or secondary electrons and typically lack an electron sensitive camera for transmitted electrons. But if the probe is positioned on an electron transparent sample and a camera is placed at a few mm distance (in the far field), diffraction can be recorded without the need for other electron optical elements (which would be lacking in an SEM). Therefore, we designed a transmission setup for our Zeiss Gemini 500 SEM based on a 6-axis piezo stage, a fiber-optically coupled CMOS camera and a stage for the camera to change the magnification of the recorded diffraction (called the camera length in TEM). We call this transmission diffraction enabled SEM the t-SEM.

A homemade scan generator and software to control all parts allows to perform automated experiments involving sample and probe motion in a highly synchronized way with up to 2000 images recorded per second. The low beam energy of this 4D-STEM setup (5-30 kV) leads to strong diffraction of thin samples (e.g. from atomically thin graphene layers) and potentially less beam damage per information
The cost of the modifications is a fraction of the initial price of the SEM and thus very small compared to the price of a STEM. In the next part we will show how this setup can be, among many other experiments, used to map strain in a semiconductor sample.

**Strain Mapping in the t-SEM**

The largest drawback of the low accelerating voltage is the comparatively low penetration depth. Thus, to demonstrate that standard samples prepared by conventional techniques can still be characterized, we investigate a GaN/AlN/Al$_2$O$_3$ structure prepared by mechanical thinning and Ar ion polishing. A secondary electron image of the sample is depicted in figure 2(a) with a marker of the mapped area and typical diffraction patterns of GaN and sapphire. To enhance the signal in the diffraction patterns, we used 30 kV and a quite parallel electron probe with a size of around 5 nm, which is sufficient here. In (b) a virtual dark field image (integrating the intensity in an annular region outside the central beam in each diffraction pattern and displaying the result as grey values) of the mapped region is given. Below, maps of the lattice distance variation relative to the Al$_2$O$_3$ substrate in growth direction and perpendicular are shown with a precision of 0.07% Distinct values can be observed for the AlN and the GaN regions and comparison with the values for bulk AlN and GaN reveal that the materials are almost fully relaxed with only very little residual strain. This indicates that high dislocation densities must be present close to the interfaces to allow this relaxation.

**Conclusion**

Momentum-resolved electron microscopy is a powerful acquisition technique and can be used to map e.g. strain very precisely in a plethora of novel and technologically relevant systems. Using precession enhances the precision further to values that compete with X-ray crystallography but offer better than 2 nm spatial resolution. With a few modifications a SEM can be turned into a scanning transmission diffraction instrument with unique benefits. Strain can be mapped in such a machine and many more 4D-STEM experiments can be envisaged.

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