

The Measurement of Strain, Chemistry and Electric Fields by STEM based Techniques

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In order to understand the properties of nano-scaled devices it is important to be able to measure the structure, composition and fields with nm-scale resolution. The introduction of latest generation aberration corrected transmission electron microscopes (TEM) make imaging of these materials more straightforward. However, there is still the need for improved characterization of large areas - hundreds of nanometers – while still maintaining nanometer scale resolution. In this presentation we will discuss three different sub-techniques of Scanning TEM (STEM): Nano beam Precession Electron Diffraction (N-PED), Energy Dispersed X-ray Spectroscopy (EDX) and Differential Phase Contrast (DPC). At CEA we routinely perform these techniques for device studies, however for dose and time optimization and improved correlation, performing all these techniques simultaneously is very important and we will discuss how this can be done.

Recently, many different TEM-based approaches have been developed to measure strain, mainly in semiconductor devices [1,2]. At CEA, we have developed the N-PED technique, which has been optimized in collaboration with the Thermo Fisher Scientific (TFS) - FEI Company -. TFS has created a new software, called Epsilon, to acquire and analyze a series of N-PED patterns. Compared to our previous homemade digital micrograph script, Epsilon is 5 times faster for acquisition and the precession speed can be as fast as 1ms. Maps of 60x60 diffraction patterns can be acquired within 10 minutes. The advantages of N-PED are its facility of implementation, its robustness, its precision (about $2 \cdot 10^{-4}$) and its applications to a wide range of samples, on or off-axis. By using a small condenser aperture of 10 μ m, a nano beam of about 1-2nm is formed on the sample. The beam is then spun - i.e. precessed - by an angle of about 0.3° around the optical axis with a precession speed of up to 1 ms. Every 50ms, the beam is shifted by generally a few nanometers and at each position a N-PED pattern is acquired on a CMOS camera. Fig. 1 shows a typical N-PED pattern, which is composed of uniform diffraction disks and strain/rotation maps realized on a transistor. By comparing the maps obtained in the silicon crystal to finite element simulations the stress in the adjacent Si₃N₄ layer could be determined. The technique has also been applied to GaN/AlN layers deposited on Si wafers and to core-shell Ge/Si₃N₄ nanowires.

We have also developed a new method to obtain accurate quantitative chemical data from EDX spectra. For a given element, X-ray cross-sections are carefully determined by examining a bulk reference sample in a SEM. A new algorithm, which is an extension of the zeta-factor method and whose name is IZAC, have been developed to take into account beam interaction and absorption of X-rays [3]. The IZAC code determines accurately both the thickness and the chemical composition of the sample. We realised that in many situations, one EDX 2D-map acquired in one given direction of observation is sufficient to build up a 3D-chemical map of the sample. This is possible by building a simple, but realistic, model of the observed sample and by using the IZAC code. For instance, Fig. 2a gives the chemical composition of ZnTe nanowire (NW) containing a CdTe dot [4].

More recently, we applied the DPC technique to measure electrical field in a range of specimens such as in-situ biased silicon pn-junctions, CMOS devices and III-V based specimens [5]. In this presentation we will discuss the advantages and disadvantages of using DPC on nanoscaled materials when compared to more established techniques such as off-axis electron holography.

References:

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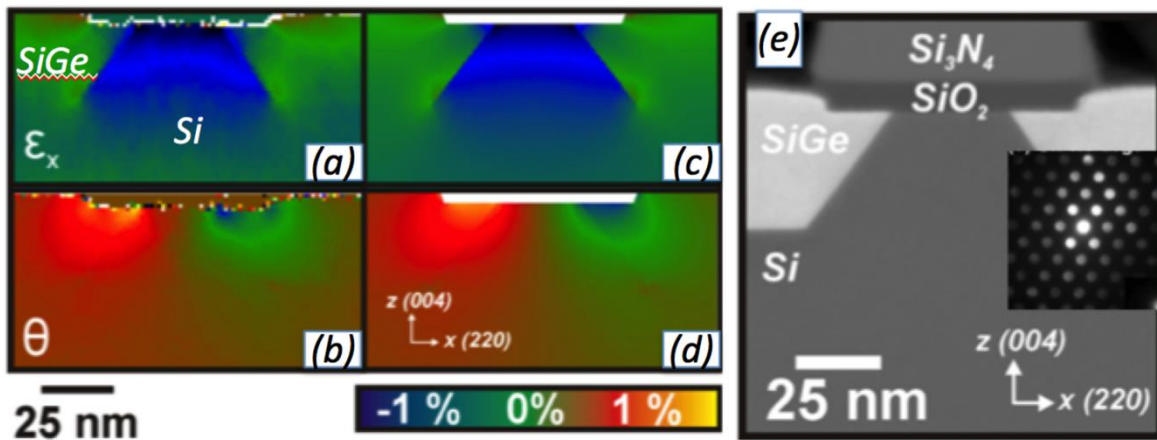


Figure 1. (a, b) Experimental and (c, d) finite element simulated ϵ_{xx} strain (a, c) and rotation (b, d) maps obtained in a transistor with SiGe source and drain. (e) STEM image of the transistor with in inset a N-PED pattern. Best fit with simulations is obtained with a 1.9 GPa stress in the Si_3N_4 layer.

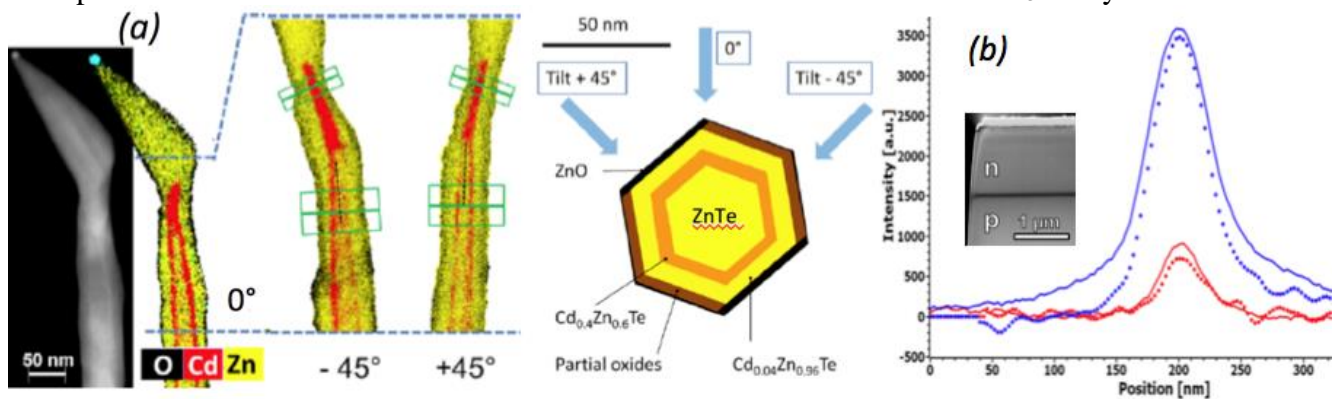


Figure 2. a) O-Cd-Zn EDX maps of a ZnTe nanowire (NW) containing a pure CdTe dot obtained with different crystal tilts. On left, a STEM image, on right a scheme of a cut of the NW below the dot: due to a lateral growth, an unwanted $\text{Cd}_{0.4}\text{Zn}_{0.6}\text{Te}$ shell is present. The 3D model can also be obtained from only one crystal tilt. b) DPC (line) and holographic phase gradient (dots) profiles taken perpendicular to a Si pn-junction, with (higher curves) or without (lower curves) a 4 volt reverse bias across the junction. The signal, which is proportional to the projected electrical field, measures the extension of the depleted regions. In inset the 4 volt DPC map from which one profile is taken (arbitrary contrast unit).