Simultaneous acquisitions and applications of DPC/OBF STEM, EDS and EELS

Dr. Yuhiro Segawa[1], Dr. Akiho Nakamura[1], Mr. Hiroki Hashiguchi[1], Mr. Yuji Kohno[1], Dr. Shigemasa Ohta[1], Senior Assistant Professor Takehito Seki[2], Professor Naoya Shibata[2]
[1]JEOL Ltd., Akishima-shi, Japan, [2]The University of Tokyo, Bunkyo-ku, Japan

Background incl. aims

The use of a segmented detector has become standard for various STEM observations, particularly for Differential Phase Contrast (DPC) STEM[1] and Optimum Bright Field (OBF) STEM[2]. DPC STEM can visualize weak electromagnetic fields such as p-n junction interfaces[3] and magnetic skyrmions[4]. In low-dose experiments with beam-sensitive materials, like zeolites and metal-organic frameworks (MOFs)[5], OBF STEM method achieves noticeably better contrast during live imaging. The applications of a segmented detector are further extending across a variety of material and life science fields. As an example, this research shows a combined analysis of these advanced imaging techniques with elemental analysis methods, EDS and EELS, simultaneously acquired in our new FEMTUS platform.

Methods

The sample was a semiconductor memory. The experiment was performed using JEM-F200, equipped with SAAF-Quad detector (an annular four-segmented detector), Dual SDD detector for EDS, CEOS Energy Filtering and Imaging Device (CEFID) with Dectris ELA hybrid-pixel electron detector, and integrated analysis platform FEMTUS developed by JEOL. In the FEMTUS platform, all detectors and cameras can be synchronized and simultaneous acquisition becomes possible with easy operation. For all experiments we chose an accelerating voltage of 200 kV, STEM mapping was performed with a dwell time of 10ms, convergence semi-angle of 6.6 mrad, and EELS collection semi-angle of 2.2 mrad limited by the central hole of SAAF-Quad detector.

Results

Figure 1 shows the result of DPC STEM and EDS/EELS elemental mapping, acquired simultaneously in a single scan. Fig. 1a shows the x-components of the center of mass (COM) DPC STEM derived from the signals of four SAAF-Quad detector channels. It can be seen that the COMx image reveals thin line contrasts around the regions indicated by the arrows. The DPC STEM method has better sensitivity for differences in projected potential, originating from both electromagnetic field and/or local chemical composition. Fig. 1b and 1c represent a magnified view of the EDS and EELS count maps, respectively. EDS mapping has an advantage in detecting heavy elements such as tungsten and titanium, which are difficult to access using the phase imaging method (DPC or OBF STEM) and EELS. As complementary information, the EELS mapping shows clear contrast for light elements (oxygen, nitrogen, and silicon) with higher S/N ratio compared to EDS. All of this information can be used to analyze the origin of DPC STEM contrast. Fig. 1d shows intensity profiles of COMx and EELS data extracted from the area indicated by the white square. The peaks of COMx intensity correspond to the increase of oxygen component, whereas the amount of nitrogen decreases in the interface region. Such combined information is very helpful to investigate the origin of phase contrast images, such as the composition difference between SiOx film and SiNx bulk region shown here.

Conclusion

In summary, we acquired DPC STEM, EDS, and EELS data of semiconductor samples simultaneously and revealed that the origin of DPC STEM signals was due to changes in the local chemical composition. Without the additional information from EDS and EELS, it was difficult to clarify whether the obtained phase contrast represents chemical composition, electromagnetic field, or just a difference in local thickness. Such simultaneous acquisition of DPC, EDS, and EELS enables us to directly understand the origin of the observed phase image contrast. Furthermore, since compared to EDS and EELS mappings, DPC STEM is very sensitive to changes in the projected potential, it will be possible to clarify compositional differences by integrating the EDS and EELS signals of regions where phase contrast differences could be observed, even under low-dose conditions. This should also be useful for the composition analysis of electron beam-sensitive materials whose structures are destroyed with just a few scans. On the day of the presentation, we will show the details of the experimental results and additional instances of simultaneous data acquisition including OBF STEM.

Graphic:

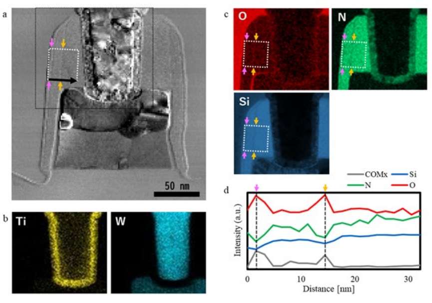

Figure 1. Semiconductor material observed by DPC, EDS and EELS
a: COMx image acquired using SAAF-Quad detector. b: EDS count maps of titanium (Ti) and tungsten (W) extracted from the area marked by the black square in a). c: EELS count maps of oxygen (O), nitrogen (N), and silicon (Si) extracted from the area marked by the black square in a) d: intensity profiles of COMx and EELS. These profiles were acquired in the direction of the black arrows, integration over the white dotted area shown in the COMx and EELS datasets.

Keywords:

Segmented-Detector, DPC-STEM, OBF-STEM, EDS, EELS

Reference:

[1] Naoya Shibata, Scott D. Findlay, Yuji Kohno, Hidetaka Sawada, Yukihito Kondo, Yuichi Ikuhara, Nature Physics, 8, 611 (2012).
[2] Kousuke Ooe, Takehito Seki, Yuichi Ikuhara, Naoya Shibata, Ultramicroscopy, 220, 113133 (2021).
[3] Yuji Kohno, Akiho Nakamura, Shigeyuki Morishita, Naoya Shibata, Microscopy, 71, 111 (2016).
[4] Takao Matsumoto, Yeong-Gi So, Yuji Kohno, Hidetaka Sawada, Ryo Ishikawa, Yuichi Ikuhara, Naoya Shibata, Scientific Reports, 6, 35880 (2016).
[5] Hiroki Hashiguchi, Akiho Nakamura, Vacuum and Surface Science, 66, 695 (2023).