APPLICATION NOTE

Integrated Differential Phase Contrast on Talos S/TEM

The ultimate low-dose STEM technique for the imaging of all elements.

Light elements are key components in the design and engineering of many advanced materials and energy management technologies—oxygen in dielectrics and superconductors, lithium ion battery materials, hydrogen in hydrogen storage materials—making their reliable imaging an important objective for atomic-resolution structure analysis. Forming an atomic-resolution image in the electron microscope that provides dose-efficient, high-contrast imaging of light elements, while also providing strong compositional contrast, has been a long-standing challenge.

Up until now, one of the most commonly applied imaging techniques for low-Z elements has been annular bright field scanning transmission electron microscopy (ABF-STEM); however, this proven technique has several disadvantages that should not be neglected. ABF-STEM is highly sensitive to defocus and thickness variations, causing the atoms to display dark contrast with bright background and vice versa. This leads to uncertainty in determining the exact positions of atoms on a grayscale image.

In this application note, we discuss the advantages of a new image formation method—integrated differential phase contrast STEM (iDPC STEM)—which has been developed by Thermo Fisher Scientific after extensive analysis on STEM imaging techniques (see references 1&2, page 4). We also demonstrate the application of this method in a variety of use cases.

The iDPC STEM method exposes low-Z elements with bright contrast and dark background, placing considerably less dependence on defocus and/or thickness. In addition, it has been shown that iDPC-STEM images have a higher signal to noise ratio compared to ABF-STEM images (iDPC is also available on the Thermo Scientific™ Themis Z, 200 & 300 TEM platform; see references 3-6, page 4). A higher signal to noise ratio provides for the possibility of low-dose imaging, which is crucial for beam-sensitive materials and charging samples.

Method

In order to use the iDPC STEM method for imaging of all elements, the user first selects the segmented DF4 detector of the on-axis BF/DF detector from the Thermo Scientific Velox DPC/iDPC control panel.

To switch to iDPC, the user selects dDPC or iDPC signals from the composite drop-down menu (Figure 1). A filter for the iDPC image can be applied using the checkbox. The strength of the filter can be adjusted in the Filter Settings panel to achieve the best image quality.

Results

Here, we demonstrate the results of the iDPC STEM method in a variety of uses cases. All images have been acquired using the non- C_s corrected Thermo Scientific TalosTM F200 S/TEM (scanning/transmission electron microscope).



Figure 1. Velox DPC Control Panel



Figure 2.1. iDPC Image of Zeolite, Figure 2.2. FT and iDPC Image of Zeolite with Digital Zoom.



Figure 2.3 and 2.4. Comparison of ADF and iDPC image of Zeolite at 200kV acquired on the same area.



3.1

Figure 3.1 and 3.2. Comparison of Annular Bright Field (ABF) and iDPC image of Polypropylene + Graphene-like Material (MG-P-N) using the same beam current/dose on the same area. *Sample* courtesy Freiburger Material Forschungszentrum, Dr. Ralf Thomann.

Figure 2.1 displays the iDPC image and embedded FFT of the same zeolite sample, taken with a beam current of only 2 pA, while still showing a resolution down to 0.16 nanometers.

Figure 2.2 features the iDPC image of the zeolite sample showing a digital zoom of the region in red on the left.

The sample in Figure 4 illustrates the contrast between graphene oxide layers, imaged with iDPC using a probe current of 1 pA. Figure 5 shows the high resolution capabilities with iDPC; HRSTEM on SrTiO₃. The Strontium, Titanium and even Oxygen columns are clearly visible.



Figure 4.1 and 4.2. Comparison of ABF and iDPC image on Graphene Oxide Layers (sharper contrast) using the same beam current/dose. *Sample courtesy of Dr. Ralf Thomann, Freiburger Material Forschungszentrum.*



Figure 5. High Resolution iDPC STEM on SrTiO₃ on Talos F200X (non - Cs corrected).



Figure 6. NAND Flash ONO Layers. 1. BF, 0 – 14 mrad. STEM at 200kV. Current 5pA. 2. DF4, 34 – 89 mrad. STEM at 200kV. Current 5pA. 3. iDPC. iDPC at 200kV. Current 5pA.

The final example centers on the ONO layers on a NAND Flash memory sample imaged in both STEM and iDPC modes (Figure 6). Clearly, in the iDPC image, the contrast of the light element layer is superior.

Conclusion

The use of light elements in the design and engineering of novel materials and energy management technologies is becoming increasingly important. The ability to determine the function and structure of these elements requires a technique that enables reliable imaging of these elements at atomic resolution. Many conventional imaging methods, such as ABF, have become proven technologies over the years but also present some disadvantages that cannot be disregarded. For this reason, Thermo Fisher Scientific introduces the newly developed iDPC STEM method to more reliably and accurately image light and heavy elements simultaneously, at atomic resolution. It exposes low-Z elements with bright contrast and dark background, placing considerably less dependence on defocus and/ or thickness. Since iDPC uses a normal STEM scanning procedure, the result appears directly and live and does not require an elaborate reconstruction scheme. In addition, iDPC-STEM images have a higher signal to noise ratio compared to ABF-STEM images. A higher signal to noise ratio provides for the possibility of low-dose imaging, which is crucial for beamsensitive materials and charging samples.

References

- 1. I. Lazić, E.G.T. Bosch, S. Lazar, Ultramicroscopy 160 (2016) 265–280
- 2. E.G.T. Bosch, I. Lazić, Ultramicroscopy 156, (2015) 59–72
- 3. I. Lazić, E.G.T. Bosch, Advances in Imaging and Electron Physics 199, (2017) 75-184
- I. Lazić et. al., Microsc. Microanal. 22 (Suppl 3), (2016) 36-37
- E.G.T. Bosch, I. Lazić, S. Lazar, Microsc. Microanal. 22 (Suppl 3), (2016) 306-307
- E. Yücelen, I. Lazić, E. Bosch, Microsc. Microanal. 22 (Suppl 3), (2016) 1458-1459



Find out more at thermofisher.com/EM-Sales

For current certifications, visit FEI.com/certifications. © 2017 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific and its subsidiaries unless otherwise specified. AN0071-EN-10-2017