Octane Silicon Drift Detector (SDD) Series for the Transmission Electron Microscope (TEM)

Introduction
The Octane SDD Series for TEM evolved from the Apollo XLTW series of detectors to provide a new family with the best solid angle and highest sensitivity available. This series features a proven windowless design, as well as an optimized detector module enclosure. Both of these features allow the maximum solid angle to be achieved in a given microscope configuration. By eliminating the detector window, the support structure of the window is removed. By removing the window support structure the solid angle is increased by almost a factor of two [1]. The customization of the module enclosure allows the detector to be placed closer to the sample, increasing the geometrical solid angle. The vacuum environment in a TEM is very favorable to using a windowless detector. There is little chance of condensing any water vapor on the cold detector (-25°C) and the detector can be quickly temperature cycled if needed.

Octane SDD Series for TEM
There are three different models of detector available in the Octane SDD Series for TEM:

**Octane T Plus**: 30 mm² active area. Entry level SDD with super ultra-thin window (SUTW) mountable on all TEMs.

**Octane T Optima**: 30 or 60 mm² active area. Windowless design optimized for specific TEM columns with solid angles up to 0.5 steradian.

**Octane T Ultra**: 100 mm² active area. Highest sensitivity SDD available, windowless with ultimate solid angle up to 1.1 steradian.

The Octane T Plus and T Optima are round, while the T Ultra detector is an oval (racetrack) shape. The new design features of the Octane SDD Series for the TEM offer significant benefits.

1. Enhancement in Sensitivity
A NiO spectrum from the Octane T Ultra is shown in Figure 1. Both the NiL to NiK ratio and the NiL to O K ratios are very high. This is due to the lack of a window between the detector and sample. The sensitivity to N is also greatly enhanced. A polymer window has a large concentration of C, which preferentially absorbs N. So, eliminating the window allows more N X-rays to enter the detector.

2. Increase in Peak Intensity
The spectrum in Figure 2 is from a SiN 50 nm thick membrane. The windowless Octane spectrum is in green, while the spectrum from a SiLi detector is in red. Both spectra were taken at the same live time and beam current. The windowless design also improves the sensitivity for higher energy lines such as SiK. The peak intensity of N is 12 times better, and the Si intensity is four times better.

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Figure 1. NiO spectrum from the Octane T Ultra.

Figure 2. Si$_3$N$_4$ collected on FEI CM200 with ST lens.

Figure 3. A spectrum from a mineral glass thin film characterized by NIST 2063a.
The sample in Figure 3 is a mineral glass thin film that is well characterized by NIST. With it, quantitative analysis can be checked or the sample can be used to calculate Cliff-Lorimer K-factors. The K-factors calculated from this spectrum are seen in Figure 4.

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TEAM™ EDS Atomic Resolution Drift Correction

The TEAM™ EDS Atomic Resolution Drift Correction was created especially for the most demanding samples in TEM data collection. The challenge is to maintain beam-sample placement to collect the proper data from samples, which are sensitive to extremely fine movement. Such a drift process requires a higher quality reference image and a faster correlation process. This routine provides both of these capabilities by using the actual image as the reference image during collection, which eliminates all the time to collect a separate reference image. Furthermore, the correlation process uses a fast Fourier transform immediately in-between collection frames and makes the necessary adjustments before starting the next frame. The result is maximized image and element signal intensity in the proper locations, which creates high resolution maps.

A sample of SrTiO₃ was analyzed on a Hitachi HD2700a STEM with aberration correction. The conditions were: 200 Kv, 100pa of beam current and 8000Kx magnification. Mapping was performed at a pixel level of 256x200 pixels. The dwell time at each pixel was 150μsec, and the number of frames acquired was 109. TEAM™ EDS Atomic Resolution Drift was applied to correct for drift in the system.

Applications

Application 1: A Layered Membrane

Using an Optima T Plus detector, the DLR Institute of the University of Cologne measured a cross-section of thin film layers on a membrane. These maps (Figure 7) were acquired on a Tecnai F-30 at 200 Kv. The conditions were 3.84 nAmp Screen Current, Condensor 2: 70 μm, 7.68usec Amp time, 8000cps, 68000x microscope magnification. 500 frames were acquired.
Figure 8. An overlay of the primary elements of interest. (Cu is from the grid and Ga and Pt are from the FIB processing.)

Figure 9 shows a line scan from top to bottom of the area. It is rotated 90 degrees for ease of interpretation.

Application 2: Clusters of Silicide Particles
The STEM image in Figure 10 was acquired on a Tecnai F-20 at 200 Kv. Silicide particles make up this cluster. While acquiring maps, a phase map was created showing the presence of five phases.

Figure 10. STEM image showing silicide particles that make up a cluster.
The orange phase was a Mn silicide, the blue phase was Ni silicide, the green phase was Co silicide, and the yellow phase was Si. The map (Figure 11) was acquired at 128 by 100 pixels. The microscope magnification was 20,000x.

A thinner area near an edge of the blue phase was quantified (Figure 12). It shows that the composition is close to NiSi. The thickness of the area is unknown, so accurate analysis is not possible. The count rate was in excess of 20,000 cps, and the total acquisition time was less than five minutes.

Application 3: A Line Scan Across a Nanoparticle
The image, spectrum and line can in Figure 13 were acquired on a Hitachi HD-2700 dedicated STEM. The bright particles are Pt-Co nanoparticles on graphite. The line scan was performed over a 5 nm diameter particle. The sample was courtesy of Nestor Zaluzec at Argonne National Laboratory.

References
[1] Alan Sandborg, Mike Coy, Brent Hammell, Reinhard Buchhold
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Conclusion
The Octane SDD Series for TEM provides chemical analysis at the highest level possible for single detectors on a column. The Octane detectors achieve much higher solid angles than the SiLi detectors that they replace, as well as higher light element sensitivity. They also free the user from liquid nitrogen.