With the upcoming release of APEX™ 2.0, we are very excited about the addition of Electron Backscatter Diffraction (EBSD) and combined Energy Dispersive Spectroscopy (EDS)/EBSD to the platform. While the inclusion of EBSD is an important aspect of the release, it also includes a significant number of new features and extensions. In particular, inclusion of the open-source HDF5 file format allows for easier data management and storage of very large datasets (with optional compression) into a single self-contained project file. Of course, all of the save and export raw data options are still present, allowing for both easy sharing and data transfer of full projects, as well as individual spectra, patterns, and analysis results.

There are also numerous changes to EDS quantification, including oxides quantification, peak-to-background ZAF, Standard Customized Coefficients, and automatic calculation of the minimum detection limit (MDL). A question that frequently comes up during EDS analysis is, “What is the MDL of the system?” While the question is simple, the answer is unfortunately quite complicated since it depends on multiple factors, including detector window, geometry, collection time, detector resolution, count rate, and sample composition. It is impossible to give a general MDL for a given element, but it is possible to calculate the MDL for a given spectrum. With the upcoming release, this calculation is available as a display in the quantification window.

For a peak to be significant, the raw peak counts must be above the background noise with 95% confidence. For a peak to be above the detection limit, the net counts (raw peak minus background counts) must again be above the noise with 95% confidence, taking both peak and background noise into account (Figure 1).
As part of the quantification routine, it is relatively trivial to back-calculate what weight concentration the MDL corresponds to and flag elements that are below the detection limit. Figure 2 shows an example spectrum from a BHVO-2 Basalt sample and illustrates how the MDL is element and collection time-dependent. Whenever a given element is found to be below the detection limit, it is flagged in the quantification table, as shown in Figure 3.

### Figure 1. Three Gaussian peaks overlaid on a noise signal. Peak 1 is below significance, Peak 2 is above significance but below the detection limit, while Peak 3 is above both significance and detection limit.

<table>
<thead>
<tr>
<th>Element</th>
<th>Line</th>
<th>Weight %</th>
<th>MDL</th>
</tr>
</thead>
<tbody>
<tr>
<td>O K</td>
<td>K</td>
<td>48.0</td>
<td>0.82</td>
</tr>
<tr>
<td>Na K</td>
<td>K</td>
<td>1.9</td>
<td>0.88</td>
</tr>
<tr>
<td>Mg K</td>
<td>K</td>
<td>3.9</td>
<td>0.67</td>
</tr>
<tr>
<td>Al K</td>
<td>K</td>
<td>7.2</td>
<td>0.67</td>
</tr>
<tr>
<td>Si K</td>
<td>K</td>
<td>20.8</td>
<td>0.67</td>
</tr>
<tr>
<td>P K</td>
<td>K</td>
<td>0.3</td>
<td>0.58</td>
</tr>
<tr>
<td>K K</td>
<td>K</td>
<td>0.7</td>
<td>0.89</td>
</tr>
<tr>
<td>Ca K</td>
<td>K</td>
<td>8.2</td>
<td>1.05</td>
</tr>
<tr>
<td>Ti K</td>
<td>K</td>
<td>1.4</td>
<td>1.24</td>
</tr>
<tr>
<td>Fe K</td>
<td>K</td>
<td>7.5</td>
<td>2.13</td>
</tr>
</tbody>
</table>

Figure 3. Quantification table highlighting P and K as being below the MDL.

There are many more things to look forward to, including Montage maps (please see Dr. René de Kloe’s recent blog “Between the Lines” for very nice examples of Montage maps being applied to fossils), EDS and EBSD batch scans, options for online software updates, live quantification of line scans, and max pixel spectrum. EDAX is very excited to share these new features and look forward to your feedback.
Setting Up the Velocity EBSD System for High-Speed Collection

Velocity™ is a high-speed CMOS-based EBSD system. There are two levels of performance available: the Velocity Plus that indexes up to 3,000 patterns per second, and the Velocity Super, which indexes up to 4,500 patterns per second. For this article, we discuss the setup for 3,000 indexed patterns per second, while a future Tips and Tricks will address the higher speeds available with the Velocity Super. As the first CMOS-based detector in the EDAX EBSD product line, there are a few differences in how to set up this system compared to legacy DigiView and Hikari CCD-based systems.

One fundamental difference between CCD and CMOS systems is how camera binning works and the implications it has on EBSD setup. When binning with a CCD system, it combines the accumulated charge for each pixel during the binning process, and then reads it as a single binned pixel. This results in an overall increase in pixel intensity and image transfer speed. In contrast, each pixel with the CMOS system has its own readout electronics. That means the total image can be read out faster than a CCD sensor, but it does not combine the charge prior to the readout. From a practical setup perspective, this also means we do not need as many binning options for the Velocity when compared with CCD-based systems.

For most EBSD mapping applications, we recommend 4 x 4 binning. This provides a 120 x 120-pixel image, which is ideal for the default values of the Hough Transform used for band detection. With this setting, the 16 pixels defined by the 4 x 4 binning setting are averaged to reduce temporal noise, but there is no increase in signal intensity. Also available are 8 x 8 and 16 x 16 binning options, but they do not increase the scanning speeds of the detector and are primarily used for reducing the size of the saved pattern file during dictionary indexing applications with OIM Matrix™.

Another difference between the Velocity series and previous systems is the absence of a camera gain setting. Previously, you could adjust both the exposure time and the camera gain. Camera gain amplifies the camera signal, but also the baseline noise. The resulting amplified image appears brighter, but also noisier. With the Velocity systems, you only adjust the exposure time. As exposure time increases, the image intensity and signal-to-noise level increase, while the camera frame rate decreases. To optimize for the highest speeds, you can modify the exposure time. For 3,000 indexed patterns per second, select an exposure time of 0.27 ms, which produces a frame rate of 3,731 frames per second.

The final setting for high-speed collection is the Camera Mode setting. This setting has two options: Standard and High-Speed. The maximum speed of the Standard Mode is approximately 2,500 indexed patterns per second, and is often sufficient for many applications. The High-Speed mode improves the data throughput rate, and allows for speeds up to 3,000 indexed points per second.

To summarize, the recommended settings for 3,000 patterns per second indexing are 4 x 4 binning with a 0.27 ms exposure time using the High-Speed mode. After choosing the settings, select a background, plus the Standard Image processing mode. At this stage, evaluate the EBSD image quality and indexing performance. With the Velocity systems, these speeds require at least 10 – 15 nA. If the pattern quality is insufficient for suitable indexing performance, it may require more beam current or improved sample preparation.
The elemental detection range for an X-ray Fluorescence (XRF) instrument is a common specification. It indicates the range of elements that the instrument can directly detect. Typically, XRF instruments that make measurements with the sample under vacuum can detect lower-energy X-ray lines and, hence, lighter elements with lower energy differentials between the electron orbitals. However, it is possible to make XRF measurements that may appear to be outside of this elemental range, given some basic knowledge about how the specimen was made or other supplemental spectroscopic measurements. In this application note, we provide a few examples to support this point.

Measuring Aluminum Coatings in the SMX System

The SMX product line is an EDAX XRF system that can measure coatings and bulk compositions. The system has a large sample chamber (nominally 34 cm wide and 56 cm deep), which measures samples at ambient atmosphere. Measuring thin aluminum coatings directly from the Al(K) signal at 1.486 keV would be challenging because the amount of Al signal in a layer is limited, and the Al(K) X-ray is readily scattered by the ambient atmosphere. However, it is possible to measure Al coating thickness by the absorption of the XRF signals emitted from the substrate (Figure 1). As XRF signals from the substrate traverse through a surface coating, absorption occurs. The thicker the coating, the more absorption occurs until a point where the surface coating becomes so thick that the substrate signal is fully absorbed.

![Absorption curve for substrate signal passing through a surface layer prior to contacting the detector.](image)

The range of measurable aluminum coating thickness depends on the absorption physics of the substrate signal in the surface layer of aluminum. When the substrate is nickel, the Ni(K) line at 7.472 keV is not readily absorbed in the aluminum surface coating. The result is that it can measure heavy layers of Al, but it limits the ability to measure thinner layers. The change in Ni signal intensity by absorption in very thin Al layers is weak, and therefore, it is more challenging to measure that small change in Ni intensity for very thin layers (Table 1).

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Minimum Thickness [μm]</th>
<th>Maximum Thickness [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>1 - 5</td>
<td>200 - 250</td>
</tr>
<tr>
<td>Si</td>
<td>0.04 - 0.05</td>
<td>~ 4</td>
</tr>
</tbody>
</table>

Table 1. Range of aluminum coating thickness measured on substrates of Ni and Si wafer.

Despite the ambient measuring atmosphere, it is also possible to measure an aluminum coating on silicon wafers even though the Si(K) signal at 1.74 keV is just above the Al(K) line and is also readily scattered by the ambient atmosphere. The difference, in this case, is that we are looking at a higher intensity of the signal, essentially a pure Si signal emitted from the bulk wafer. It turns out that measuring the thin Al layer thicknesses via absorption of the Si substrate signal is ideal for measuring layer thicknesses in the range of 100 nm. The Si signal is readily absorbed in the aluminum coating because the Si(K) energy is just above the absorption edge for the electronic transition responsible for the Al(K) fluorescence. This shifts the entire measuring range down with respect to that of a Ni substrate, e.g., very sensitive to thin Al layers but readily absorbed in a few microns of an Al deposition. The approximate measurement range of Al coatings on Si wafers is in Table 1. Note, that the absorption phenomenon described here applies to any type of surface coating, not just aluminum.

Measuring Trace Metals in Plastics with the Orbis System

Many plastics are comprised of C, N, O, and H, which are difficult XRF signals to quantify due to their low energy, absorption effects, low measuring precision, and small sampling volume. If the nature of the plastic is already known, for example, from an IR measurement, then it is possible to quantify the trace metals in the plastic material. Given a single calibration standard, the Orbis quantification routine can model the plastic sample matrix and quantify trace metals for applications, such as compliance with RoHS-type environmental regulations. When measuring high-energy lines like Cd(K), it is necessary for the quantitative routine to account for the sample thickness as well since higher energy X-ray lines can be observed from a greater depth within the sample. Table 2 shows an example result where an ABS resin standard (1 mm thick pellet) has been measured against a PVC calibration standard. The PVC pellet is 14 mm thick.
When making measurements of this type, care should be taken to account for sample inhomogeneity, particularly when the calibration standard is comprised of extruded pellets, where variations may occur from pellet to pellet.

**Measuring Stoichiometric Compounds with the SMX Quantification Routine**

Sometimes there are situations where the material to be qualified is comprised of various compounds. Surface treatments for aluminum brazing products are an example of this. Typical fluxes for this application include potassium fluoroaluminate complexes of known stoichiometry. While it is difficult to measure the Al or F signals directly in the SMX system, the quantification routine in the SMX system can use information about known stoichiometries to formulate compounds. It is then possible to directly measure potassium or other metals involved in the formulation compounds and then back-calculate the amount of fluorine and aluminum present in the matrix. The inclusion of fluorine and aluminum into the sample matrix by way of stoichiometric compounds also helps to complete the modeling of the X-ray physics taking place in the sample matrix, allowing for more accurate results over a broader range of sample composition.

**Conclusion**

Given basic information about the samples needing characterization, it is possible to input that information into the XRF modeling routines to describe elements that may not be within the official elemental range of the instrument. If the metal being deposited on a substrate is known, it is possible to characterize the deposition thickness by modeling the absorption physics of the deposition layer and measurement of the substrate signal. If the nature of the polymeric sample matrix from documentation or IR spectral measurements is known, it is possible to model the sample matrix and measure the trace metal content. If the stoichiometries of various compounds in a material are known, we can measure one element within the stoichiometric compound and model the remainder of the compound which was not directly detected. This highlights one of the often-overlooked advantages of XRF spectrometry, e.g., the ability to model the X-ray physics of a sample matrix without necessarily measuring every element within the sample matrix directly.
2020 Worldwide Events

**May 25-27**
Japanese Society of Microscopy (JSM) Osaka, Japan

**June 1-5**
Microscopical Society of Canada (MSC-SMC) Sherbrooke, QC

**June 24-26**
EDAX 2020 Ann Arbor, MI

**August 2-6**
Microscopy & Microanalysis (M&M) 2020 Milwaukee, WI

**August 3-7**
Denver X-ray Conference (DXC) Rockville, MD

**August 16-20**
Int’l Materials Research Congress (IMRC) Cancun, Mexico

**August 23-28**
European Microscopy Congress (EMC) 2020 Copenhagen, Denmark

**September 14-18**
MAFS/SWAFS/SAFS Joint Meeting Atlanta, GA

Note: We are closely monitoring developments relating to COVID-19. Please visit http://www.edax.com/support/training-schools for the most up-to-date information on our training courses.

2020 Worldwide Training

To help researchers obtain the most from their equipment and to increase their expertise in EDS microanalysis, WDS microanalysis, EBSD/OIM, and Micro-XRF systems, we organize a number of operator courses at the following EDAX facilities.

### EUROPE

**EDS Microanalysis (APEX™ EDS)**

- April 27-29
- May 4-6
- November 9-11
- November 30-December 2

**EDS Microanalysis (TEAM™ EDS)**

- September 28-30

**EBSD OIM Academy**

- September 14-17
- November 2-5

*Presented in English
#Presented in German

### JAPAN

**APEX™ EDS**

- May 14
- October 15
- November 19

**TEAM™ EDS**

- June 11
- July 9

**CHINA**

**EDS Microanalysis**

- June 2-4
- September 1-3
- December 1-3

**EBSD OIM Academy**

- May 12-14
- September 22-24
- December 8-10

**EDS Microanalysis**

- Shanghai (ACES)

**EBSD OIM Academy**

- Shanghai (ACES)

Note: We are closely monitoring developments relating to COVID-19. Please visit http://www.edax.com/support/training-schools for the most up-to-date information on our training courses.
Shibasis Chatterjee

Shibasis is EDAX’s new Sales Manager in India and joined us in January 2020. Located in Kolkata, his responsibilities include overseeing all sales activity across the region and maintaining close relationships with key customers and Electron Microscope Manufacturers (EMMs).

Prior to EDAX, Shibasis was the Manager of Product Marketing and Applications at Carl Zeiss India (Bangalore) Pvt. Ltd. for over nine years (2010-2019). In 2006, he earned a Bachelor of Technology in Applied Electronics and Instrumentation from Maulana Abul Kalam Azad University of Technology (MAKAUT). Shibasis completed his Master of Business Administration with a concentration on Marketing from the Indian Institute of Social Welfare and Business Management in 2010.

Shibasis and his wife, Sompy, have been married for nine years. The couple has an eight-year-old daughter named Debadrita. In his spare time, Shibasis enjoys reading books and listing to music.

Sill Wang

Sill Wang joined EDAX in April 2019 as a Service Engineer. Based out of Guangzhou, China, he helps customers solve their problems. His duties include repair, maintenance, and service support. Sill also provides users with familiarization training and schedules and plans maintenance visits, installations, and trouble calls in his region. He has a passion for his work, and it gives him a feeling of accomplishment when he can help customers with their difficult problems.

Prior to EDAX, Sill worked as a Research Assistant at the Guangzhou Energy Research Institute. Although he enjoyed his position, he decided to seek a new challenge rather than sit in the same lab every day. In 2008, Sill earned a bachelor’s degree in Applied Physics from Northeast University. He continued his education and received a master’s degree in Chemical Engineering from the Chinese Academy of Sciences in 2014.

Sill is engaged to his fiancée, Liu Yang. They started dating their sophomore year in college and plan on getting married in 2020. In his spare time, Sill enjoys not only watching but also playing soccer every weekend. He also likes to work out at the gym at least three times per week. Sill believes that is you have a healthy body, you will have a good life. He also feels that working out gives him the energy he needs to perform his job.
The Research Service Centers (RSC) in the Herbert Wertheim College of Engineering provide access to characterization and process instrumentation to support and enhance the University of Florida’s research, education, and public service missions. Expert staff members at the RSC assist and guide students, faculty, and industry users, so they get the most effective and appropriate use of the facilities.

The RSC utilizes four EDAX systems, including three Pegasus EDS-EBSD Analysis Systems on its FEI Helios, FEI Helios NanoLab 600i, and TESCAN MIRA 3 LMH Scanning Electron Microscopes (SEMs) and an Element EDS System on its CAMECA SXFive Electron Probe Micro Analyzer. The facility uses EDS analysis to do mapping correlation of Wavelength Dispersive Spectrometry (WDS) data and EBSD to acquire crystallographic information.

The RSC faces issues with data integration across analytical techniques. Many systems require the correlation of data from multiple sources when performing analysis and characterization. These data correlations are fundamental for the accurate understanding of behavior, response, and properties of certain materials and systems. Most of the users facing these types of data integration issues are researchers in geology, materials science, and engineering.

“We chose EDAX because it was the only analysis system with a hardware-software combination capable of the integration of WDS and EBSD mapping data,” said Engineer and RSC Director, Dr. Luisa Amelia Dempere. “The integrated EDS-EBSD-WDS system gives us the ability to use elemental trace analysis to elucidate the development of specific crystallographic configurations, microstructural transformations, or grain orientations.”

For more information about the RSC and the University of Florida, please visit: https://rsc.aux.eng.ufl.edu/.