EDAXinsight

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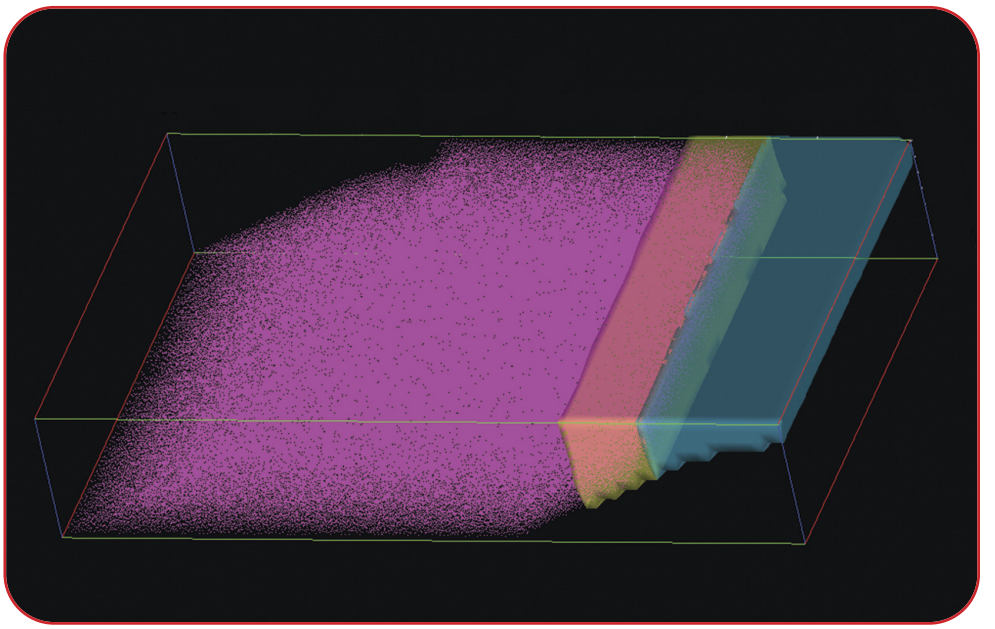
INSIDE





EDAX NEWS

# 3D Visualization of EDS Map Analyses From FIB Slices

Energy Dispersive Spectroscopy (EDS) in a Scanning Electron Microscope (SEM) was primarily designed for spectral acquisitions and composition quantification of important sample features. However, it has been used to map the elemental distributions of elements for decades. The EDS system controls the electron beam scanning over an area of interest and collects all of the X-rays for each pixel. It then extracts those elements with a high intensity or those selected by the user to provide a map image for each element. This provides the analyst with results to understand the 2D spatial distribution of the elements within the sample. Modern analysis techniques enable the analyst to examine the X-ray data for unique elemental mixtures and/or phases and provide the phase distributions.

A focused ion beam (FIB) can be employed to remove material from the sample. Most times the operation is used to cross-section a defect and provide a depth image through the feature. Another method of analysis includes the removal of a thin slice of material over a large area to expose a new surface. In this way, a series of image slices can provide results for the understanding of the 3D nature of the feature. EDS analyses can be performed on each slice to provide a number of elemental image maps. The number of slices can be small (<5), but modern automatic collection schemes can provide a high number of slices (>50). The volume of EDS data for so many slices within these analyses can make interpretation difficult and time consuming. In addition, understanding the true 3D morphology of the material from the series of 2D slices can be problematic.

A more practical method of understanding and interpreting the elemental data slices is to accumulate and visualize the data as a 3D volumetric display. The ability to rotate, zoom, and isolate key features aids in the interpretation. Previous 3D visualization software packages have focused on taking a stack of image slices and producing a 3D volume.

(Continued from Page 1)

Elemental maps could be displayed simultaneously by color coding according to intensity. The usual rotate/tilt/zoom functions designed for simple images were adapted for elemental maps, but provided little additional interpretive information.

EDAX has partnered with a sister company within AMETEK to provide a 3D solution for EDS data that performs both imaging and analysis operations within the same package. Our partner created its 3D software to be both a visual and analytical tool for interpretation of Atom Probe Tomography (APT) data. EDAX is using their extensive 3D imaging experience to provide the best 3D tool for EDS data. The developers have extended the source of typical 3D EDS data sets from simple elemental maps to full spectral imaging data sets for each FIB slice. When sub-sets of the data are extracted for interpretation, a complete spectrum is also extracted, from which full quantification can be performed. In this way, the most comprehensive visual and analytical interpretation of 3D EDS data can be performed.

**Examples**

A number of displays for a few samples are provided which illustrate a few of the capabilities of the new 3D EDS system.

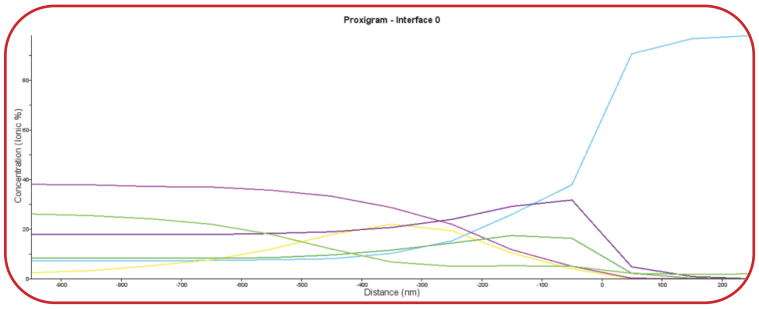



*Figure 2. A proxigram showing the elemental distributions normal to the O-K interface.*

The first example is from a CdTe multilayer structure. The display (cover image) is a hybrid that shows Cd-L as magenta dots, S-K as an orange volume and O-K as a blue transparent volume. Note the sharp interface between each of the volumes. From this data (Figure 2), a cumulative linear composition profile (proxigram) is created, which shows the elemental distributions normal to the O-K interface. The same sharp profile is observed in this plot as seen in the visual display.

This example shows the distribution of all of the X-rays with a rare-earth modified steel sample. Figure 3 shows the typical 2D elemental maps of the primary elemental lines. Interpretation using the individual slices is very difficult because of the spatial overlap of all of the elemental contributions and the difficulty in mentally combining the areas.

Figure 4 shows the 3D distribution of all of the X-rays as dots. Because there is so much information displayed at once, interpretation is complex.

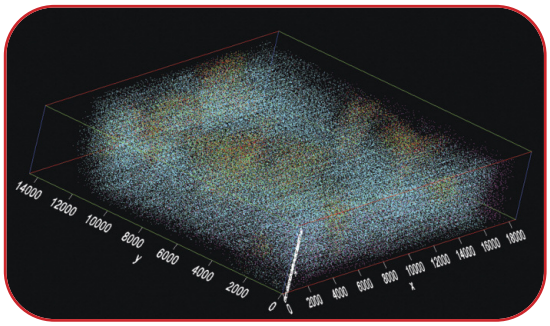


*Figure 4. The 3D distribution of all of the X-rays as dots.*

A preferred method is to place iso-concentration surfaces through the volume and only select a limited number of elements. In Figure 5, only the Nd-L enriched regions are shown. Note the distinct shape of each isolate particle.

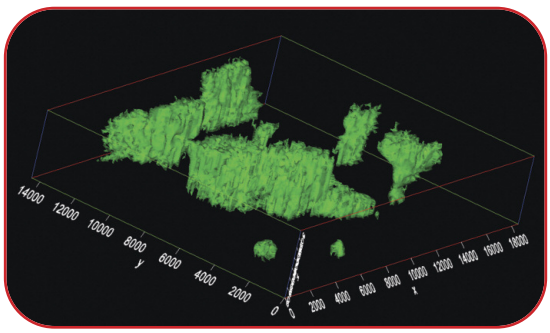


*Figure 5. A 3D distribution showing only the Nd-L enriched regions.*

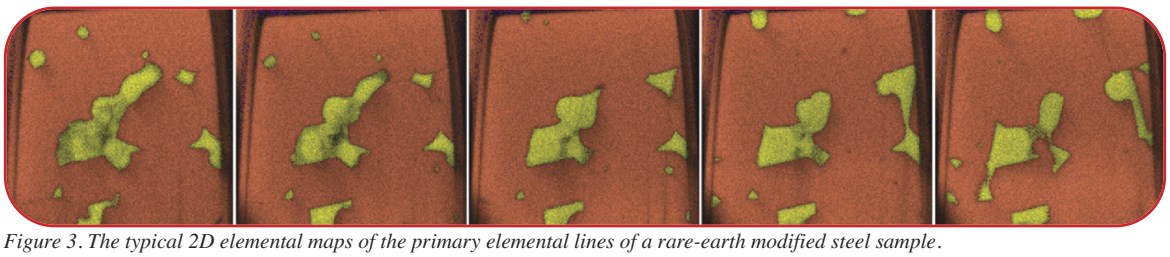


*Figure 3. The typical 2D elemental maps of the primary elemental lines of a rare-earth modified steel sample.*

(Continued from Page 1)

A cumulative spectrum is automatically provided for the whole data set (Figure 6). However, interpretation of the structure depends on understanding the spectrum of individual particles. These capabilities are shown in the spectra in Figure 7, which shows that the particle contains a significant enhancement of Nd-L and Pr-L compared with the average material.

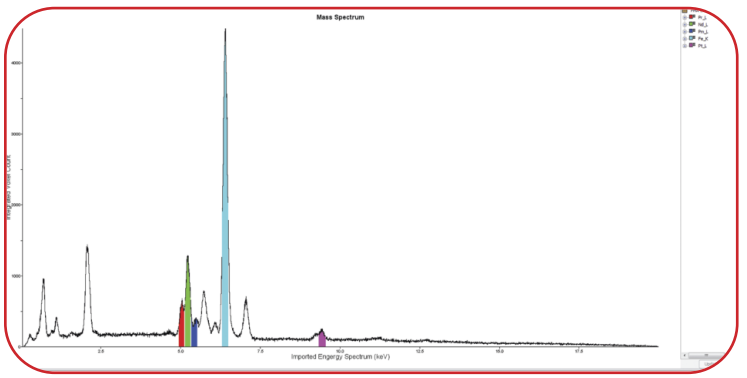



*Figure 6. A cumulative spectrum automatically provided for the whole data set.*

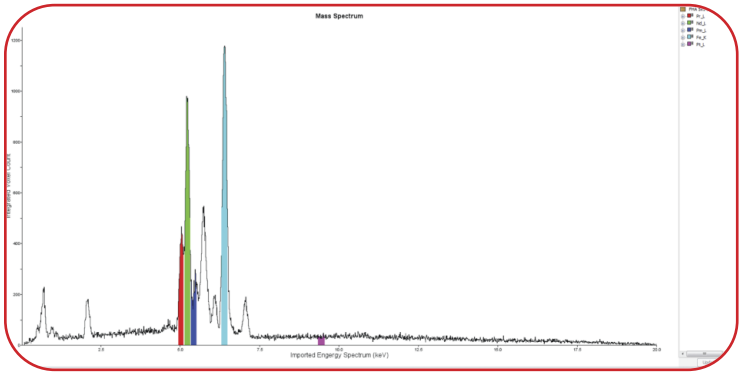



*Figure 7. Shows that the particle contains a significant enhancement of Nd-L and Pr-L compared with the average material.*

In Figure 8, a cumulative linear composition profile (proxigram) is created around particle 2, which shows the elemental distributions normal to the Nd-L interface. As expected, the Fe-K contribution decreases and the Nd-L and Pr-L contributions increase inside the particle.

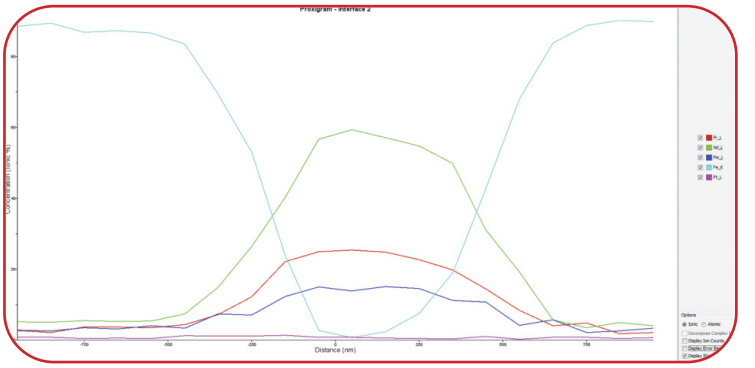



*Figure 8. A cumulative proxigram created around particle 2, showing the elemental distributions normal to the Nd-L interface.*

It is also possible to define a sub-volume within the data set. In this instance (Figure 9), a cylindrical volume is selected and oriented.

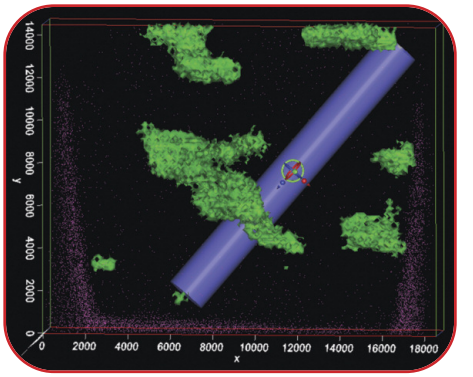



*Figure 9. A cylindrical volume of a defined sub-volume within the data set.*

A 1D composition profile (Figure 10) is derived from slices within this shape to provide a simple profile across the major particle in the analysis. As expected, the same elements are segregated as observed previously. Also note the sharpness of the interface between the matrix and the particle.

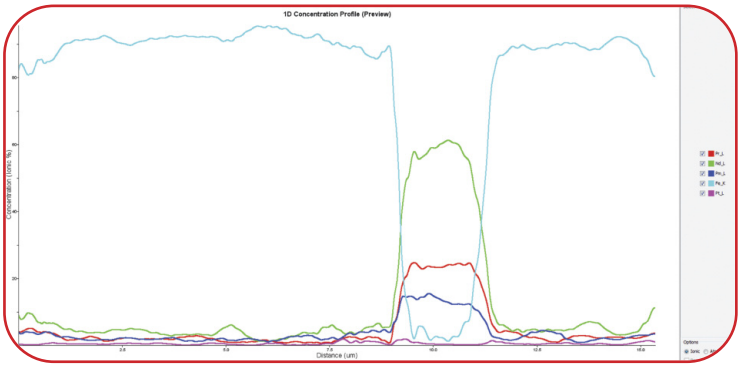



*Figure 10. A 1D composition profile derived from slices within the cylindrical shape to provide a simple profile across the major particle in the analysis.*

EDAX Smart Insight

APPLICATION NOTE

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

The Octane SDD Series for TEM was introduced recently at the 2014 Pittcon exhibition. The design evolved from the Apollo XLTW series of detector introduced in 2010. 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. This takes away the shadowing of the support and the subsequent reduction in solid angle 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.

These detectors are available in 30, 60, and 100 mm$^2$ active areas. The 30 and 60 mm$^2$ detectors are round, while the 100 mm$^2$ detector is an oval or racetrack shape. A NiO spectrum from the 100 mm$^2$ detector is shown below.

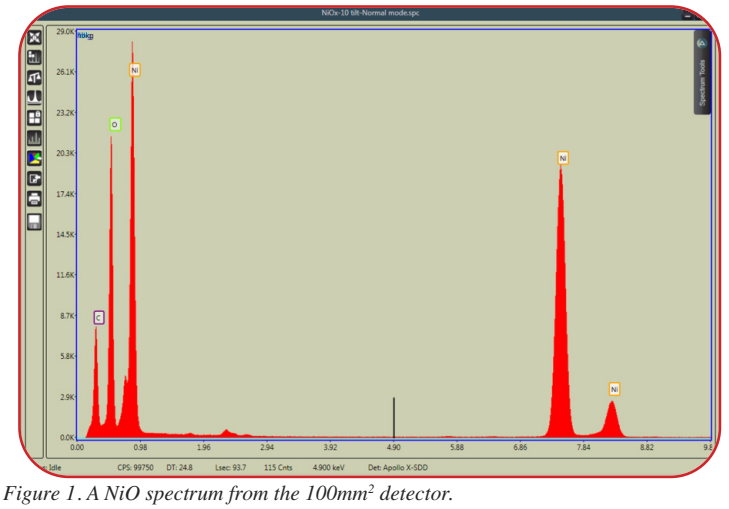


*Figure 1. A NiO spectrum from the 100mm$^2$ detector.*

The NiL to NiK ratio is very high, as well as the NiL to O K ratio. This is due to the lack of a window between the detector and sample. In addition, the sensitivity to N is greatly enhanced. A polymer window has a large concentration of C, which preferentially absorbs N. So, eliminating the window allows much more N X-rays to enter the detector.

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.

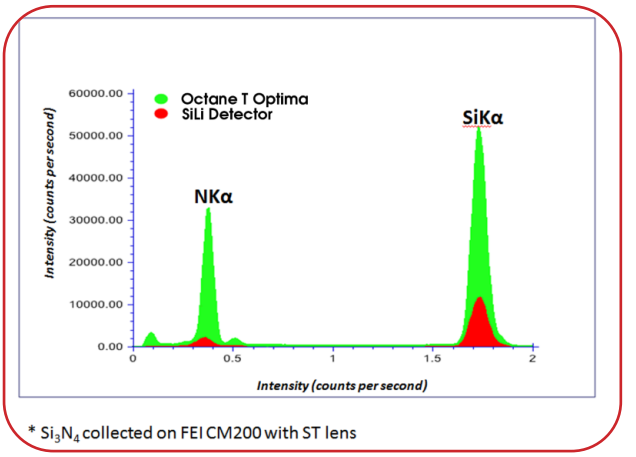



*Figure 2. A spectrum from a SiN 50 nm thick membrane.*

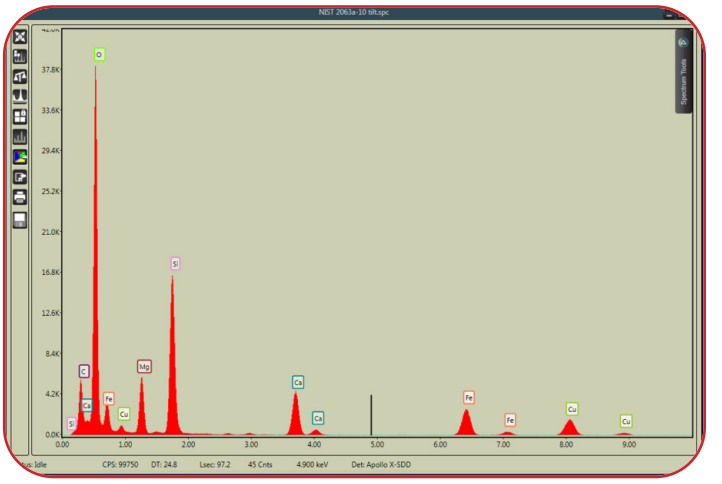


*Figure 3. A mineral glass thin film that is well characterized by NIST.*

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:

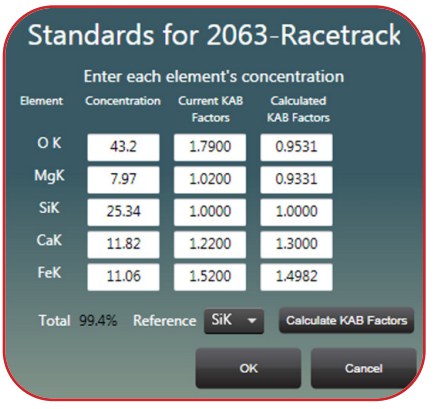



*Figure 4. The Cliff-Lorimer K-factors for the spectrum in Figure 3.*

(Continued from Page 4)

## Application Results

The new 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 new 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 $SrTiO_3$ 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.

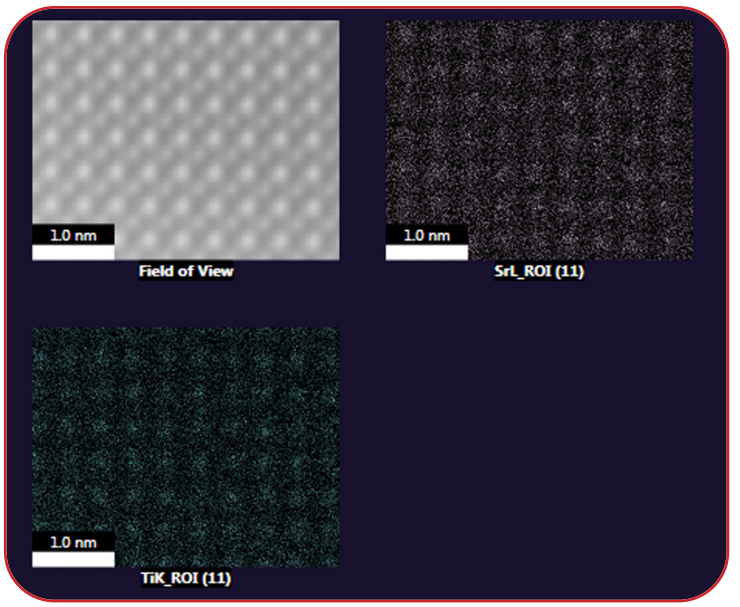



*Figure 5. STEM image and SrL and TiK maps for a sampe of $SrTiO_3$.*

The STEM image and the SrL and TiK maps are shown in the figure above. The SrL bright spots align with the bright areas of the STEM image, and the TiL spots align with the small centered spots.

Applying image processing to the net intensity images and overlaying Sr and Ti give a clear image of the atom locations.

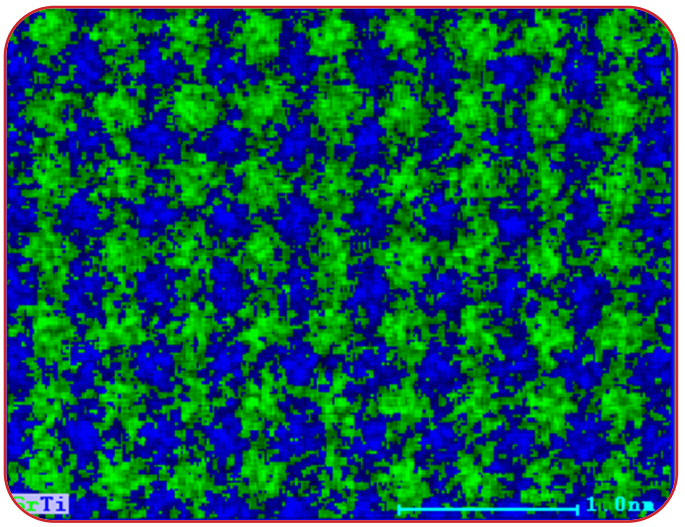



*Figure 6. Image generated by applying image processing to the net intensity images and overlaying Sr and Ti. SrL is green and TiL is blue.*

## 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.

## References

[1] Alan Sandborg, Mike Coy, Brent Hammell, Reinhard Buchhold M&M 2013

EVENTS AND TRAINING

# Worldwide Events

| | | | |
|---|---|---|---|
| **August 4-7** | | **September 15-18** | |
| Microscopy & Microanalysis | Hartford, CT | Int'l Conference on Electron Microscopy | Kraków, Poland |
| **August 24-29** | | **September 17-19** | |
| International Conference on Textures of Materials | Dresden, Germany | Materialography | Rostock, Germany |
| **September 3-5** | | **September 21-24** | |
| Japan Analytical & Scientific Instruments Show | Chiba, Japan | German Mineralogical Society | Jena, Germany |
| **September 7-12** | | **September 21-24** | |
| International Microscopy Congress | Prague, Czech Republic | European Microbeam Analysis Society | Leoben, Austria |

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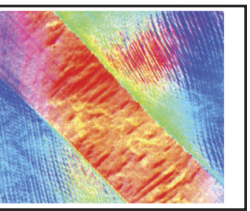
**Visit the EDAX Booth #202 at Microscopy & Microanalysis 2014**

EDAX will be demonstrating the full suite of leading TEAM™ characterization solutions, now including the fastest and most sensitive EBSD camera on the market and both new 3D and innovative PRIAS imaging.

For information on demos and presentations, please visit: www.edax.com/m-m



# 2014 Worldwide Training

To help our present and potential customers 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 EDAX facilities in North America; Tilburg, NL; Wiesbaden, Germany; Japan, and China.

## EUROPE

| | |
|---|---|
| **EDS Microanalysis** | |
| September 11-12 | Tilburg* |
| November 6-7 | Tilburg* |
| TEAM™ EDS | |
| September 23-25 | Tilburg* |
| December 2-4 | Wiesbaden# |
| **EBSD** | |
| September 15-17 | Tilburg* |
| November 3-5 | Tilburg* |
| **TEAM™ Pegasus (EDS & EBSD)** | |
| November 10-14 | Wiesbaden# |
| **TEAM™ WDS** | |
| October 14-16 | Tilburg* |
| **Orbis: Course & Workshop** | |
| October 28-30 | Wiesbaden# |

## JAPAN

| | |
|---|---|
| **EDS Microanalysis** | |
| Genesis | |
| October 9-10 | Tokyo |
| November 13-14 | Osaka |

## CHINA

| | |
|---|---|
| **EDS Microanalysis** | |
| TEAM™ EDS | |
| November 4 | Guangzhou |
| Decemer 1-4 | Shanghai (ACES) |
| Genesis | |
| September 15-18 | Shanghai (ACES) |
| **EBSD OIM™ Academy** | |
| October 20-23 | Shanghai (ACES) |

## NORTH AMERICA

| | |
|---|---|
| **EDS Microanalysis** | |
| TEAM™ EDS | |
| September 23-25 | Mahwah, NJ |
| **Micro-XRF** | |
| October 7-9 | Mahwah, NJ |

*Presented in English
#Presented in German

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AMETEK®
MATERIALS ANALYSIS DIVISION

# Arjun Dalvi

In June 2013, Arjun joined EDAX as the Sales Manager in India. Working out of Mumbai, his sales territory includes India, Bangladesh, Nepal, and Sri Lanka. Arjun is responsible for developing new market areas and building and maintaining good working relationships with electron microscope manufacturers.

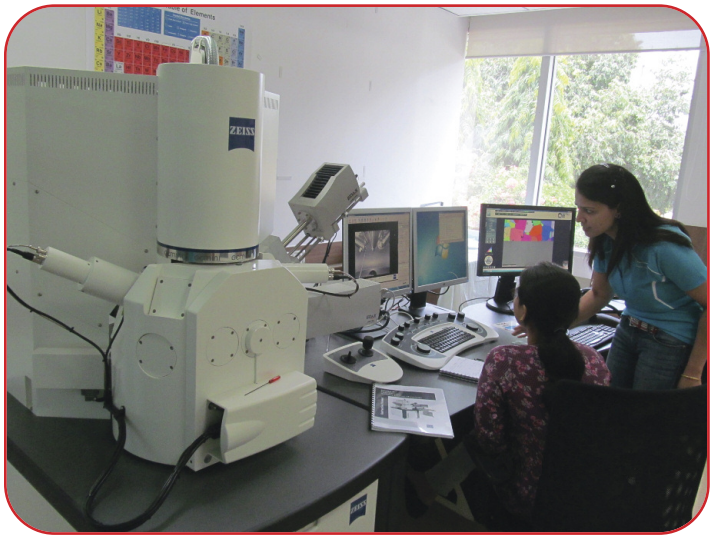
Prior to EDAX, Arjun worked at Elspec Ltd. as the Country Manager for Business Growth in the field of Power Quality Solutions and Analysis in India. From 2005-09, he was a regional manager at Hinditron Ltd. Arjun was responsible for ED-XRF and WD-XRF business in the steel and cement industries in western India. He served as a service engineer at Icon Analytical from 2003-05. Arjun performed the installation and service of FEI scanning electron microscopes and EDAX energy dispersive spectroscopy systems in western India.

*(left to right); Arya, Anusha, and Arjun Dalvi.*

Arjun received his bachelor's degree in Instrumentation and Controls Engineering from the University of Mumbai in 2003. One year later, he earned a Post Graduate Diploma in Business Management and Marketing from the Welingkar Institute of Management Studies.

Arjun lives with his wife, Arya and four-year old daughter, Anusha "Manu". In his spare time, he enjoys reading books, playing cricket, and spending time with Manu.

# EDAX Opens Demo Facility in India




*EDAX demo facility in Bangalore, India.*

EDAX has opened a highly equipped demo facility in Bangalore, India to increase the availability of important materials characterization technologies to scientists and microscopists around the world. The newly appointed laboratory houses a Zeiss Sigma VP Field Emission (FE) Scanning Electron Microscope (SEM) with an EDAX Trident system, including Octane Energy Dispersive Spectroscopy (EDS) Silicon Drift Detector (SDD), high speed Hikari Electron Backscatter Diffraction (EBSD) camera and LEXS Wavelength Dispersive Spectrometry (WDS) detector.

"The introduction of this advanced level system is an exciting addition to our worldwide demo labs and allows EDAX to stay at the leading edge of microanalysis, adding to our strong reputation as the technology leader in the scientific community," comments Tara Nylese, Global Applications Manager at EDAX. "It is our goal to understand and meet the real world needs of our customers so that we can continue to provide novel and valuable applications solutions to our users in all locations and many disciplines. With the opening of the new lab we are making excellent progress in this direction."

With the Trident system, local scientists in India will now have the ability to analyze their materials using EDAX's proven analysis tools, with TEAM™ Phase Mapping for compound chemical analysis and elemental distribution. Using EBSD, they can then understand the underlying microstructure of samples to reveal how grain and orientation data relates to materials properties. Finally, a WDS analysis will take the investigation to the most advanced levels with maximized sensitivity for trace level compositions and the most difficult spectral peak differentiation. The FE SEM can magnify samples several hundred thousand times, which allows visualization of nanomaterials in the tens of nm and smaller. The new laboratory is located in a pleasing setting within the AMETEK facilty in Bangalore. Visitors will work alongside scientists and engineers from other AMETEK Business units. The availability of all of these technologies in a single location will provide EDAX customers with a comprehensive and useful analytical experience.

CUSTOMER NEWS

# University of Notre Dame Nuclear Science Laboratory

Researchers from the Nuclear Science Laboratory (NSL) at the University of Notre Dame pursue a broad program in low energy nuclear physics with emphases on Nuclear Astrophysics and Nuclear Structure. In recent years, they have begun working with various departments across campus for various inter-disciplinary projects.

As part of this initiative, researchers at the NSL are collaborating with the Curator of Ancient and Medievel Manuscripts from Rare Books and Special Collections in the Hesburg Library, Dr. David T. Gura, on a project to study a medieval manuscript [1] using an Orbis Micro-XRF System to non-destructively analyze the manuscript's ink pigments. The Orbis is housed in the Center for Environmental Science & Technology (CEST) to support University researchers in their work encompassing electronics, catalysis, materials, and cultural artifacts. Preliminary micro-XRF analyses, concerning the medieval manuscript, have proven very promising. The simplicity of the device and analysis software make it an ideal tool for undergraduate research projects. Performing 2D scans as shown in Figure 1 allows for non-destructive determination of the various pigments used to create these documents. The elemental distribution from micro-XRF provides information on the inorganic elements in the ink and paint pigments, which confirms or rejects many of the possible known pigment compounds used at the time of the writing of the manuscript. Typically, complementary techniques like Raman spectroscopy can then be applied to verify the specific pigments used. Coupled with the reported provenance of the document, this type of information is invaluable in determining how manuscripts of that origin were written, as well as identifying counterfeits. This is just one example of the wide range of items being studied with the Orbis. It has quickly become one of the most frequently used analytical systems in CEST and has become a valuable addition to the research programs across campus.

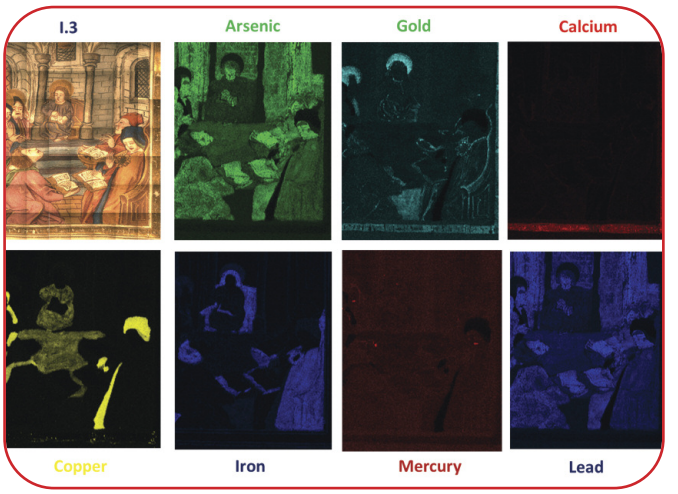



*Figure 1. Non-destructive analysis of the various pigments used to create medieval manuscripts using the Orbis Micro-XRF Elemental Analyzer.*

*[1] University of Notre Dame, Hesburgh Library, Frag. I.3.*

*Contributed by: Researchers at the Nuclear Science Laboratory, Khachatur V. Manukyan, and Dr. David T. Gura*

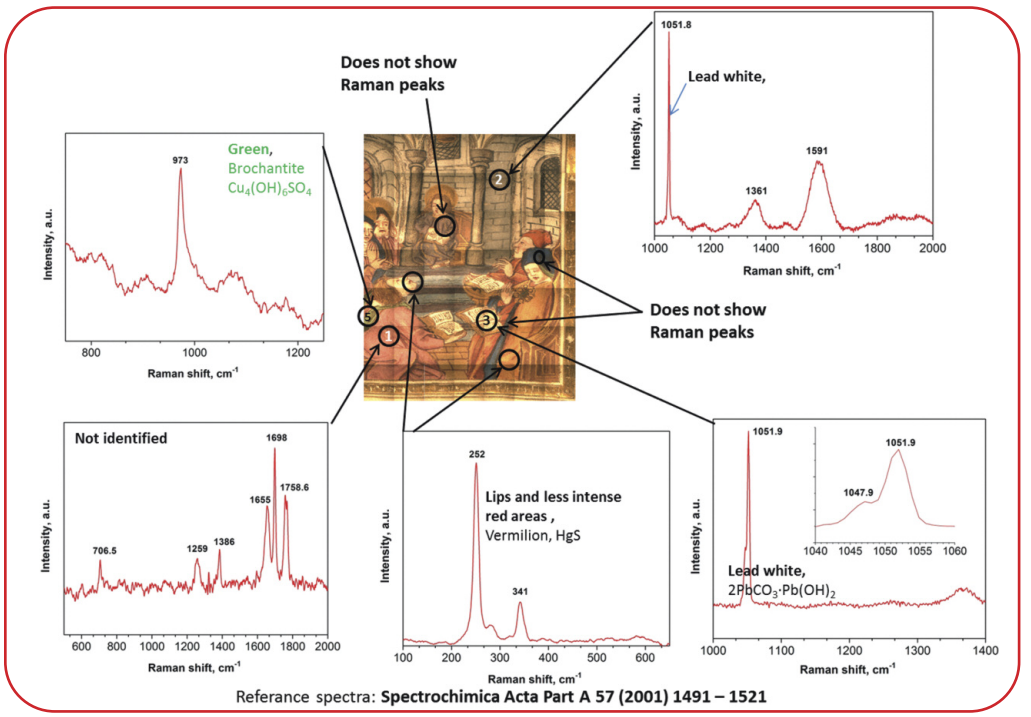



*Figure 2. The precise pigments used are verified using Raman Spectroscopy.*

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