EDAXinsight

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Happy Holidays from all of us at EDAX

EDAX NEWS

Fast Acquisition of Quality EBSD Data on Multiple Sample Types Using the New Hikari Super Detector

Users rarely want to collect data slowly. They are busy, juggling many responsibilities and want to be able to collect data quickly and easily. But they also want quality, useful data. Collecting bad data fast is not their goal. The new Hikari Super Electron Backscatter Diffraction (EBSD) detector, part of the Hikari Camera Series, is the fastest EBSD detector available, and is capable of rapidly collecting accurate data from a variety of samples to meet user needs.

To achieve fast EBSD data collection, the CCD sensor is binned to use smaller pixel resolution images that can be acquired with a faster frame rate and higher signal sensitivity. With the Hikari Super camera, the 640 x 480 pixel resolution sensor can be binned into 16 x 16 blocks, and patterns of 30 x 30 pixels can be acquired at frame rates greater than 1400 patterns per second. A 30 x 30 pixel EBSD pattern often looks pixelated, but the Hough transform, used to detect band positions within the EBSD pattern, is optimized in the TEAM™ software to work at this resolution at the fastest acquisition speeds.

Figure 1 shows that the indexed points per second (ipps) are important, as they indicate that not only are the patterns collected at this speed, but the bands are also detected and an orientation solution was obtained using the TEAM™ triplet indexing algorithm. The indexing success rate is greater than 99% at this speed, using confidence index filter values greater than 0.2.
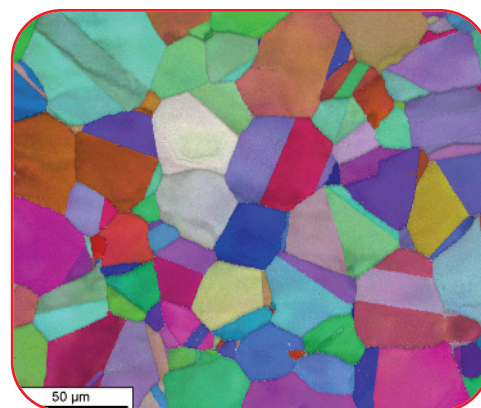

Figure 1. An orientation map collected at an acquisition speed greater than 1400 ipps from an Inconel 600 nickel superalloy sample.

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Nickel superalloy is an easy sample for EBSD. It has a relatively large average atomic number, which increases the amount of backscattered electrons and increases the detected signal level. It has a relatively large grain size and is reasonably free of deformation, which would degrade EBSD pattern quality. This is why we use it as one of the reference standards with each EBSD system. So what happens if we choose samples from the real world that are inherently more difficult, and try to collect fast data?

Our first real world sample is a friction stir weld of an aluminum alloy. The data is collected from the weld nugget, where the grain size is smaller. The smaller grain size means a smaller step size is required to measure the microstructure, and the smaller step size means we have more data points collected adjacent to grain boundaries. With the lower average atomic number of aluminum, the electron interaction volume is larger, which means we should collect more overlapping EBSD patterns near grain boundaries. The triplet indexing approach is designed to help deconvolute these overlaps, but as we decrease pixel resolution and increase acquisition speeds, at some point it becomes difficult to accurately determine the correct resolution. In Figure 2, as expected, most of the non-indexed points are located near grain boundaries. However, the 1400 ipps data does give reasonable microstructural data at these speeds, and for many applications (grain size, texture) this data is sufficient. Of course, an OIM™ Analysis cleanup routine could also be used to improve the visual appearance of the data, but it is important to assess the data quality prior to cleanup to determine if and when this is appropriate.
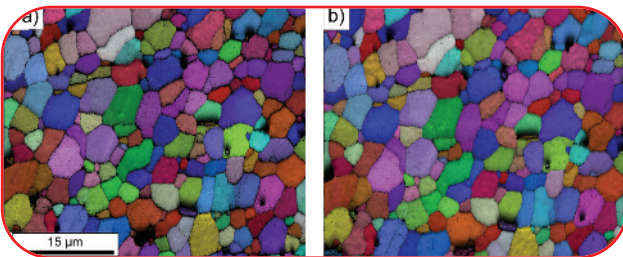

Figure 2. Orientation maps collected at 1400 (a) and 1000 (b) ipps from the aluminum weld nugget. The indexing success rate is 90% at 1400 ipps and 97% at 1000 ipps. Non-indexed points are colored black.

Our second real world sample is a brass alloy cold rolled to 11% reduction. This process introduces plastic deformation into the microstructure, and the deformation reduces EBSD pattern quality. With increasing deformation and the smaller pixel resolution used for the fastest data collection, it becomes harder to accurately detect band positions and determine crystal orientation. In Figure 3, the GROD maps show the plastic deformation fields within each grain. Comparing the 1400 ipps and 1000 ipps, it can be seen that regions of low confidence indexing in the 1400 ipps data correspond to areas of higher plastic deformation. However, the 1400 ipps data does provide
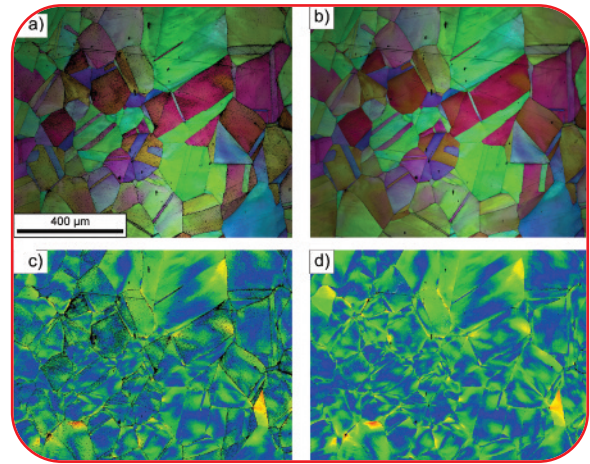

Figure 3. Orientation maps and grain reference orientation deviation (GROD) angle maps from data collected from the brass sample at 1400 ipps and 1000 ipps.

a decent representation of the deformed microstructure at these collection speeds. For this 1400 ipps data, over a million data points were collected in under 12 minutes.

Both of these examples have been cubic materials. High speeds are usually more easily obtained on cubic materials because the EBSD patterns are not as complex, and therefore do not require a high pixel density EBSD pattern to accurately detect the EBSD diffraction bands. Alpha phase titanium alloy is used for our third real world sample (Figure 4). This titanium phase is hexagonal. The indexing success rates were 99% at 800 ipps, 98% at 1000 ipps and 97% at 1150 ipps. While the indexing success drops slightly, at these fast acquisition speeds the non-cubic data is still representative and acceptable. These results clearly show that higher speed collection from non-cubic materials is possible with the Hikari Super.

In summary, using the Hikari Super EBSD camera and TEAM™ software, high-speed collection is possible on a wide range of samples, including low atomic number samples, deformed samples, non-cubic samples and multiphase samples. The Hikari Camera Series collects data at high speed when throughput is essential and performs at the same high indexing rates under challenging conditions.
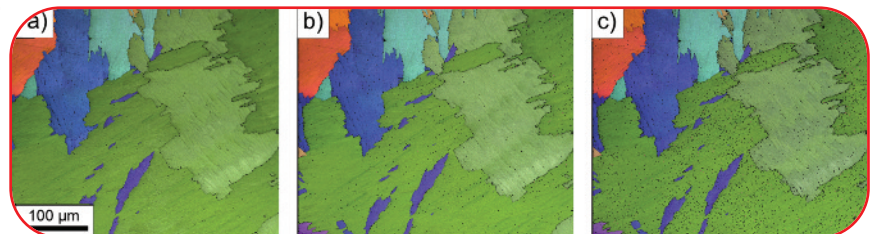

Figure 4. Orientation maps collected from this sample with EBSD patterns at 80 x 80 pixel resolution collected around 800 ipps (a), 60 x 60 pixel resolution collected around 1000 ipps (b) and 48 x8 48 pixel resolution collected around 1150 ipps (c).

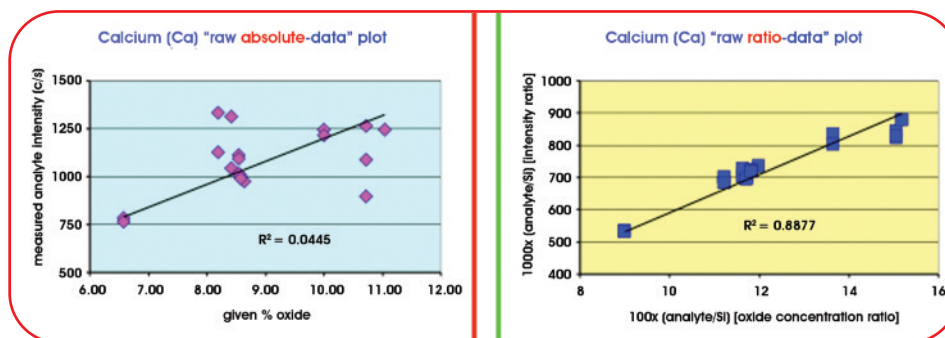# Utilizing Internal Intensity Ratios in Micro-XRF

Figure 1. Ca intensity plots showing the benefit of the internal ratio method (right plot). Wt% quantification is achieved by normalizing the results to 100 wt%; ratioing to a single element and assuming intensity variations from sample topography only.

In X-ray Fluorescence (XRF) analysis, elemental intensities can be affected by sample topography and thickness if the sample is not thick enough to be considered a "bulk" sample. This can make quantitative or comparative analysis difficult to perform. To compensate for the influence of sample topography on absolute elemental intensities, one can employ an internal ratio methodology where the elemental intensity of one element is compared to the intensity of another element from the same sample. This is useful, for example, when analyzing small glass fragments of varying size and shape non-destructively with micro-XRF. Figure 1 shows an application of this method for the quantification of Ca in SiO_2-based glass fragments.

The internal ratio method of calculating elemental intensity ratios is implemented in Vision32 and OrbisVision software packages.

Figures 2a & 2b. Figure 2a shows the path for finding the Internal Ratio mode. Figure 2b shows the set up window, where the ratio elements are defined.

Furthermore, this intensity ratioing mode can also be used in conjunction with automated multi-point acquisition, allowing large batches of ratios to be processed without the need for manual calculations or cutting and pasting.

To set up this automated processing feature:
1. Label all elements of interest in the spectrum using the Peak ID list, including the ratio elements.
2. In the Quantification panel of the Vision software, open the Options window, and select the dropdown menu for "Intensity Mode", as shown in Figure 2a.
3. Select "Internal Ratio" and a side window will appear, containing a list of all identified elements in that spectrum.
4. Highlight an element and type the ratio element in the entry box above (elemental symbol followed directly by K, L, or M), as shown in Figure 2b. Elements without a ratio will be calculated as an absolute intensity.
5. Click OK to exit the Options window. If all the ratios have not been filled in, an alert message will appear (Figure 3).
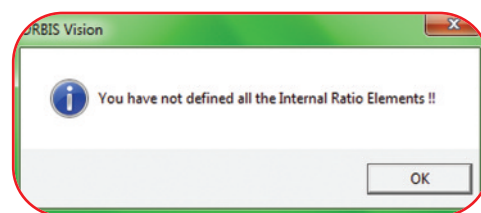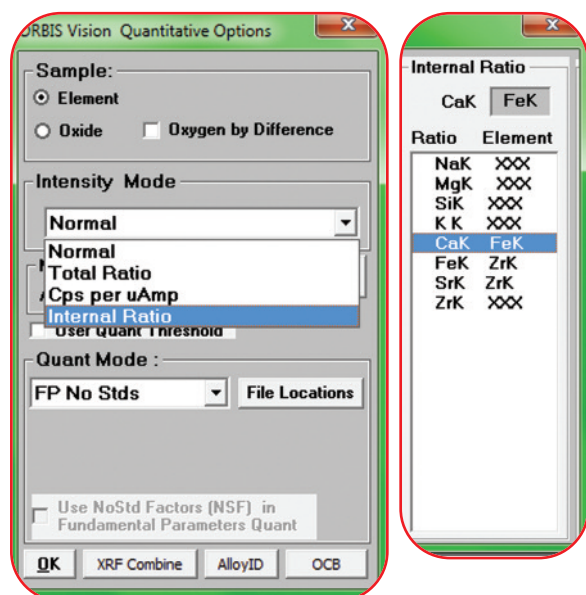

Figure 3. Alert message if all the ratios have not been filled in.

6. If the ratio list is correct, click OK to proceed.
7. At this point, all spectra quantified for intensities, individually or in a batch, will be calculated using the internal ratios defined. The software will implement the internal ratio calculations for both live, multiple point acquisitions and spectral reprocessing of a series of spectra.

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Phase Identification Through Mapping and Spectrum Matching

With the addition of Spectrum Matching to the EDAX TEAM™ software package, another powerful and time saving tool is just a few clicks away. Spectrum Matching is an automatic function that allows the user to search through a custom built spectrum library to find similar spectra. This greatly simplifies identification of unknowns by comparing them to a group of potential candidates and reduces the complexity of finding discrepancies and similarities between spectra.

Figure 1. EDS spectrum with two matching spectra overlaid.

The matching of the active spectrum to those found in the library file is based on a chi squared goodness of fit test, comparing either concentrations or spectra directly. The match sensitivity is adjustable, giving the user full control over the match results, ranging from completely identical to roughly in the same ballpark. The potential matching spectra are overlaid on the original spectrum with the match percentage giving a measure of how similar or dissimilar the spectra are along with a visual representation of the differences. Figure 1 shows an example spectrum with two overlaid spectra with a match value of 81.53% for SbS and 62.64% for $CaSO_4$. A visual inspection quickly tells us that the discrepancies between the sample and the best matching spectrum are to be found in the Si, Ti, Ba and Zr content.

Building a spectrum match library in the TEAM™ software is made easy through the included spectrum search utility. The user is able to search the full database of spectra stored on the system either through manual selection of specific project nodes or by adding filters to identify spectra with the desired characteristics. The filters include acquisition date, presence of given elements (with range in atomic %, weight % or net intensity), sample name and kV used. Once the spectra are selected, they are saved to a Spectrum Matching Library file, which can be shared amongst users and systems. It is even possible to add all data in the system database to a Spectrum Matching Library file in order to finally answer the nagging question of "Hmm, where did I see this before?"

Combining phase mapping and Spectrum Matching highlights the benefits gained from both techniques. In the current example, we have built a spectrum library based on 15 different mineral standards and applied the library to a high quality 512x400 pixel map of an automotive composite sample. During the one hour data acquisition, the phase mapping routine automatically separated and color coded the different phases of the sample as shown in Figure 2.
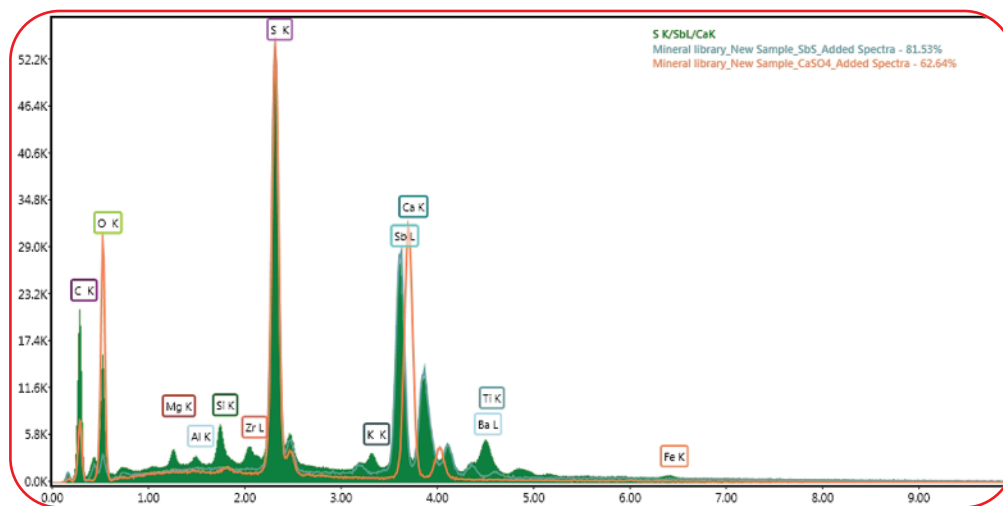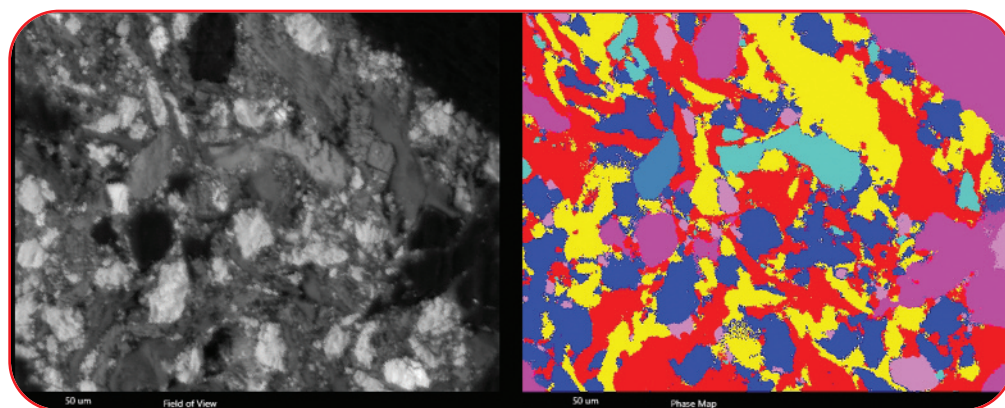

Figure 2. SEM Image (left) and phase map (right) of the composite sample.

(Continued from Page 4)

Upon identification of the phases, the TEAM™ software automatically generates a phase spectrum from the pixels in each individual phase. These phase spectra can easily be pulled up in the software and compared to a match library. Figure 3 illustrates the result of extracting the blue phase spectrum from the phase map and matching it to the minerals library, resulting in an 85.33% fit to a barite standard. Figure 3 also shows the Ba atomic % map alongside the phase map, confirming the presence of Ba in the blue phase. By matching the other phase spectra, we can rapidly identify the red phase as almandine garnet, steel blue as magnetite, turquoise as strontium sulfate, yellow as a mix of barite and titanium dioxide, and violet as lead sulfate, while fuchsia is assigned to the carbon embedding material.

While this example shows the matching routine applied to a mineral sample, Spectrum Match is a versatile tool that finds a multitude of applications. These include failure analysis and quality control where defects can be compared to a library of potential contaminants, process control where changes in distribution and composition can quickly be identified with the combination of phase mapping and spectrum matching, or reverse engineering where material constituents can be matched against a library of standard components found in the material. Whichever the application, users will now be able to find their match with the click of a button.
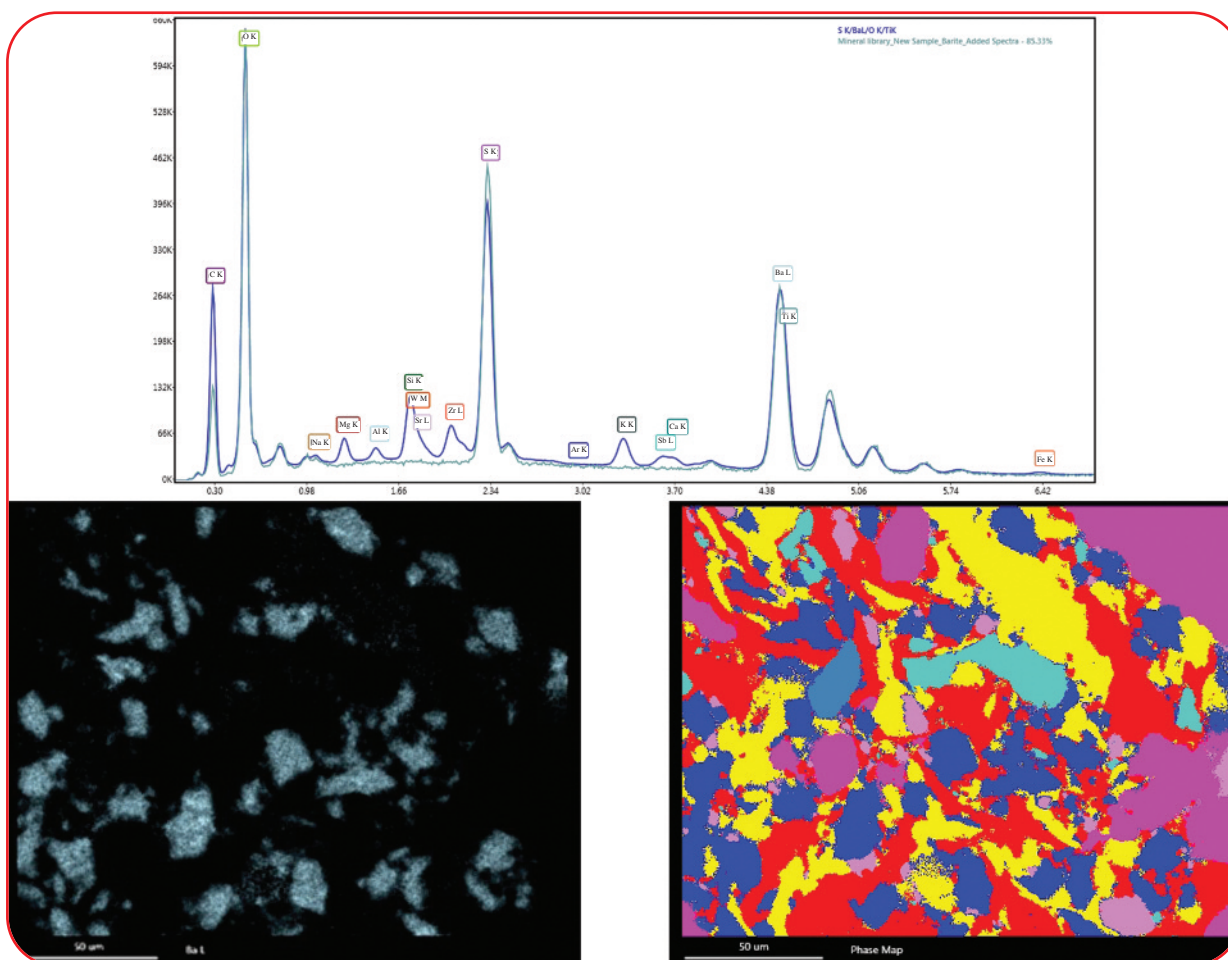

Figure 3. Sum spectrum of the blue phase matched to the mineral library (top), Ba atomic % map (lower left) and phase map (lower right).

Worldwide Events

February 16-20
American Academy of Forensic Sciences (AAFS) Orlando, FL

February 25
FIBSEM 2015 Laurel, MD

March 3-5
International Conference on Nano Science and Technology Chandigarh, India

March 8-12
Pittcon 2015 New Orleans, LA

March 15-19
The Minerals, Metals & Materials Society Orlando, FL

April 6-10
Materials Research Society (MRS) Spring San Francisco, CA

April 14-17
Analitika 2015 Moscow, Russia

May 13-15
Japanese Society of Microscopy Kyoto, Japan

Please visit www.edax.com/Event/index.aspx for a complete list of our tradeshows.

2015 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	
TEAM™ EDS	
February 10-12 February 23-25 May 18-20	Tilburg* Wiesbaden# Wiesbaden#
Genesis	
March 23-25 April 14-16	Wiesbaden# Tilburg*

EBSD	
February 25-27 March 16-18 June 8-10	Wiesbaden# Tilburg* Tilburg*

TEAM™ Pegasus (EDS & EBSD)	
February 23-27	Wiesbaden#

TEAM™ WDS	
March 3-5 May 20-22	Tilburg* Wiesbaden#

TEAM™ Neptune (EDS & WDS)	
May 18-22	Wiesbaden#

XRF	
March 24-26	Tilburg*

*Presented in English
#Presented in German

JAPAN

EDS Microanalysis	
TEAM™ EDS	
June 11-12 July 2-3	Tokyo Osaka
Genesis	
February 5-6 April 16-17	Tokyo Osaka

CHINA

EDS Microanalysis	
TEAM™ EDS	
March 24-26 September 8-10	Shanghai (ACES) Shanghai (ACES)
Genesis	
June 9-11	Shanghai (ACES)

EBSD OIM™ Academy	
April 14-16	Shanghai (ACES)

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
February 23-24 March 17-19 May 18-22	Draper, UT Mahwah, NJ Mahwah, NJ

EBSD OIM™ Academy	
February 25-27 June 16-18	Draper, UT Mahwah, NJ

Micro-XRF	
March 24-26	Mahwah, NJ

TEAM™ Pegasus (EDS & EBSD)	
February 23-27	Draper, UT

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(left to right): Hoa and Sia Afshari.

Sia Afshari

In September 2014, Sia joined EDAX Global Marketing as the X-ray Fluorescence (XRF) Program Manager in Mahwah, NJ. He is responsible for expanding the XRF product line and developing new products.

Prior to EDAX, Sia was the product manager at RMD Instruments, the project manager at B&L Analytical Division and a scientist at Diano Corporation. For over 25 years, he has been involved with development and marketing of analytical instrumentation, non-destructive testing (NDT) applications and spectrometric methods especially with XRF/X-ray Diffraction (XRD) systems.

Sia received Bachelor of Science and Master of Science degrees in nuclear engineering from the University of Massachusetts Lowell with graduate work in material science. He was trained in X-ray technology by Steve Neff and Ron Jenkins.

Sia and his wife, Hoa live in Burlington, MA. They enjoy travel and outdoor activities.

(left to right): Tate, Matt, Finn, Melissa and Parker Nowell.

Matt Nowell

Matt joined TexSEM Labs (TSL) in 1995. He was part of the team that pioneered the development and commercialization of EBSD and Orientation Imaging Microscopy (OIM™). After EDAX acquired TSL in 1999, Matt worked with the applications group to continue to develop EBSD as a technique and integrate structural information with chemical information collected simultaneously using EDS.

At EDAX, Matt has been involved in a number of roles, including product management, business development, customer and technical support, engineering, and applications support and development. Currently, he is the EBSD product manager in the Draper, UT office. Matt has published over 80 papers in a variety of application areas. He enjoys interacting with scientists, engineers and microscopists to help expand the role that EBSD plays in materials characterization.

Matt earned a bachelor's degree in Materials Science and Engineering from the University of Utah in 1995. He helped manage the University's MSE EM lab. Matt taught students to use SEM, TEM and XRD equipment and learned to appreciate how much easier it is to measure orientations with EBSD.

Matt and his wife, Melissa have enjoyed traveling to various EM conferences together over the years. They have three children, Parker (17), Tate (9) and Finn (7). Finn was born four months early, giving Matt the opportunity to collect various samples for EBSD analysis while Finn was in the NICU. In his spare time, Matt enjoys playing golf and pondering if changing the texture of his clubs will affect his final score, learning about Minecraft and relearning Calculus.

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Bergen County Academies
Nano-Structural Imaging Lab

Located in Hackensack, NJ, the Bergen County Academies (BCA) is a free public magnet high school for gifted and talented students. It provides advanced curriculum, technologies and opportunities for students that are normally not available in their home school district, including a graduate level independent research program. Currently, BCA has an enrollment of approximately 1,100 students in grades 9-12.

The Nano-Structural Imaging Lab at BCA is open to all students who need to characterize a sample with the use of electron microscopy or Energy Dispersive X-ray Spectroscopy (EDS). Students in the nanotechnology program are synthesizing a wide variety of nanomaterials for applications in material and biological sciences. Through the use of a Transmission Electron Microscope (TEM), the lab has the ability to characterize samples in terms of morphology, but previously could not determine the composition of these materials. Students need to understand the composition of a material to get a full understanding of their sample and determine whether their synthesis methodology is working as expected. This information is used in a variety of national and international competitions, publications and conferences.

By adding an EDAX Apollo XLT2 Analysis System to the TEM, BCA students are now able to get a complete understanding of the materials they are synthesizing and can determine the effectiveness of their synthesis technique. The EDS system is also used to teach students how to choose the appropriate equipment necessary for the type of data they are trying to collect and to build their technical skill set.

This year, the Nano-Structural Imaging Lab at BCA started a new class called Topics in Science and Research, in which freshmen rotate through three of the five research labs. In the Microscopy segment of the course, transmission electron microscopy, spectroscopy and spectrophotometry are utilized to analyze nanoparticles. Students have the opportunity to gather their own data, including TEM images and EDS data.

"The EDAX Apollo XLT2 Analysis System is very effective in analyzing the composition of nanomaterials. This would be a very important tool for any laboratory involved in synthesis of nanomaterials for any application. It could also be useful in the quality control process for any product that can be analyzed at the nanoscale," said Alyssa Waldron, Electron Microscopy Research Teacher.

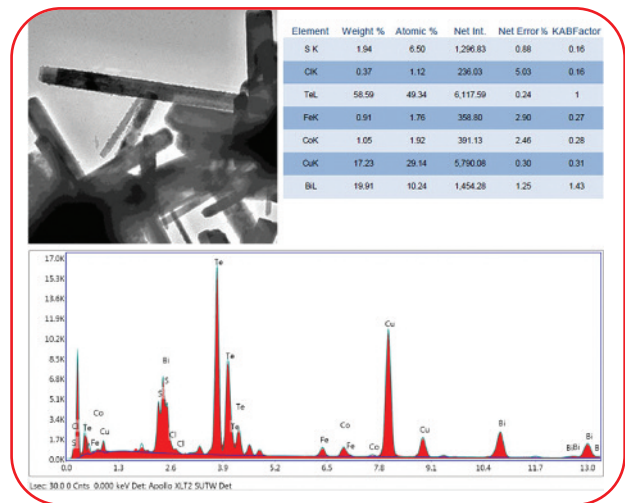| Element	Weight %	Atomic %	Net Int.	Net Error %	KABFactor
S K	1.94	6.50	1,296.83	0.88	0.16
Cl K	0.37	1.12	256.03	5.03	0.16
TeL	58.59	49.34	6,117.59	0.24	1
FeK	0.91	1.76	358.80	2.90	0.27
CoK	1.05	1.92	391.13	2.46	0.28
CuK	17.23	29.14	5,790.08	0.30	0.31
BiL	19.91	10.24	1,454.28	1.25	1.43

Figure 2. TEM micrograph (top left), quantitative EDS analysis (top right) and spectrum (bottom) of bismuth telluride nanorods synthesized by BCA students.

Figure 1. Mr. Craig Queenan (left) and Mrs. Alyssa Waldron (right) teaching the students in the Topics in Science & Research: Microscopy course how to use the EDAX Apollo XLT2 Analysis System to acquire elemental data from a sample, while students operate the microscope.

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