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INSIDE

EDAX NEWS

NPAR – A Novel Approach to Accelerating and Extending EBSD Indexing Performance

A critical aspect of the automated analysis of Electron Backscatter Diffraction (EBSD) patterns for the determination of crystallographic phase and orientation is the detection of the diffraction bands via image processing. If the diffraction bands are found consistently and correctly, the indexing routines generally work very well. Conversely, if the bands are not found, indexing performance will degrade.

Band detection performance depends on the signal-to-noise ratio (SNR) within the EBSD pattern. Noise and decreased SNR can be introduced into the EBSD patterns through the use of camera gain to amplify the signal level from the EBSD camera. The use of camera gain allows the cameras to operate at faster frame rates, while keeping the beam current and effective dynamic range constant. As long as the diffraction bands can be detected at a given gain/noise level, EBSD indexing performance will be acceptable. Figure 1 shows the indexing success rate as a function of camera noise for a range of engineering materials (dual phase titanium, rolled aluminum, poly-silicon, and yttria-stabilized zirconia). Two types of behavior are observed. For the Al, Si, and Zr materials, indexing success rates are initially constant, until a critical noise level is reached at which indexing performance begins to drop. For the Ti material, indexing performance immediately drops with the addition of noise and decrease in SNR.

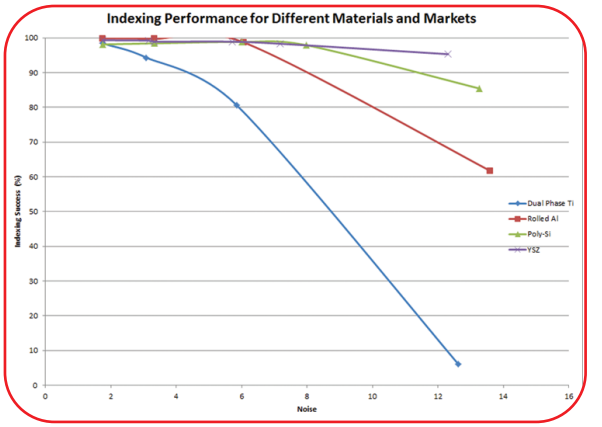

Figure 1. The indexing success rate as a function of camera noise for dual phase titanium, rolled aluminum, poly-silicon, and yttria-stabilized zirconia.

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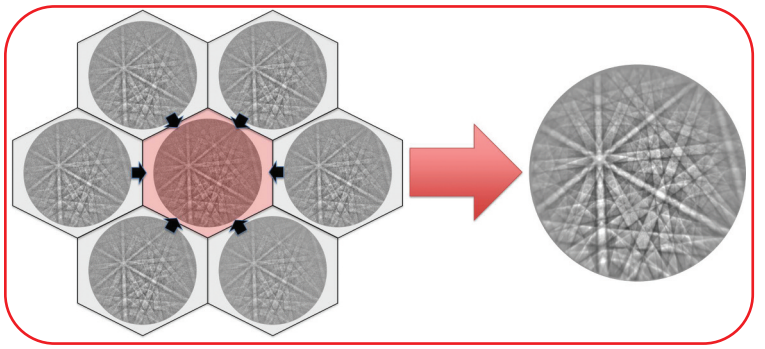

Figure 2. A schematic showing how NPAR averages the EBSD pattern at each point with the EBSD patterns from each of its neighboring points to reduce noise.

When operating at conditions that decrease indexing results, there are a few traditional options available to improve performance. The first is to decrease the camera gain level to reduce noise and improve signal-to-noise. The camera exposure time is then increased to maintain camera signal levels, which reduces the acquisition speed. A second approach is to increase the Scanning Electron Microscope (SEM) beam current, which allows the gain and noise levels to be decreased. However, this approach is not always practical, it is due to either SEM or sample limitations. For example, some non-conductive or semi-conductive samples can only tolerate a certain beam dosage before charging becomes an issue. A third approach is to average multiple camera frames in order to reduce temporal noise effects. The drawback to this approach is that it again reduces the acquisition time by a factor of the number of frames averaged.

With the release of TEAM™ 4.3, Neighbor Pattern Averaging and Reindexing (NPAR) is now available. This new software package provides an innovative and patent-pending approach to improving EBSD pattern signal-to-noise while maintaining faster acquisition speeds. As shown schematically in Figure 2, NPAR averages the EBSD pattern at each point with the EBSD patterns from each of its neighboring points to reduce noise. With the use of NPAR, the degradation of indexing performance with increasing noise is minimized, and the result is significantly better than conventional indexing, as shown in Figure 3.

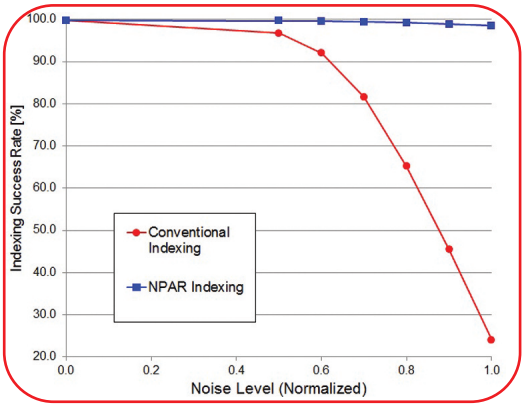

Figure 3. Shows how with the use of NPAR, the degradation of indexing performance with increasing noise is minimized and is significantly better than conventional indexing.

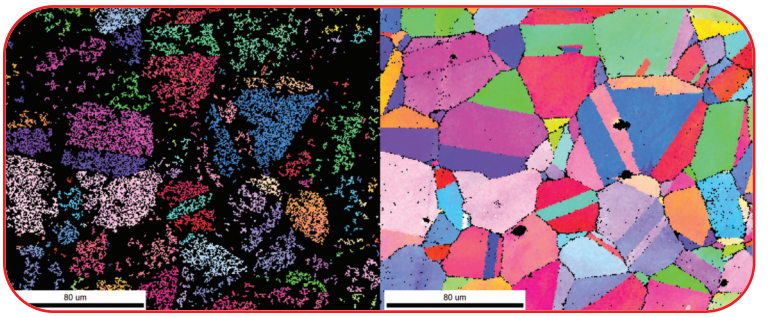

Figure 4. EBSD data collected from an Inconel 600 Nickel superalloy at 500 pA beam current at 500 indexed points per second without NPAR (left) and with NPAR (right).

This new capability allows users to push EBSD system performance beyond traditional limits. For example, Figure 4 shows EBSD data collected from an Inconel 600 Nickel superalloy at 500 pA beam current at 500 indexed points per second. With standard indexing, these conditions give a 22% indexing rate, but when NPAR is applied, this improves to 96%. While a Nickel alloy could tolerate higher beam currents, Figure 5 shows results from a non-conductive ceramic sample collected with and without NPAR. The non-NPAR data was collected at 20 kV and 5 nA beam current, but charging effects are easily visible. For the NPAR data, the SEM conditions were changed to 12 kV and 1.5 nA beam current, while maintaining the same collection speed and increasing camera gain and EBSD pattern noise. The NPAR data clearly shows resolved grain structure without charging distortions.

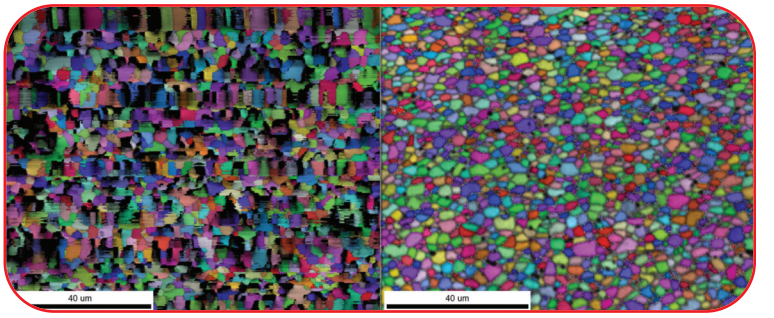

Figure 5. Results from a non-conductive ceramic sample collected without NPAR (left) and with NPAR (right).

NPAR provides a unique method of reducing EBSD pattern noise and facilitating faster data collection on both routine and difficult samples. This approach also works well on deformed material, provided the step size used is smaller than the spatial scale of deformation. In these cases, orientation precision is improved through better band detection via increased SNR. NPAR is now available as an optional software module with TEAM™ 4.3.

Click here to watch a brief video overview of NPAR.

Understanding Electron Backscatter Diffraction (EBSD) Background Corrections

Understanding how different background corrections work on EBSD is an important concept to learn. A raw EBSD pattern is low contrast and noisy, making Kikuchi patterns hard for both the human eye to see and the computer to index. A basic background correction will make the Kikuchi pattern stand out to both, but understanding the different kinds of background corrections in the TEAM™ software can increase the quality of both band detection through the Hough transform and indexing.

Figure 1. The current recipe, in this case a customer recipe made in the recipe builder, is shown on the image processing tab under the EBSD Camera settings. The modify button leads to the recipe builder.

The two basic backgrounds for EBSD are the Smart Background and the Scanning Electron Microscope (SEM) Area. The Smart Background changes the magnification of the microscope by a factor of four (1000x to 250x). The purpose of this is to average out the background of many grains and patterns compared to the magnification the user is working at. Issues with this background can arise. The biggest issue is grain size. If the grain size of the sample is relatively large compared with the magnification being used, the background will be skewed towards the orientation of the large grain. The other issue is that at low magnifications the automatic zoom out can cause the field of view to include either the sample holder or free air.

The SEM Area background can be useful in situations when the Smart Background may not be the best choice. The SEM Area background uses the current field of view as the background. This is useful when the user is trying to avoid a texture present in the sample or when the large grain size may require a background to be collected at lower magnifications.

Both of the above methods use EDAX's standard image processing recipe of static background correction and intensity histogram normalization. While these routines are sufficient for most samples, there are times when other methods of background correction may be more appropriate. This commonly occurs when running single grain samples. A static background in the case of single grain samples is the actual signal. Removing this as the background would give the user a reduced pattern or remove it completely. This kind of sample is perfect for Dynamic Background Subtraction, which runs a blur filter on the background, smoothing out the background instead of removing it entirely. The amount of blur can be controlled by increasing the number of passes.

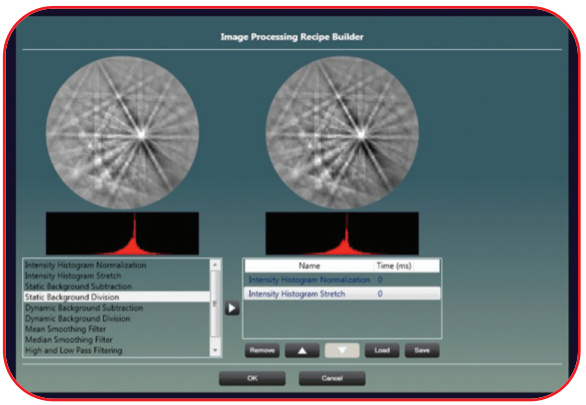

Figure 2. The built in recipe builder allows the user to customize any background to suit his or her needs.

The new Atom Probe Assist mode has added a background recipe, which incorporates the Dynamic Background Subtraction with a Median Smoothing Filter to help increase the contrast of processed patterns on Atom Probe tips.

While the above mentioned backgrounds are sufficient for most samples, the TEAM™ software includes a total of 10 different types of backgrounds that can be combined in nearly limitless ways and orders to help increase the pattern contrast and improve indexing success.

APPLICATION NOTE

Discriminating Glass Fragments Using Micro-XRF Spectrometry with Poly-Capillary Optics

Glass fragments are an ideal type of forensic trace evidence for a number of reasons including: their chemical composition does not vary over time, fragments are generally recovered in sizes large enough to be analyzed using a variety of analytical techniques, and the composition and properties of a single sheet of glass are relatively homogeneous (Trejos et al., 2013). Fragments from a broken source can be compared to fragments of questioned origin to determine whether the questioned fragments are consistent or inconsistent with the fragments from a known source.

Elemental analysis via Micro X-ray Fluorescence (Micro-XRF) is typically used in conjunction with measurement of other properties of glass fragments (i.e. refractive index, density and color) to compare questioned and known fragments. However, the topography and thickness variation of glass fragments leads to undesirable variations in the results. In lieu of actual compositional measurements, American Society for Testing and Materials (ASTM) E2926-13 describes a method of ratioing peak intensities of relevant elements in order to minimize the effects of fragment thickness and topography.

As advances have been made in Micro-XRF spectroscopy with regard to the exciting intensity and flux of X-ray focusing optics and larger area detectors, it has become difficult to completely correct for all spectral artifacts due to pulse pileup. However, by suppressing the intensity of the parent peaks through the use of primary beam filters, the resultant sum peaks can be eliminated and comparisons of trace elements across the entire spectral range become viable. The drawback in using a primary beam filter is that the detection limits for lower energy elements, such as Mg, are degraded. This can be overcome by collecting the low energy portion of the spectrum without a filter and the higher energy portion of the spectrum with a filter and then combining the results.

To demonstrate the methodology, fragments from soda bottle glasses purchased in 2011 and 2015 from two name brand sodas have been used. Soda bottle glass is very similar to common window glass, having large concentrations of SiO_2 and CaO, but with significantly less MgO. Bottle fragments were analyzed using an EDAX Orbis PC Micro-XRF Elemental Analyzer equipped with a Rh-target tube, a poly-capillary optic with a spot size < 30 µm (FWHM) at MoK_α and

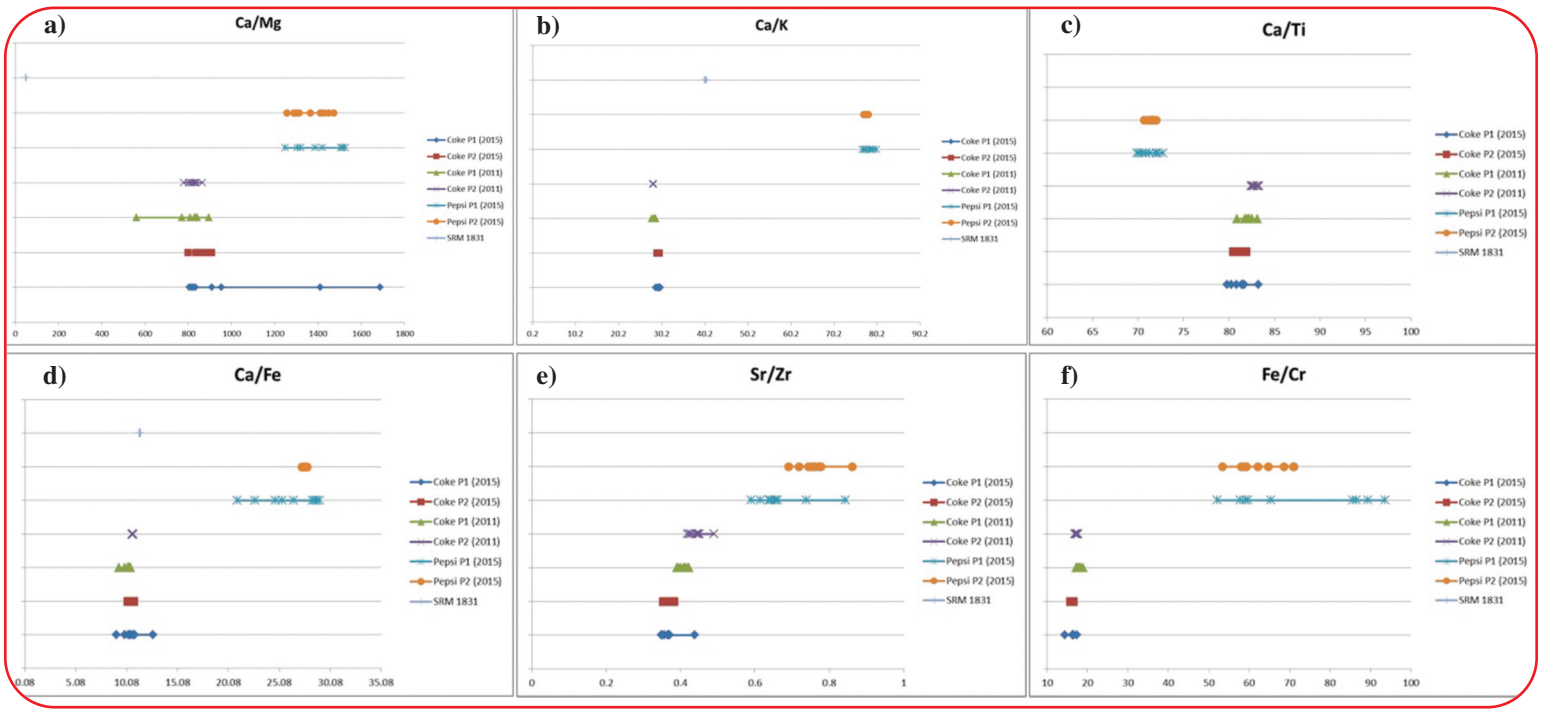

Figure 1. Peak ratios for all measurements plotted and compared as per section 10.7.3.1 of the ASTM. a) Ca(K_α) to Mg(K) ratio, b) Ca(K_α) to K(K_α) ratio, c) Ca(K_α) to Ti(K_α) ratio, d) Ca(K_α) to Fe(K_α) ratio, e) Sr(K_α) to Zr(K_α) ratio, and f) Fe(K_α) to Cr(K_α) ratio. Expansion of the horizontal axes has moved some data sets shown in the legend outside of the range of view.

(Continued from Page 4)

a silicon drift detector (SDD) operating at roughly 140 eV resolution. The glass samples were analyzed at nine different positions using no primary beam filter and then again with a filter to suppress the Si(K) and Ca(K) peaks.

In Figures 1a through 1f, peak ratios for all measurements have been plotted and compared as per section 10.7.3.1 of the ASTM. If the peak ratio data ranges of two fragments do not overlap, then they may be considered to be inconsistent. The elemental intensities for Ca, Ti, Cr, Fe, Sr and Zr were derived from the filtered spectra, while Mg intensity comes from the unfiltered spectra. Five of the six plots show clear differences between the Coke and Pepsi fragments. All six plots show consistency between the respective pairs of glass fragments originating from the same bottle (e.g. Coke 2011 fragment 1 to Coke 2011 fragment 2) as should be the case.

It is a much more challenging problem to distinguish between the Coke bottle fragments purchased from 2011 and 2015, four years apart. Whether this is practical with Micro-XRF depends on the consistency of the elemental composition of the bottles in the manufacturing process over time. If there are differences, the best place to look is probably the trace elements, such as Sr, Zr, K, Ti, Cr, Mn and Fe, where larger relative manufacturing variations may exist over time. An example of variations over time of trace elements in manufactured glass can be found in Trejos, et al. (J. Anal. At. Spectrom., 2013).

In Figure 2, peak ratio plots for Ca/Ti, Sr/Zr, Ca/K and Ca/Cr are shown with the horizontal scales expanded to highlight the Coke bottle fragment comparisons. The Sr/Zr ratio, which is good to check since these elements are impurities originating from the refractory materials of the furnace, does not show a significant difference. The Ca/K and Ca/Cr ratios show promise but there is some overlap on the edges of some of the ratio ranges. It may be useful to repeat the analysis to see if the scatter ranges can be reduced and then review if there is a significant difference. It should also be remembered that the Micro-XRF results are used in conjunction with other physical test measurements, which may prove to be useful in distinguishing the glass fragments from the same soda bottle brand.

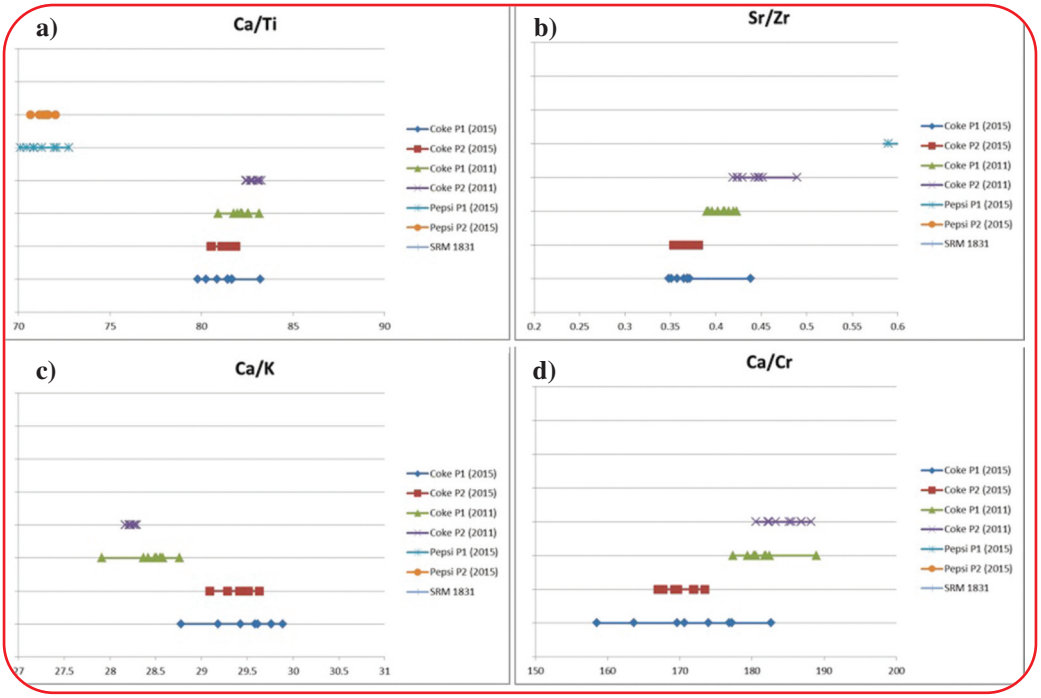

Figure 2. Peak ratio plots shown with the horizontal scales expanded to highlight the Coke bottle fragment comparisions. a) Ca(K_α) to Ti(K_α) ratio, b) Sr(K_α) to Zr(K_α) ratio, c) Ca(K_α) to K(K_α) ratio, and d) Ca(K_α) to Cr(K_α) ratio. Expansion of the horizontal axes has moved some data sets shown in the legend outside of the range of view.

EVENTS AND TRAINING

2016 Worldwide Events

January 31 - February 4		**February 22-27**	
Australian Conference on Microscopy and Microanalysis	Sydney, Australia	American Academy of Forensic Sciences	Las Vegas, NV
February 14-18		**February 22 - March 11**	
The Minerals, Metals & Materials Society (TMS)	Nashville, TN	Münsterkurse	Münster, Germany
February 18-20		**March 6-10**	
Texas Society of Microscopy (TSM)	Houston, TX	Pittcon	Atlanta, GA
February 19		**March 20-23**	
FIB SEM 2016	Laurel, MD	Arab Lab	Dubai, UAE

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2016 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	
January 26-28 February 23-25 April 11-13 May 31-June 2	Wiesbaden# Tilburg* Weiterstadt# Tilburg*
Microanalysis	
March 10-11	Tilburg*
TEAM™ EBSD	
March 7-9 April 13-15	Tilburg* Weiterstadt#
TEAM™ Pegasus (EDS & EBSD)	
March 7-11 April 11-15	Tilburg* Weiterstadt#
TEAM™ WDS	
March 15-17	Tilburg*
XRF	
April 5-7	Tilburg*

*Presented in English
#Presented in German

JAPAN

EDS Microanalysis	
Genesis	
February 18-19 April 7-8	Tokyo Osaka
OIM™ School	
Basic	
January 26-27	Tokyo
Entry	
May 10-11	Tokyo
Practice	
April 21-22	Tokyo

CHINA

EDS Microanalysis	
TEAM™ EDS	
March 8-10	Shanghai (ACES)
EBSD OIM™ Academy	
March 21-23	Shanghai (ACES)

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
February 2-4 February 22-23 May 16-20	Mahwah, NJ Draper, UT Mahwah, NJ
TEAM™ EBSD	
February 24-26	Draper, UT
XRF	
April 5-7	Mahwah, NJ
TEAM™ Pegasus (EDS & EBSD)	
February 22-26	Draper, UT

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(left to right): Fu Yantian and Lisa Liu.

Lisa Liu

Lisa joined EDAX on June 1, 2013. She is the sales assistant for the Beijing, China office. As part of her job, Lisa coordinates sales, service, marketing and commercial affairs in all of China. She is responsible for the entire sales and service process, including: contract review, order placing, payment collection, parts/accessories/equipment purchase processing and management, sales support and marketing activities.

Prior to EDAX, Lisa worked as an engineer change coordinator for global X-ray and sales operations at GE Healthcare in China from 2009-12. She earned a bachelor's degree from Beijing International Studies University in 2007.

Lisa lives in Beijing with her husband, Fu Yantian. In her spare time, Lisa enjoys traveling, outdoor activities, and watching movies with her family.

(left to right): Billy, Jen and Kayla Mehner.

Jennifer Mehner

Jen joined AMETEK on January 5, 2015 as the human resources generalist supporting both the EDAX and SPECTRO business units in Mahwah, NJ. Prior to this, she was a contractor with the company for nine months before taking over the role permanently. In her over 20 years of human resources experience, Jen has been involved in all facets of the department, including: recruiting and hiring, training and development, retention, payroll, benefits, compliance, and exit formalities. Her strengths are employee relations and conflict resolution.

Previously, Jen was a human resources specialist for Medco. She also served as a human resources manager in the retail sector with Target for 10 years. Jen has an associate's degree in human resources from Passaic County Community College.

Born and raised in New Jersey, Jen has two children, Billy (12) and Kayla (10). She enjoys skiing in the winter and going to the beach in the summer. Her children keep her very busy with their activities, including coaching her daughter's softball team. A huge New York Giants fan, Jen loves watching football on Sundays.

First European X-ray Fluorescence (XRF) Workshop was a Great Success

At the beginning of November we held our first XRF workshop in Europe, as it is an EDAX tradition to have a workshop in the first week of November in Germany. Over the last 10 years, we have done a lot of different workshops and user meetings about Electron Backscatter Diffraction (EBSD), Energy Dispersive Spectroscopy (EDS), particle analysis and Wavelength Dispersive Spectrometry (WDS). This year was our first XRF session and almost 50 participants registered for the event.

Our goal was to demonstrate to customers a wide range of XRF applications, so we installed our Orbis system and also the SMX-BEN from our new XLNCE XRF series in our demo lab in Wiesbaden. These are both standalone systems. With the help of IfG in Berlin we also installed their iMOXS μ-XRF system on our Scanning Electron Microscope (SEM) in the lab and we organized the workshop jointly.

The first day of the event was filled with presentations about different applications with all three systems. We had some very interesting talks. EPFL Lausanne in Switzerland explained how the Orbis system supports them in their daily work in the crystal growth laboratory; Vito NV from Belgium showed that it is possible to use the Orbis for a wide variety of analysis, for example air analysis, water tests, and research for Alzheimer's disease. Customers from TU Ilmenau, Germany and KU Leuven, Belgium talked about how useful XRF is in their daily work and Continental Automotive GmbH, Germany demonstrated that the Orbis can also be used for particle analysis and parts cleanness analysis.

After a lunch break, Dr. Matthias Procop and Sabrina Günther from IfG in Berlin gave more details on the combination of EDS and μ-XRF on the SEM. This was complemented by application examples based on the combination of these two systems on layer structures or corrosion problems presented by AUDI, Germany.

Figure 1. The participants in the 2015 European XRF Workshop.

Figure 2. Workshop presenters (left to right) Oliver Senftleben (AUDI), Gerd Teichert (TU Ilmenau), Mathias Procop and Sabrina Günther (IfG), Andreas Kümmel (Continental Automotive GmbH), Bruce Scruggs (EDAX), Harry Verhulst (EDAX), Christine Vanhoof (VITO NV, Belgium), Andrew Lee (EDAX), Arnaud Magrez (EPFL Switzerland), Tom Van der Donc and Pieter L'Hoëst, (KU Leuven, Belgium), Michaela Schleifer (EDAX) and Christiane Lettmann (EDAX).

The first day finished with an introduction to our new XLNCE XRF series and a lot of application examples using our SMX-BEN tabletop system.

On the following day, participants had a chance to watch demonstrations of all three systems: Orbis, SMX-BEN and iMOXS. Our application specialists explained the systems, showed them the software and did some measurements. A lot of interesting discussions arose during these demos and at the end the participants asked about our plans for a workshop in 2016 and expressed their interest in joining again.

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