EDAX FOCUS

Inside This Issue

Vol.7 No.4

TEAM™ EDS With Smart Features Will Change the Way You Do Analysis Forever!

EDAX is once again leading the way in EDS technology with TEAM™ EDS. This advanced analysis system was designed with the latest software platform optimizing the system for multicore and multiprocessor environments. Along with the implementation of Smart Features that provide analytical intelligence, the user can be assured of easily obtaining quality and reliable results easily and quickly. The core components of the system are based on the Smart Features that allow for optimal set-up of analysis parameters to be automatically selected for the most easily obtained results on the market today.

Smart Diagnostics

The modern interface allows for an open layout and maximizes the display area for what really matters, your results. This layout and the interaction that the system allows is unique in the industry. The system allows for user specific profiles to be created upon login that are particular to that user's preference. During the login process, the Environment Panel (EP) is monitoring and reporting on the operating conditions of the data collection environment and the SEM. These Smart Diagnostics monitor the column, stage, detector, and magnification of the system as well as provide operators with vital information at a glance. The Smart Diagnostics will let you know if a condition has changed by sending an alert message to help you diagnosis if there is a problem with your collection parameters. The tool also provides guidance during the phase mapping process to ensure you receive the best data in the time you have available. For those users who like more control over the system, the EP also provides access to the Tier II panel where all of the advanced setting and controls are for those users that like to determine or adjust parameters for analysis.

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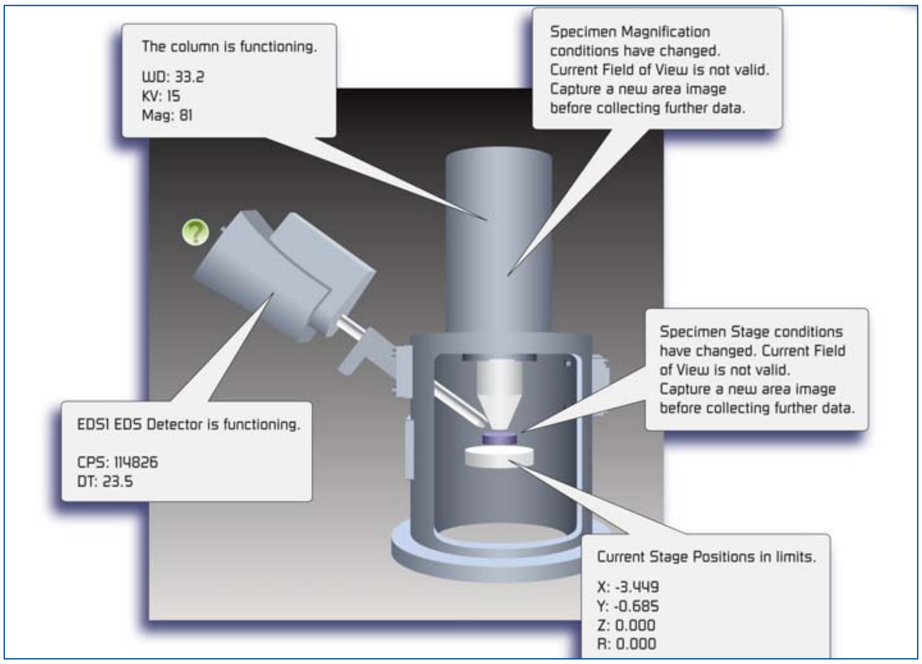

Figure 1 - Environment Panel (EP)

TEAM™ EDS With Smart Features Will Change the Way You Do Analysis Forever! (Cont'd. from Pg. 1)

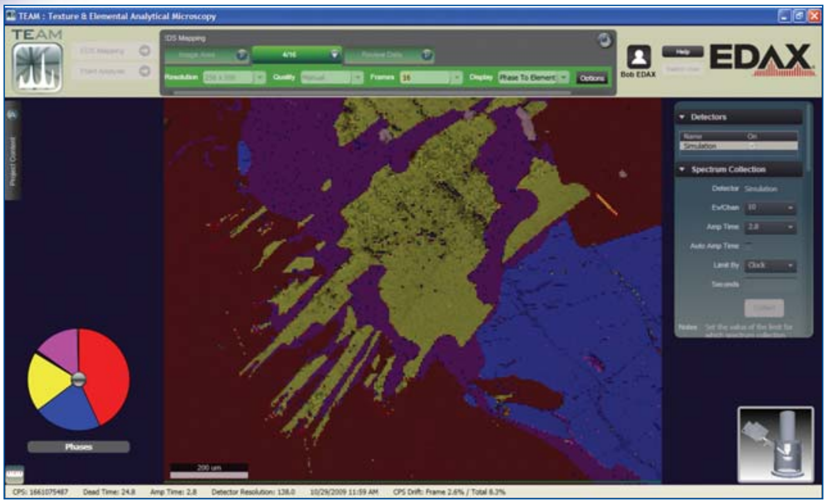

Figure 2 - Phase to Element - Phase maps with supporting elemental information.

Interactive Smart Mapping Like You Have Never Done Before!

TEAM™ Smart Phase Mapping is at the core of this system. The user can easily choose the quality of their data that is required for their map. This will help the system determine the length of time needed to collect to match their requirements. Start the data collection and the system will automatically collect a smart preview spectrum. It will set up the initial table of elements being mapped and will update it during the mapping process. TEAM™ will begin to make a provisional analysis of the EDS data being collected at each point and assign a phase according to the combination of elements being measured. This chemistry is displayed in a unique interactive pie chart. During your collection you can choose to display your information as Phase to Element, Element to Phase, and Counts per Second. Whatever method you feel gives you the best information for your problem.

Smart Acquisition with Point Analysis Tools that Enable Fast, Easy Quantification with the Revolutionary EXpert ID

Collecting the best image can sometimes be tedious work but with TEAM's auto optimized image enhancement it prevents the need to "fine tune" the appearance. Auto Signal to Noise selects best frame integration, reduces noisy images, gives best appearance, enhances data and reduces noise. The point analysis tool can be used to quantify individual measurements during collection from various sites over the specimen's area. Individual points, multiple points, selected areas, and linescans can all be performed. TEAM™ EDS utilizes EXpert ID, the next generation of element identification and quantification. One step peak identification, utilizing a combination of known peak locations and rules based theory, provides the user with the confidence in their results. The new Smart Quant routine implements a quantitative routine for samples tilted up to 70 degrees. EDAX's calculated Bremsstrahlung is the newest knowledge in sciences; it is based on the absorption edges of the elements; as a minimum it has one adjustment range but the user can add as many as needed.

No matter what your analysis needs are, you will need to preserve the correct shape of the features. Smart Drift Correction requires no input from the users to determine the parameters for analysis and can dynamically correct for drift as it changes during an analysis.

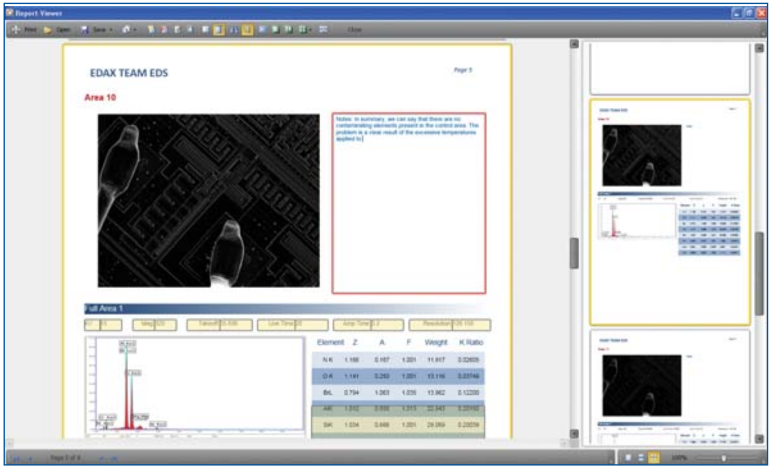

Figure 3 - Smart Data Review.

Smart Data Review

Smart Data Review completes the system by providing an innovative layout and project tree that allow for quick review and reporting of images, maps, and spectra. Data can be loaded into a report viewer by pressing a single button in TEAM™. Text can be added and it can be saved in over 20 different types of file formats to meet every user's requirements.

Characterizing Fracture Surfaces with Dual EDS Detectors

Introduction

Performing EDS analysis on a specimen with rough surface such as a fracture surface can be difficult due to shadowing effects as shown in the schematic in Figure 1.

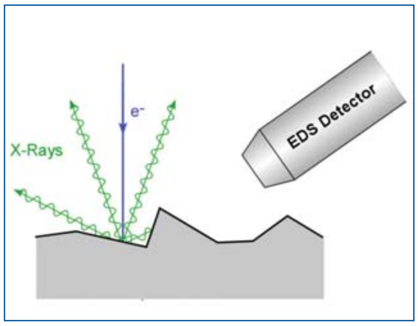

Figure 1 – (a) Schematic of X-ray scattering at a single point where a “hill” prevents X-rays from reaching the EDS detector.

An additional complexity results from the surfaces significantly inclined towards the detector and from other surfaces sloping away from the detector.

An example is shown in Figure 2 for a fracture surface in a sandstone. The EDS Si map clearly shows shadowing. While the shadowing is evident, it is difficult to determine whether a dark area in the map is dark due to low Si content or is dark because of shadowing.

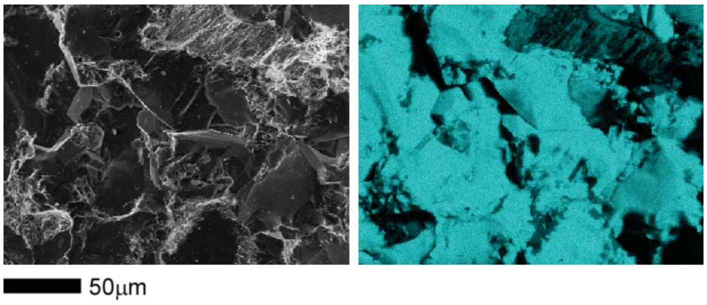

Figure 2 – Secondary electron image (left) and a silicon EDS map (right) of a sandstone fracture surface.

The shadowing effects can be mitigated in two different ways: (1) collect a set of maps from an area, rotate the sample 180° and then collect the same set of maps over the same area, rotate the results and merge them with the results from the original scan. (2) Use two well-balanced detectors with different azimuthal angle simultaneously and then merge the two sets of maps. A problem with the first method is that it is time consuming and often the results do not successfully merge.

Dual Detectors

An example of two EDS detectors mounted on an SEM is shown in Figure 3. Using dual EDS detectors enables the shadowing effects to be mitigated. This approach is not only much more efficient as the area needs to be mapped only once, but also reduces any errors associated with registering the two sets of maps using a single detector. However, simply summing the two datasets together is not adequate to properly capture the missing data. A better approach would be to combine the data by selecting only the maximum signal at each pixel. Most often a normalizing procedure is required to eliminate the effects of topography because the count rates might be below average when viewed by one detector while the other detector is completely shadowed. A normalization procedure that is commonly available is to analyze the merged dataset with a k-ratio or ZAF procedure.

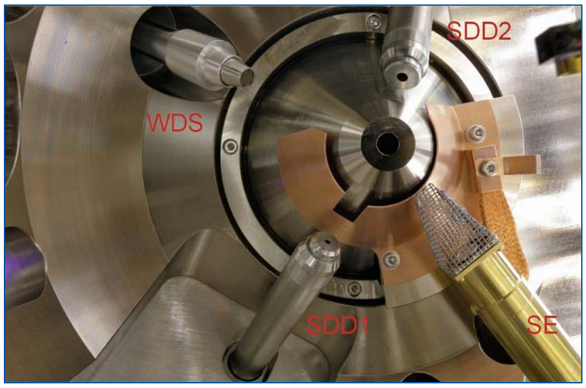

Figure 3 – Interior view from below of an SEM (JEOL 700F) with dual silicon drift detectors.

Example

Figure 4 shows a backscattered electron image (BEI) from a fracture in an aluminum alloy. The sample has an extremely rough surface. Several different phases seem to be evident in the sample as a dendritic structure can also be observed.

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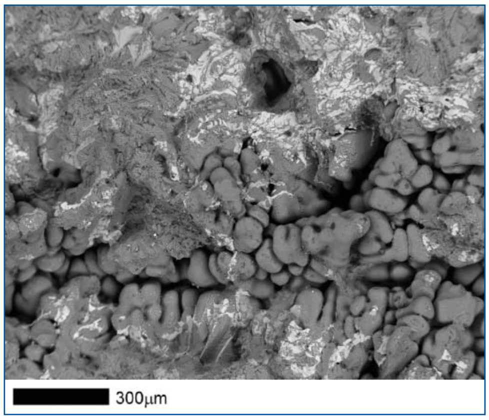


Figure 4 – Backscattered Electron Image (BEI) of a fracture surface in an aluminum alloy sample.

Characterizing Fracture Surfaces with Dual EDS Detectors

(Cont'd. from Pg. 3)

Figure 5 shows EDS maps from the two different detectors. In this case, the full spectrum constitutes the region of interest (ROI). Once again, the shadowing is quite obvious in these maps.

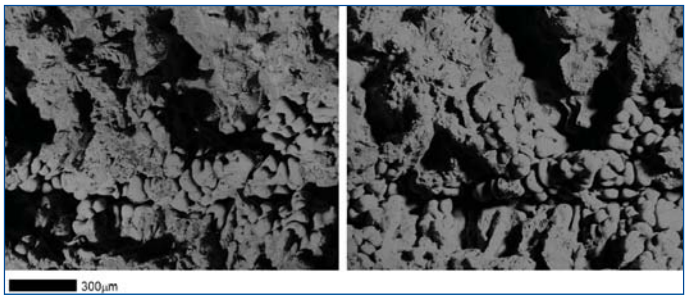


Figure 5 – EDS maps of fracture surface obtained using two different detectors.

Figure 6 shows an EDS map constructed by simply summing the data from both detectors. The white areas represent areas that provided signal to both detectors, the black areas did not provide signal to either of the two detectors and the gray areas provided signal to only one of the two detectors. As the ROI is for the entire spectrum, the contrast in the map is an artifact arising from the signal distribution between the two detectors and is not chemical.

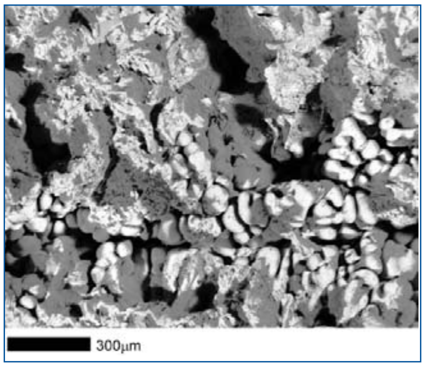


Figure 6 – Sum of the EDS maps of the fracture surface obtained using two different detectors.

The sum map for the full spectrum ROI can be used in conjunction with EDS maps for real elemental ROI's. However, in these elemental maps instead of summing the ROI data at each pixel from both detectors together, the maximum counts between the two ROI data sets are used. These maps are overlaid on an inverted and binarized sum map for the full spectrum ROI. In these combined maps, points in white are areas that cannot be detected using either of the two detectors. Points in black indicate areas where the element is not present. Figure 7 shows an example for two elements: copper and silicon.

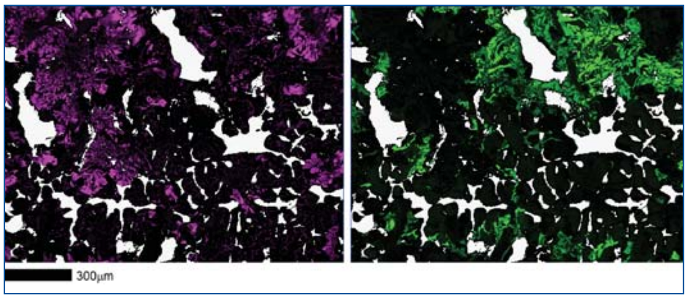


Figure 7 – Maximum signal EDS maps for Cu and Si from the fracture surfaces.

However, this map still has effects of topography in it. The mid-toned areas may be due to topographic effects or points with less content of the specified element. In order to resolve this ambiguity, the maximum signal data should be normalized and a ZAF correction has been used. Figure 8 shows the results of the ZAF normalization on a map where all of the elements are combined together in a single map. The areas for which no signal could be obtained using either detector is now shown in cyan.

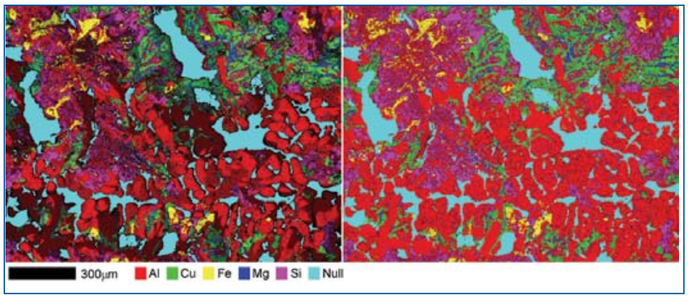


Figure 8 – Maximum signal EDS maps are shown in overlay mode both before (left) and after (right) ZAF normalization.

Conclusions

Dual detectors are very useful for resolving the shadowing and topographic effects associated with rough surfaces. This can help in a variety of application areas such as characterizing fracture surfaces, wear surfaces or as-machined surfaces. In order to have the map with the clearest impression of the chemistry without the ambiguity of sample topography, the maps should be merged using a maximum signal function and the dataset should then be normalized.

Case Study: Phase Identification Using a Trident System

Identifying unknown phases in a specimen can be a challenging task. In some cases an EDS analysis alone is sufficient, but often the combination of EDS with crystallographic information obtained from EBSD is necessary. Occasionally, overlapping peaks cannot be resolved with EDS therefore; the higher resolution of a WDS detector is required to separate the peaks. This article is an example of a Trident system, the combination of EDS, EBSD, and WDS, to identify an unknown mineral in a sulfide rock sample.

For phase identification, first the chemical composition needs to be determined. The EDS spectrum in (Figure 1) indicates that the main elements present are Cu, S, and Bi. The large peak at 2.4 keV shows an overlap of the lines of both S and Bi. In order to determine if other elements are present in this overlap region, the Halographic Peak Deconvolution (HPD) function in Genesis was used. HPD generates a simulated spectrum based on the identified elements and the actual EDS detector resolution. This allows the operator to verify if any elements are overlooked.

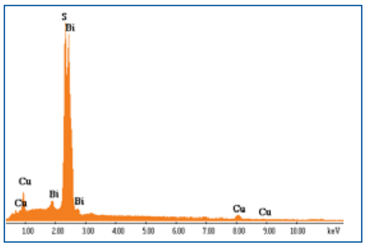


Figure 1 – EDS spectrum of the unknown phase. Spectrum collected at 15 kV for 100 live seconds on an Apollo 10 Silicon Drift Detector.

In (Figure 2a) the HPD overlay does not match the shape of the complex peak pattern which indicates that the element selection is incorrect. In (Figure 2b), Pb is added to the element list and now the HPD closely matches the EDS spectrum.

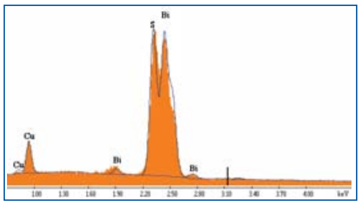


Figure 2a – Detail of the EDS spectrum with HPD overlay of S and Bi.

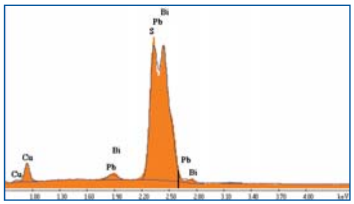


Figure 2b – Same energy region, now showing HPD after adding Pb.

When there is such a strong peak overlap of multiple elements in the EDS spectrum, the measurement error in the quantification results may be significant. Therefore, to confirm the presence of these elements and allow accurate quantification, a WDS energy scan was collected over this overlapping region in EDS. The higher resolution of the WDS detector allows separation of the overlapping S Ka, Pb Ma, and Bi Ma, lines (Figure 3).

In nature a sequence of Pb-Cu-Bi-S minerals occurs:

Aikinite	Cu Pb Bi S_3
Krupkaite	Cu Pb Bi_3 S_6
Hammarite	Cu_2 Pb_2 Bi_4 S_9
Lindstromite	Cu_3 Pb_3 Bi_7 S_{15}

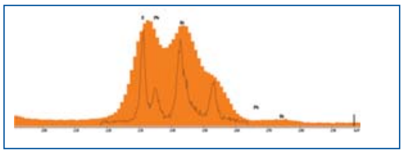


Figure 3 – WDS energy scan from 2.18 keV to 2.64 keV (LEXS) superimposed on EDS Spectrum.

The composition measured using standardless quantification with EDS corresponds to $Cu_{1.5}Pb_{2.2}Bi_{3.1}S_6$. WDS spectra were run on a LEXS in reduced scan mode for 10s/channel and using 10nA beam current. For standards in WDS quant, PbS (galena), pure Bismuth and pure Copper were used. The composition measured from WDS corresponds to $Cu_{1.08}Pb_{1.12}Bi_{3.1}S_6$.

The differences in composition as determined by EDS and WDS do not allow identification of the phase unequivocally. EBSD is used to match the diffraction pattern against the crystal structures of all candidate phases. The EBSD pattern and indexing result for the best solution is given in (Figure 4).

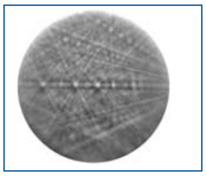


Figure 4 – EBSD pattern and indexing solution. The crystal structure matched krupkaite, $CuPbBi_3S_6$. The crystal structure of all candidate phases was taken from the AMCS mineral database.

WDS quantification results are consistent with the EBSD data, identifying krupkaite as the unknown phase. EDS is less accurate due to the severe peak overlap. After successful identification of the phase, the structure file can be used in OIM™ (Orientation Imaging Microscopy) EBSD mapping to obtain the distribution and orientation of these grains in the rock sample (Figure 5).

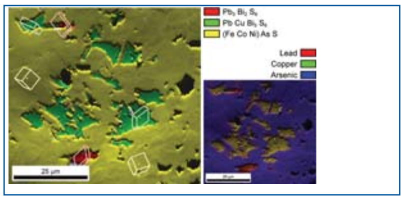


Figure 5 – EBSD phase map (left) and EDS blended color map illustrating the krupkaite (green) distribution in a (NiCoFe)AsS matrix.

The combination of EDS, WDS, and EBSD allows identification and analysis of phases, which may be difficult to analyze with any of the individual techniques alone.

TRAINING AND EVENTS

World-Wide Events

January 20-22	2010 EMST	Suratthani, Koh Samui, Thailand
February 14-18	TMS (Minerals, Metals & Materials Society Meeting)	Seattle, WA
February 22-26	2010 ICONN (International Conference on Nanoscience and Nanotechnology)	Sydney, Australia
February 22-27	AAFS (American Academy of Forensic Sciences)	Seattle, WA
February 28 - March 5	Pittcon	Orlando, FL
March 3-5	2010 EMSI (Electron Microscopy Society of India)	Mumbai, India
March 22-26	6th Asia Mining Congress 2010	Singapore
March 23-26	Analytica	Munich, Germany
March 29-30	UK EBSD Meeting	Derby, UK

***Please see our website, www.edax.com for a complete list of our tradeshows

World-Wide 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, we organize a number of Operator Courses at the EDAX facilities in North America; Tilburg, NL; Wiesbaden, Germany; and Japan.

Europe

Tilburg = (T) (in English)
Wiesbaden = (W) (in German unless stated otherwise):

EDS Microanalysis:

- February 2-4 (T)
- March 11-12 (T)
- March 23-25 (T)
- March 29-31 (W)
- June 1-3 (T)
- June 17-18 (T)
- September 14-16 (W)
- September 16-17 (T)
- September 28-30 (T)
- November 11-12 (T)
- November 23-25 (T)

EBSD:

- March 8-10 (T)
- June 14-16 (T)
- September 13-15 (T)

WDS (LEX):

- April 13-15 (T)
- October 12-14 (T)

WDS (TEXS):

- May 4-6 (W) English

Pegasus:

- February 1-5 (W)
- May 17-21 (W)
- October 18-22 (W)

Japan

Microanalysis Courses:

◆ February 3-5	Tokyo
◆ April 7-9	Osaka
◆ June 9-11	Tokyo
◆ July 7-9	Osaka
◆ October 6-8	Tokyo
◆ November 10-12	Osaka

For more information on our training classes, please visit our website at:
www.edax.com/service/user.cfm

North America

EDS Microanalysis:

◆ March 16-18	Mahwah, NJ
◆ April 26-30	Mahwah, NJ
◆ June 22-24	Mahwah, NJ
◆ July 13-15	Draper, UT
◆ September 14-16	Mahwah, NJ
◆ October 18-22	Mahwah, NJ
◆ November 9-11	Mahwah, NJ

EBSD OIM™ Academy:

◆ April 13-15	Mahwah, NJ
◆ August 24-26	Mahwah, NJ
◆ October 5-7	Draper, UT

Pegasus:

◆ February 1-5	Draper, UT

EDS Particle:

◆ May 11-13	Mahwah, NJ

Micro-XRF: (Orbis only)

◆ March 30 - April 1	Mahwah, NJ
◆ September 28-30	Mahwah, NJ

EMPLOYEE SPOTLIGHT

Tina Wolodkowicz joined EDAX in September 1997. She is a Senior Sales & Marketing Coordinator, based out of the EDAX corporate headquarters in Mahwah, New Jersey. In her role, Tina supports the Americas Sales Team. Her main responsibilities include quotations, bids in conjunction with government paperwork, keeping the customer database current, as well as coordinating a number of tradeshows each year. Tina also has frequent telephone contact with customers and purchasing agents, and works closely with our Logistics and Service departments.

Tina was involved in similar work prior to joining EDAX. Her job focus was geared toward tradeshows and marketing as well. Tina found this type of work interesting and viewed it as a growing experience. After five years she was ready to take on additional responsibilities and that is when she joined EDAX.

During her years at EDAX, Tina has received numerous promotions, earned a BS degree in Business from Dominican College, located in Orangeburg, NY, and has gotten married.

Tina is a native New Yorker from the suburbs. She enjoys writing poetry, photography, doing crafts, and gardening. Tina and her husband Rob hike and exercise together. They enjoy going to the movies and taking vacations to the Virgin Islands. Family and friends are a very important part of their lives and they are hoping to start a family of their own sometime in the near future.

Björn Bergsten is the Senior Regional Manager for EDAX Asia, located in Singapore. He is responsible for the sales and service organizations in Japan and China, and manages a distributor network in the ASEAN region. Born and raised in Sweden, Björn received his MSc degree at Linköping Technical University.

Björn began his career in the 1970’s as a Microprobes (EPMAs) Service Engineer with ARL (Applied Research Laboratories). He joined Philips Sweden in 1981 as a Sales Engineer for Philips Electron Microscope and EDAX in the Nordic countries.

In 1988, Björn moved to the Netherlands to assume the role of European Sales Manager for EDAX. His early focus was the start-up of the Tilburg office and the creation of a distributor network in Europe. In 1995, Björn moved to the USA to take on the role of EDAX Product Manager at the Mahwah, NJ headquarters. In this role, he led the successful launch of the Phoenix EDS Microanalysis system. Björn also played a role in the collaboration with TexSEM Laboratories (TSL) that resulted in the EDAX acquisition of TSL in 1999 and the launch of the world’s first truly integrated EDS/EBSD system, the Pegasus. In 1999 Björn was promoted to VP Sales & Marketing, responsible for EDAX products worldwide. In 2008 Björn moved to Singapore where he was appointed to Senior Regional Manager for Asia.

Björn has been involved with the sale of EDAX products for almost 30 years. He has travelled extensively all over the world to meet customers and EDAX sales partners. He enjoys interacting with worldwide customers and listening to their requirements to help fulfill their needs.

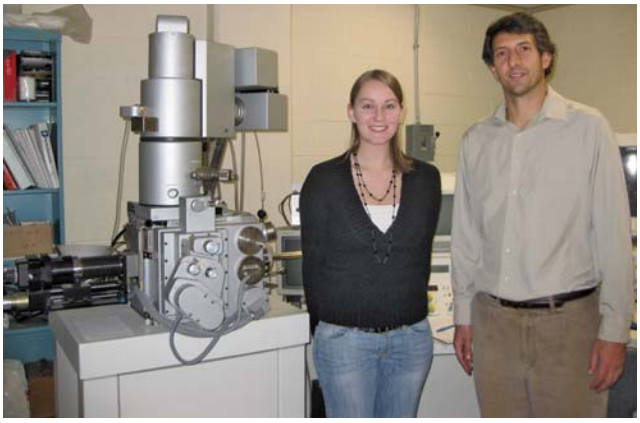
In his spare time, Björn enjoys relaxing in the sun and spending time with his sons Karl (28 years) and Erik (22 years).

Michigan State University

The College of Engineering at Michigan State University (MSU) provides an unprecedented learning environment to its students through innovative teaching, research, and outreach. The Chemical Engineering and Materials Science Department, formed by the merging of two established programs in 2001, offers unique opportunities in the areas of microelectronics, biomaterials, environmentally friendly materials, and nanotechnology. The department consists of 29 faculty members, eight staff members, 400 undergraduates, and 115 graduate students. At Michigan State, students enjoy access to outstanding laboratories for biochemical engineering, composite materials processing, and characterization of metals, ceramics, and polymers. Included in the suite of analytical techniques available to students in the College of Engineering is electron backscatter diffraction.

MSU acquired its first EDAX EBSD system in 2002, and has contributed to the field of EBSD in several novel research areas. In a project led by Professor Carl Boehlert, EBSD is employed in the evaluation of alloys for the purposes of grain boundary engineering, creep testing, and evaluating special boundaries. In an SEM equipped with a tensile stage, in-situ creep testing is performed to determine cracking mechanisms. The EBSD is used to study and characterize crack growth in the in-service condition. In 2008, graduate student Wei Chen presented findings on fatigue crack growth in titanium boride alloys at the Microbeam Analysis Society Topical Conference on Electron Backscatter Diffraction, and in 2009, graduate student Sara Longanback was awarded a Microbeam Analysis Society Distinguished Scholar Award for her study of in-situ tensile creep deformation of cobalt-based superalloy.

Graduate students Leung Wang and Yiyi Yang evaluate deformation at grain boundaries in commercially pure titanium. Under the guidance of Professors Martin Crimp and Thomas Bieler, and in collaboration with the Max Planck Institut für Eisenforschung, Wang and Yang identify active deformation by observing crystal deformation sliplines and twin boundaries in the scanning electron microscope images. The EBSD is used to evaluate how crystal rotations develop as materials are deformed.



Professor Carl Boehlert and graduate student Sara Longanbach in the Michigan State University laboratory, equipped with EDAX EBSD.

EBSD is also employed at MSU in the analysis of fine-grain niobium sheet for the evaluation of superconducting radio frequency (SRF) cavities. Professor Bieler, along with graduate student Derek Baars, examines texture in the materials and determines relationships between the deformed, recovered, and recrystallized microstructures for the purpose of improving production of niobium sheets.

Through exchange of feedback and ideas, Dr. Boehlert, Dr. Bieler, and Dr. Crimp have contributed to the development of EBSD methodology, and have aided further development of the OIM™ suite of products. The work conducted at Michigan State University is greatly respected by the EBSD community, and the list of future research projects is ever-expanding.

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AMETEK®
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EDAX®
advanced microanalysis solutions

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