EDAX FOCUS

EDAX Introduces OIM™ 6.0 Software

Inside This Issue

Vol.8 No.2

EDAX continues to lead the industry with its recent release of OIM™ 6.0 Software. Orientation Imaging Microscopy (OIM) is a technique used to obtain crystallographic orientation, grain boundary character, and phase distribution information from single and polyphase crystalline materials through the collection of SEM based Electron Backscatter Diffraction (EBSD) patterns. The OIM™ 6.0 product family consists of the OIM™ Data Collection, OIM™ Analysis and Delphi applications. OIM™ Data Collection is used, in conjunction with an EBSD detector and SEM, to automatically capture and analyze EBSD patterns to compile OIM™ datasets. OIM™ Analysis is the standalone application used for the creation of maps, plots, and charts detailing the sampled microstructure as well as highlighting and partitioning of data for advanced analysis.

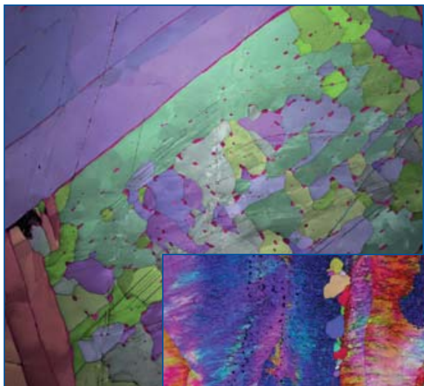


Figure 1 - A combined image quality and orientation map from the Gibeon meteorite.

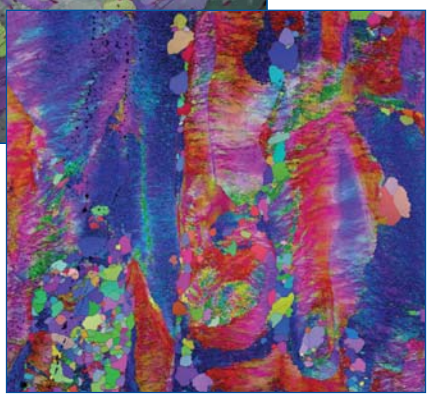
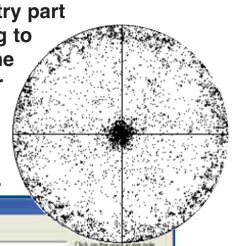
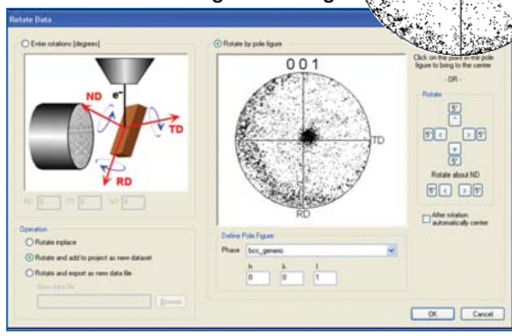


Figure 2 - A combined image quality and orientation map from a partially recrystallized steel.

OIM™ 6.0 Software is the first microanalysis package to be written for 64-bit processor and Microsoft® Windows 7 compatibility. OIM™ Analysis 64-bit allows for very large scan files to be handled on systems with a 64-bit processor and operating system. Some recent examples of analysis include datasets that are an order of magnitude larger than current systems (>40 million data points).

Figure 3. To center the cluster near the center of the pole figure, the user would have to estimate the correct rotations in the manual entry part of the dialog and close the dialog to apply the rotations. Now, a click at the cluster rotates the data so the cluster is at the center of the pole figure. This is reflected in the pole figure in real time and the rotations applied to the data set after "Oking" the dialog.





OIM™ 6.0 Software is Easier to Use

- Expanded QuickGen and TemplateFunctionality: Easier initial access to the most commonly used functions in OIM™ Analysis and "one-button" complete analysis using template files with a comprehensive template library
- Full Image Area Mapping: Map full image area with scan resolution selected automatically by the number of points desired in the map, resulting in quicker time-to-mapping
- Easier Data Rotations: Fast data rotation based on manual selection using pole figures for easy alignment of data (see Figure 3)
- Interactive page colors make quick measurements easier to understand and utilize
- The system level settings are password protected, though the improved user interface allows users access to options needed for data collection without changing critical system settings (see Figure 4) (Cont'd on Pg. 2)

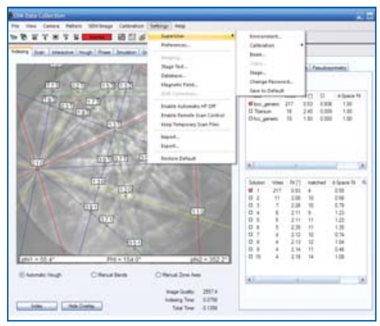


Figure 4 - Improved user interface.

EDAX Introduces OIM™ 6.0 Software (Cont'd. from Pg. 1)

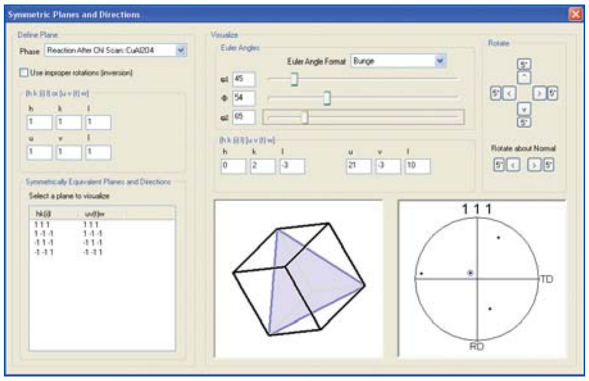


Figure 5. Improved access to information about crystal planes with advanced visualization tools for understanding the underlying crystallography used by OIM.

Improved Access to the Information You Need Most

- Improved advanced visualization tools to more easily understand the underlying crystallography such as crystal planes and misorientation axes
- Interactive status bar displays useful information at each datapoint
- Data Processing Log automatically records post processing performed on datasets

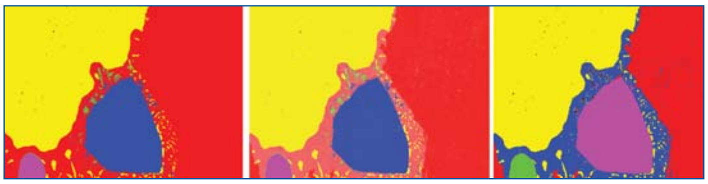


Figure 6. Enhanced functionality with Chi-Scan PCA Component Variation. 70% tolerance map is shown on left - notice the red phase. When the component variation map is shown (middle), the red phase divides into two shades of red. When the tolerance is increased to 75% (right), the initial red phase is divided into two unique phases.

The Enhanced Functionality Provides the User with:

- Improved Chi-Scan technology, EDAX's patented combined EBSD-EDS tool for multiphase analysis: Component deviation mapping, EDS spatial shifting, and EDS kernel averaging have all been implemented to improve phase differentiation, especially for nanoscale measurements
- Enhanced Batch Processor: More comprehensive automated analysis of multiple datasets using a range of template types. Useful for both routine analysis and 3D or in-situ measurements for faster and more repeatable and consistent data analysis
- Crystallographic Plane Matching: Identify and visualize specified planes aligned across a boundary and aligned with 2D grain boundary trace. Useful for investigating phase transformations and orientation relationships
- Fast Fourier Transform (FFT) Image Processing: Improved EBSD pattern quality with a FFT-based image processing routine that can be combined with other algorithms and saved as a recipe for later use
- Non-Cubic CSL Boundary Analysis: Extends CSL boundary analysis to hexagonal, trigonal, and tetragonal unit cells for materials such as magnesium, zirconium, and alumina

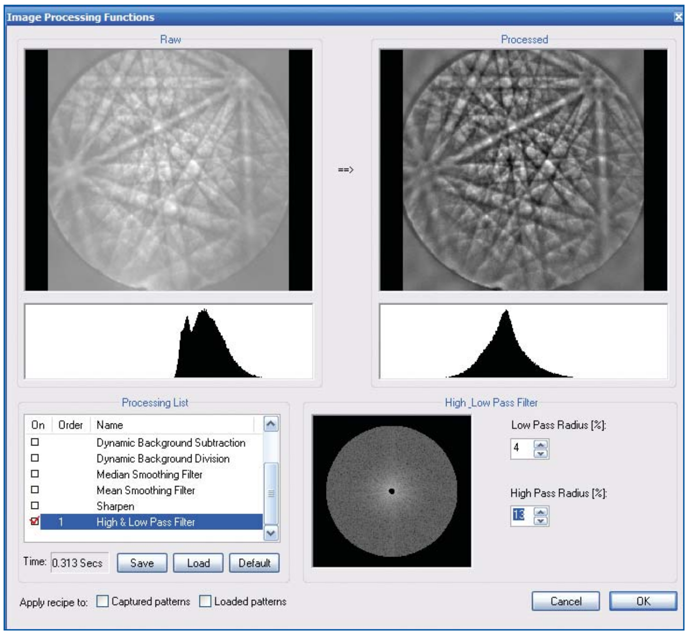


Figure 7. Enhanced functionality with additional image processing routine based on Fast Fourier Transform (FFT). Can be combined with other processing routines and saved as a recipe for later use.

With the addition of these new features OIM™ 6.0 Software continues to lead the way with the most advanced EBSD tools in the industry. OIM™ 6.0 Software still implements its unique triplet indexing algorithms and patented Confidence Index for superior EBSD pattern analysis and subsequent data quality.

TIPS & TRICKS

Scanning Spatial Resolution Limits

The resolution limit for microanalysis is commonly considered to be the interaction volume of the technique. While true, the resolution limit has other constraints when mapping is considered. Often, the barrier to resolution while mapping is charging.

Charging is a concern for electron microscopy, but especially with microanalysis. When compared to imaging with electron microscopes, microanalysis commonly has longer dwell times, higher voltages, and higher currents. This can mean damage to the sample, drifting of the beam placement and, in some cases, even alterations of data.

Charging is the accumulation of electrons within a localized region on the sample surface. With increases in current, more electrons are put on the surface. The charging is not due to nominal current or current density but rather to charge flux, the amount of charge introduced to a surface over an interval of time. Therefore, higher currents can be used without noticeable charging effects provided the flux remains low.

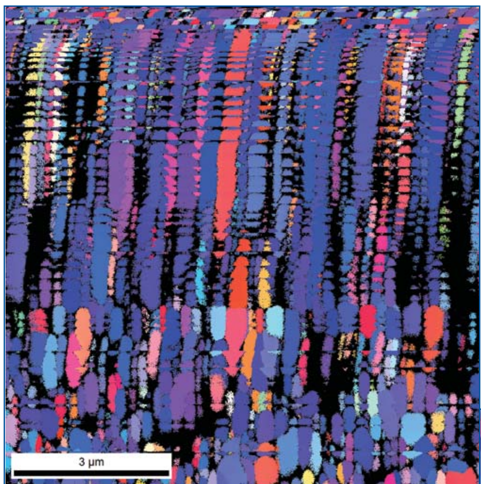


Figure 1.

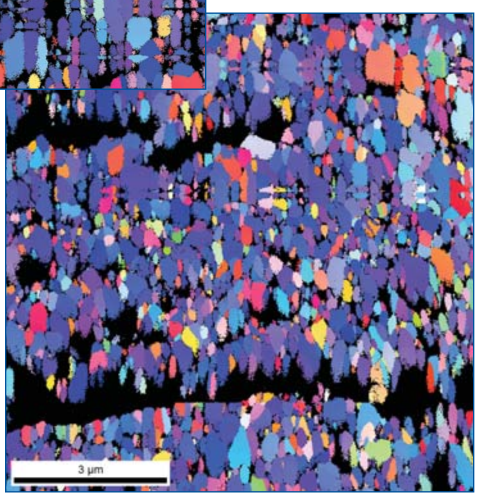
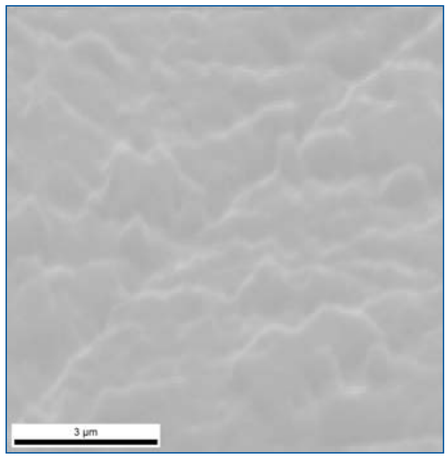


Figure 2.

To reduce the charge flux on the surface, the user can leave the current high while reducing the dwell time. For OIM, this can be done by increasing the camera binning or the gain in the camera console. Increasing the binning will decrease the resolution however it will also increase the active area of the CCD sensor contributing to each pixel.

The surface charge is not constrained to the area under the beam itself, the charge density will disperse outward as well. To avoid charging effects, the sampling step size of the scan must be larger than the “charged” region on the surface. Reducing the flux will shrink the charged region, but even with higher flux, the charging effects can be skirted by using a larger step size as shown in Figure 2.

Figure 3.



The example shown is a nickel thin film on copper, mounted with silver paint. The sample is very conductive, but even under these conditions charging effects are visible. Figures 1 and 2 were both collected at the same sample point using the same camera and microscope parameters. Figure 1 used a 10nm step size while Figure 2 used a 15nm step size. Surface roughness exacerbated the charging problem (visible in Figure 3). While all charging artifacts are not removed in Figure 2, the reduction is quite dramatic considering all other parameters were held the same. Increasing the step size by 50% reduced the total flux on the sample surface by about half and nearly eliminated sampling of previously charged regions.

Faster scanning of the surface will aid in mitigating charging effects; not just with EBSD but nearly all electron microanalysis techniques.

EDS NEWS

Optimization of Collection Parameters for Site Specific Feature Microanalysis

Elemental analysis often requires samples that are ideally flat, polished, and infinitely thick to the electron beam in order to ensure high analytical accuracy. However, it is common practice for analysts to work with sample types that are not ideal. Sample features will require adjusting collection conditions to generate data confined to those areas of interest. Features may include particulates on a substrate, inclusions within a matrix, thin and multilayer films, and discrete phases from polyphase materials. By adjusting SEM collection parameters, the microanalyst can create X-ray signal generation from predominantly the area of interest, which will result in elemental data collection distinguishable from surrounding material.

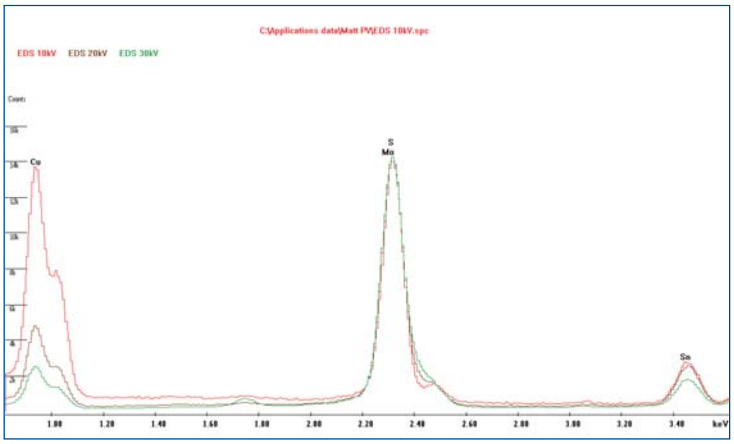


Figure 1 – EDS Spectra at 10kV (red), 20kV (brown) and 30kV (green) indicate a change in the high energy side of the S peak, potentially indicating an increase in the MoL peak.

Electron beam induced X-ray volume of interaction is dependent on a variety of factors including material and atomic number, accelerating voltage, and beam current. A SEM kV range from 2-30kV is a typical usable range sufficient to excite X-ray signal from elements Be through U. The expected X-ray signal microvolume can increase to several microns: 4 microns or more would not be unusual for lower energy X-rays at higher kV. Increases in beam current are much less influential on spatial resolution. SEM probe size may increase by several nanometers in the 1-10nA beam current range, resulting in an X-ray signal generation increase in the tens of nanometer range. Changing either of these electron beam parameters has a greater effect with elements of a lower atomic number, Z. Therefore, a microanalyst who strives to optimize signal collection from identified features can vary electron beam parameters to produce X-rays targeted to specific feature site.

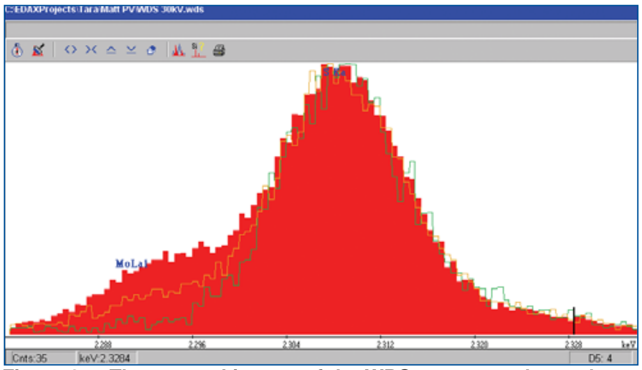


Figure 2 – The zoomed in area of the WDS spectrum shows the presence and increase of MoL more clearly - 30kV (red) yields the highest MoL peak compared to 20kV (yellow) and 10kV (green).

Two examples illustrate the ability of a microanalyst to alter SEM conditions to produce an X-ray signal corresponding to the desired feature. In the first example, a multilayer photovoltaic thin film contains layers consisting of several different characteristic elements including CdS and Mo. Incremental increases in kV from 10, 20, and 30kV create a deeper excitation volume for characteristic X-rays. In the overlay of these three different kV's, (see Figure 1), the S K peak in the EDS spectrum becomes broader at the higher energy side, possibly indicating the presence of MoL. However, the increase in MoL is difficult to ascertain via EDS alone due to the overlapping nature of the S K peak at 2.307keV from the CdS upper layer with the MoL peak at 2.293keV in an underlying layer. Therefore, WDS was used to verify presence and increase in MoL. (Cont'd on Pg. 5)

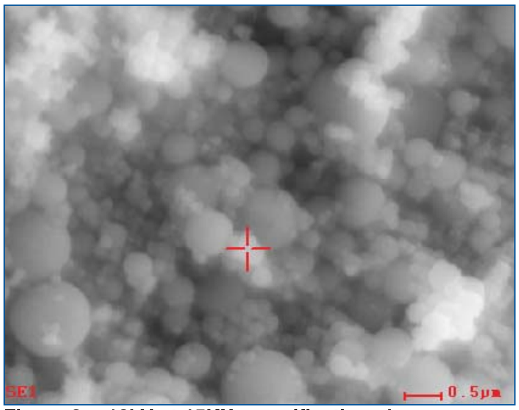


Figure 3 - 10kV at 15KX magnification shows a brighter particle of interest, approximately 400nm.

Optimization of Collection Parameters for Site Specific Feature Microanalysis (Cont'd. from Pg. 4)

MoL peak size becomes more apparent at the higher kV where deeper penetration is expected, confirming the presence in the underlying layer see (Figure 2). Quantitative assessment, not performed here, would be more accurate with WDS data comparisons.

A second example uses a common nanoparticle sample. Due to their high atomic surface area and enhanced material properties, usage of nanoparticles is increasing for a wide range of applications including biomedical work, ceramics, and composite materials. Variation in nanoparticle size and chemistry will alter final product material properties. Characterization of individual nanoparticles can be essential in total composition analysis. A visibly different 400nm nanoparticle was selected for elemental analysis to determine chemistry as compared to the surrounding nanoparticles, see Figure 3. At an accelerating voltage of 10kV and a beam current of 11nA, the interaction volume of the electron beam and as a result the EDS spectrum has a broadened peak, indicating the possibility of PtM overlapping with NbL, see Figure 4. The WDS overlay on the EDS shows spectra from different particles (in brown). The green clearly shows the bright particle is platinum and the darker particles are niobium.

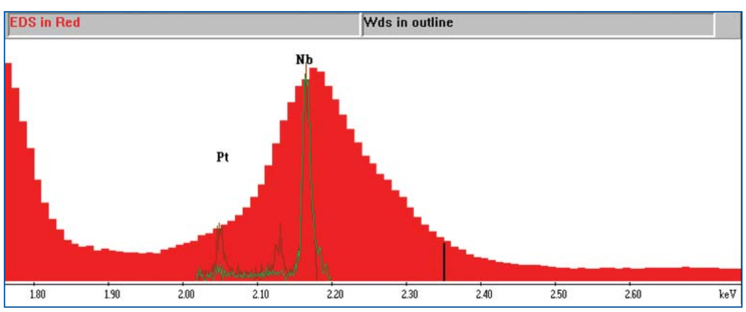


Figure 4 – EDS spectrum in red shows a broadened NbL peak indicating the possibility of Pt. The brown WDS spectrum outline shows an X-ray a collection from bright particle that confirms Pt, whereas the green outline of surrounding particles shows Nb.

For smaller particles, such as the 150nm particle in Figure 5, a reduced accelerating voltage of 5kV is used to decrease the interaction region. Two different beam currents, 7nA and 1.3nA were used to demonstrate lower beam current, which adequately confines the signal into only the platinum particle. This can be seen in the WDS spectra overlay in Figure 6 showing higher PtM intensity and reduced surrounding particle Nb signal.

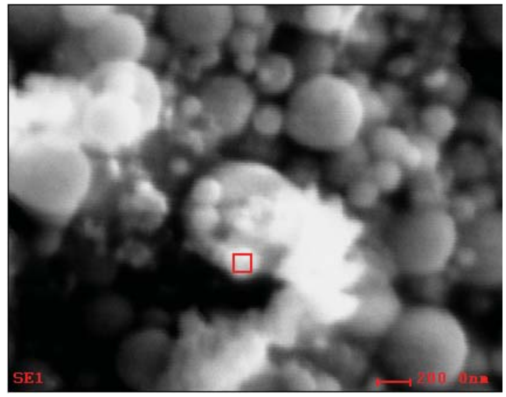


Figure 5 – 5kV SEM image at 35KX magnification show is approximately 150nm nanoparticle on a larger nanoparticle.

Characteristics of electron beam conditions allow microanalysis to isolate X-ray signal generation to features of interest in order to maximize elemental analysis quality and accuracy. An understanding of how adjustments to collection parameters affect the analysis area is important. Applying proper conditions will ensure acquisition of the desired data and enhanced performance.

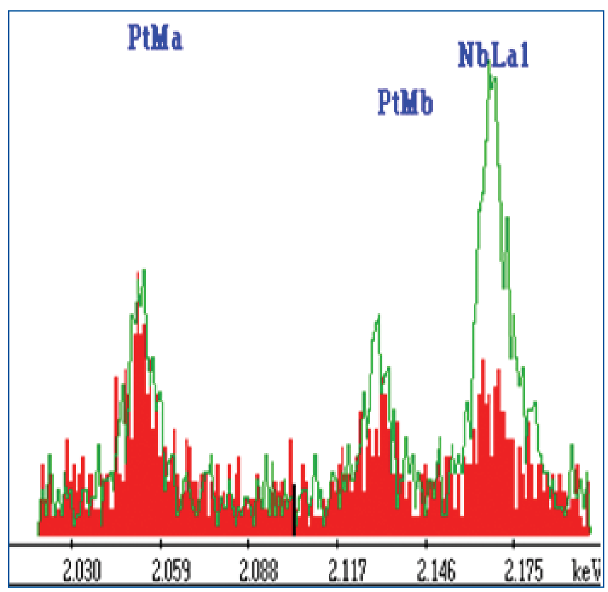


Figure 6 – A spectrum from lower beam current (in red) shows reduced NbL signal (from underlying particle) and therefore, lower volume of interaction isolated to the Pt particle compared to the higher beam current spectrum (in outline).

TRAINING AND EVENTS

World-Wide Events

September 1-3	JAIMA Expo 2010	Chiba, Japan
September 15-18	Archäometrietagung	Bochum, Germany
September 19-22	Micro & Nano Engineering	Genda/Italy
September 19-24	IMC17	Rio, Brazil
September 28 - October 1	HET Instrument 2010	Amsterdam, Netherlands
September 29 - October 1	Metallographie-Tagung 2010	Leoben, Austria
October 7-8	Bauchemietagung	Dortmund, Germany
October 17-21	MS&T (The Materials Sci. & Tech. Expo.)	Houston, TX
November 29 - December 3	MRS (Materials Research Society)	Boston, MA

***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:

- ◆ September 14-16 (W)
- ◆ September 16-17 (T)
- ◆ September 28-30 (T)
- ◆ November 11-12 (T)
- ◆ November 23-25 (T)

Pegasus:

- ◆ October 18-22 (W)

EBSD:

- ◆ September 13-15 (T)

WDS (LEXS):

- ◆ October 12-14 (T)

Japan

Microanalysis Courses:

- ◆ 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:

- ◆ September 14-16 Mahwah, NJ
- ◆ October 18-22 Mahwah, NJ (Includes TEAM™ EDS Software)
- ◆ November 9-11 Mahwah, NJ

EBSD OIM™ Academy:

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

Micro-XRF: (Orbis only)

- ◆ September 28-30 Mahwah, NJ

Marita Lally joined EDAX in February 2000. She is the Assistant Human Resource Manager/Payroll Specialist in our EDAX Mahwah, NJ office. In 1986, Marita received a BA in Management Information Systems from Pace University, Pleasantville, NY.

Prior to joining EDAX, Marita worked as a payroll processor for the Monroe-Woodbury School District in Central Valley, NY. Additionally, she worked for several years as the Payroll Manager for Loews Theatres Management Corp. in Secaucus, NJ.

In her current role, Marita is responsible for payroll, benefits, and Human Resource programs for identified business units within the Materials Analysis Division. Her primary responsibilities include processing payroll and employee benefits for EDAX, CAMECA, and SPECTRO. Other functions include monitoring Short Term Disability, workers compensation, OSHA compliance, 401k administration, and maintaining various programs including tuition reimbursement and company leased cars. Marita also conducts annual employee training programs including Preventing Workplace Harassment and benefit open enrollment presentations. She is also an active member of the Activities Committee, which organizes various company parties, picnics, and social functions within the Materials Analysis Division.

Marita is a resident of upstate New York. In her spare time she and her husband Patrick enjoy the outdoors, camping, and hiking. They maintain an active and busy lifestyle with their three teenage children, Coleen, Joseph, and Daniel.

Mike Tedeschi joined EDAX in August 1998 as the IT Manager. He is located in our EDAX Mahwah, NJ office. Prior to joining EDAX, he was employed with Philips Electronics, first as a Computer Programmer and later as a Programmer Analyst.

In 1983, Mike earned his degree in Computer Programming from The Computer Processing Institute. He is also certified in HP-UX 10.x System Administration and Windows 2003 Server Administration. He began his career in IT as a Junior Programmer with Profit Control Systems in Pinebrook, NJ.

In his current role, Mike is responsible for all facets of the EDAX network, including MFG Pro, server administration, end-user support, and IT disaster recovery. Some of his primary functions of end-user support include network access privileges, PC configuration, Lotus Notes E-mail and databases, and remote access. Mike also provides various departments with support for projects which have IT-related components. He works closely with AMETEK Corporate IT colleagues in various capacities. Most recently, Mike became a member of AMETEK Working Groups to assist in determining the direction of AMETEK IT standards.

In addition to supporting EDAX, Mike is also responsible for providing IT support for the SPECTRO and CAMECA U.S. business units.

Mike is a life-long resident of Northern New Jersey. In his spare time, he enjoys music, golfing, and bowling. His daughter Jennifer recently graduated from Vernon High School and is preparing to attend St. Joseph's University in Philadelphia, PA and his son Matt will be a junior at Vernon High School this fall.

Manchester Metropolitan University, UK

The world of materials is a very complex field and our ability to understand it is based on analytical techniques one can employ to gather bits and pieces of information. In general terms there is no single analytical technique that exists which can provide a non-collaborated description of a material or process. Many naturally occurring or model materials have an inhomogeneous internal structure and require point-by-point analysis in order to describe them accurately. Even having achieved this, a full understanding of the phenomena can only be acquired by synthesis of data from different analytical methods.

In 2006, with the help of various companies, Manchester Metropolitan University (MMU) developed an analysis system that allows information collection by different techniques to be performed from the same point on a sample. The system is based on the Zeiss Supra 40 Variable Pressure FEG SEM and has the world's most comprehensive set of analytical techniques on a single microscope. The EDAX Trident System forms a part of the analytical constellation. The analytical system is located in a UK National Facility for multi-function nano-scale analysis of material and provides free analysis for UK academia. This versatile system allows a wide variety of materials to be analyzed.

Professor Roy Wogelius's group from Manchester University has used the system to investigate copper distribution in fossilized organic matter. The fast counting ability and wide collection angle of the new EDAX Apollo 40 Silicon Drift Detector has allowed MMU to identify copper in samples on the level of 200 ppm.

A combination of elemental mapping with Cathodoluminescence has allowed reconstruction of paleo-fluid processes within sedimentary basins and hydrocarbon reservoirs for Professor Kevin Taylor, Environmental Geographical Sciences Staff, MMU. The maps improve the prediction of the petrophysical properties of sub-surface hydrocarbon reserves and patterns of mineral deposition in economic ore deposits.

Oxidation of Chromium-containing alloys is studied by the Surface Coating and Characterization Research Group at MMU. A difficulty arises due to the oxygen K line sitting between two chromium L lines. It is close to impossible to quantify oxygen presence reliably using the EDX detector. The situation is dramatically improved by using the EDAX Wave Dispersive LEXS unit. In this case, all three lines are resolved and further careful deconvolution allows composition quantification.

Some materials, especially in nano-particulate form (often found from environmental pollution) are modified under electron beams. In these cases, the ability to perform analysis at low electron beam energy and at liquid nitrogen temperature is the only way to perform analysis. MMU will apply the Apollo 40 high efficiency counting facility and new particle analysis software from EDAX to the task. Information about the elemental composition is then supplemented by micro Raman data to identify chemical compounds.

"EDAX is always a great source of knowledge and support for us. Their training courses are exceptionally well organized and are run by experts. At the time when some companies have downsized the amount and quality of technical support, EDAX is still exceedingly responsive and supportive. They have great products, bring new ones to the market, listen to the customers and are alert to what else we, as the customers, would like to have", noted Dr. Vladimir Vishnyakov, Department of Chemistry and Materials.

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AMETEK MATERIALS ANALYSIS DIVISION

EDAX advanced microanalysis solutions

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