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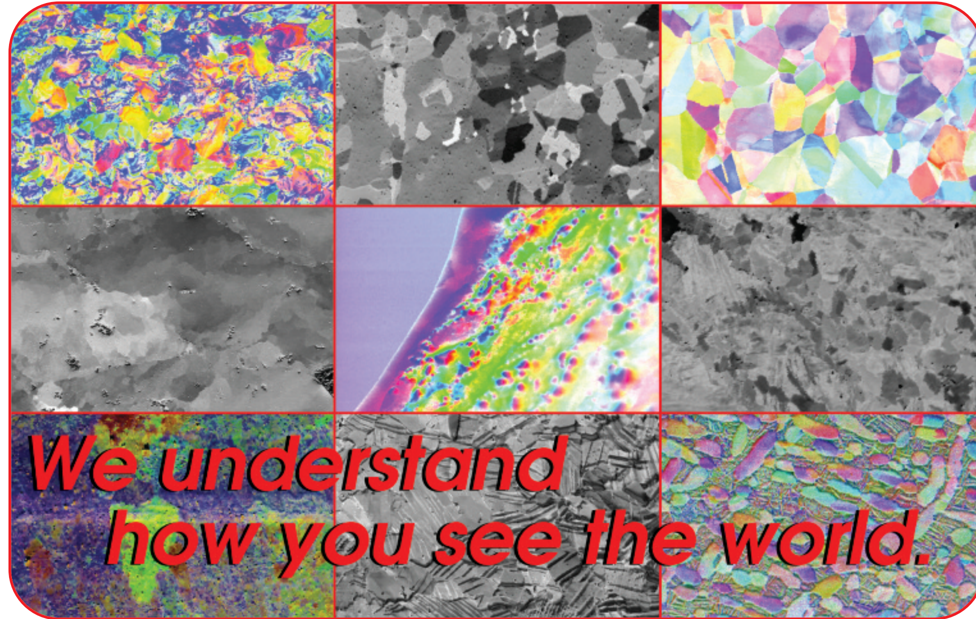
EDAXinsight

March 2014

Volume 12 Issue 1



EDAX NEWS

EDAX Brings Octane Silicon Drift Detector (SDD) Technology to the Transmission Electron Microscope

At Pittcon 2014, EDAX announced the expansion of its highly successful Octane SDD Series to include three new models for the Transmission Electron Microscope (TEM). Based on the state-of-the-art design of the company's Octane Scanning Electron Microscope (SEM) series, the Octane TEM portfolio offers the same market leading performance and delivers the ultimate in analytical capability for the TEM.

While silicon drift technology has been prevalent in the SEM marketplace for over 10 years and has almost completely replaced the Si(Li) detector, this conversion has been much slower for the TEM. There is no inherent increase in solid angle for an SDD versus a Si(Li) detector and the benefits of the early SDD detectors for the SEM - capacity to process very high count rates and no need for liquid nitrogen - did not correlate as well with TEM, where count rates are inherently low and liquid nitrogen is required for the operation of the microscope. Additionally, the early generations of SDDs had drawbacks, with lower signal to noise ratio and a poorer light element resolution than their Si(Li) predecessors. The early SDD did not therefore have a lot to offer for the TEM.

It is only recently that SDDs have been designed to maximize the analytical performance of the TEM. In its progression towards present day Octane detectors, EDAX has advanced SDD technology through a series of innovations. The current SDDs offer the best light element resolution ever and improved signal to noise ratio, which exceed those of earlier generations and surpass the capability of the older SiLi detectors.

In addition to the improved performance of the SDD chip itself, EDAX introduced the first windowless detector solution to the TEM market in 2010, with the release of the Apollo XLTW. The removal of the window and supporting grid offer an increase in the effective solid angle.

(Continued from Page 1)

The benefits include up to a 500% increase in light element detection and a 35% overall increase in detection of higher Z elements (Figure 1). The detector is protected from issues of high backscatter electrons or loss of vacuum via safety interlocks, which automatically retract the detector when needed. As the SDD detector does not require cooling to the same temperatures as the SiLi detector, icing is a non-issue.

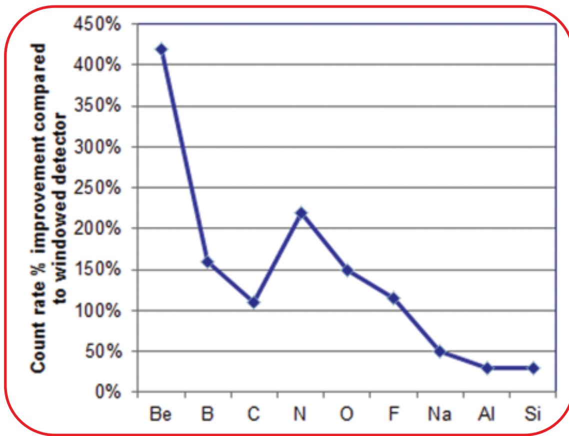


Figure 1. Improvement ratio by element for windowless SDD.

Another great advancement in SDD technology is the development of large area detectors to gain additional solid angle, which allows for collection of greater signal under given conditions (Figure 2). This is especially critical in the analysis of TEM foils, where samples typically cannot withstand tremendous beam current. The solid angle (Ω) is calculated using the formula A*DTS^2, where A is the effective detector area and DTS is the detector to sample distance. In order to increase solid angle, one must balance increasing the effective detector area with keeping the detector distance to sample short. The TEM provides additional challenges as the EDS manufacturer must contend with more

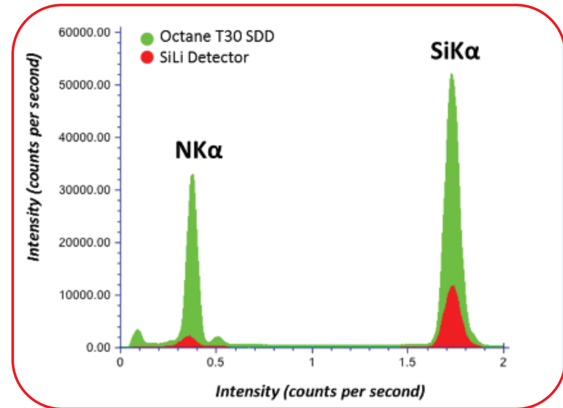


Figure 3. Comparative spectra for Si(Li) and Octane Optima of Si_3N_4 collected on FEI CM200 with ST lens.

complex pole configurations and often, multiple detectors. Specifically, there is a hard limit to increasing solid angle using a circular detector as it becomes shadowed in the upper and lower regions. To tackle this issue, in 2012 EDAX introduced a 100 mm^2, oval-shaped detector with a market-leading 1.1 steradian (sr) solid angle.

The Octane SDD Series for the TEM includes the Octane T Plus, which is a 30 mm^2 SDD with a super ultra-thin polymer window (SUTW) providing an entry point into SDD technology for the TEM; the premium level Octane T Ultima, offering solid angles up to 1.1 sr; and the Octane Optima, the first TEM SDD system on the market to be specifically optimized for individual TEMs. The Octane Optima offers a solid angle of .3-.5 sr depending on the pole piece, as a great balance of price and performance (Figure 3). All Octane TEM Systems come with TEAM™ EDS for TEM software, offering the user the ease of use and analytical intelligence, for which TEAM™ software is well known. Specific algorithms for TEM quantification are included, in addition to standard Smart Features like Smart Data Reporting and EXpert ID.

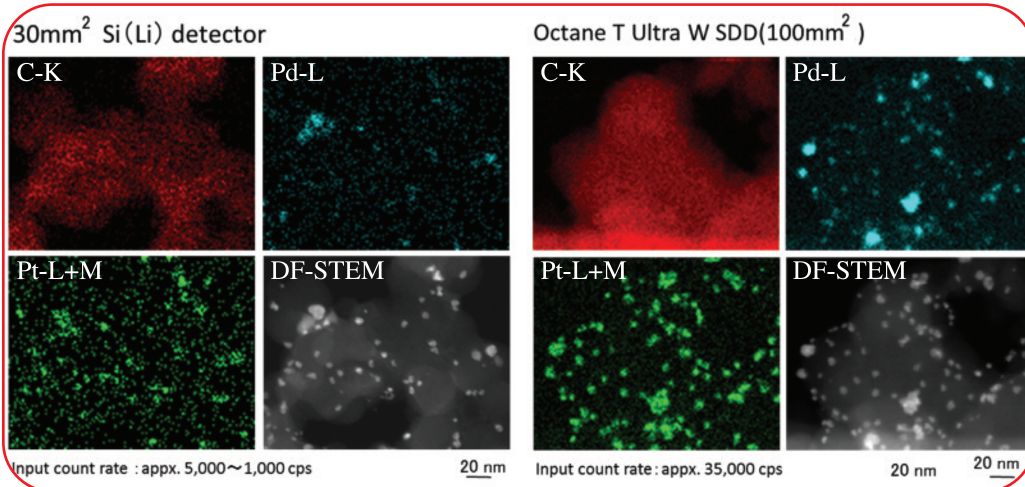


Figure 2. Comparison of Si(Li) and 100 mm^2 SDD maps.

With the launch of the Octane SDD Series for the TEM for both new systems and field upgrades, EDAX now offers the ideal solution for all TEM applications. With a range of SDD sizes with column specific designs available, Octane TEM detectors are able to maximize solid angles by optimizing both size and geometry, ensuring users the best analytical performance from their TEM. The promise of SDD technology has now been delivered to the TEM.

Orbis Vision Version 2.1 Update – Revised KENO Function

One of the key quantitative features in the Orbis Vision Micro X-ray Fluorescence (Micro-XRF) software is the Known Elements Not Observed (KENO) function, which allows compensation of known values for elements that were not detected in the spectrum. This is particularly useful in quantifying samples which have known concentrations of organic elements, since micro-XRF is typically unable to detect elements lighter than sodium. With the recent release of Orbis Vision version 2.1, the KENO function has been updated, so that whenever quantification is performed, the Fundamental Parameters (FP) routine will directly use whatever values are active in the KENO window and apply them for both standard-less and standardized measurements. This allows the user to use calibration standards that may have different KENO elements and values than those which are in the sample. This is shown in the following example, where an FP calibration is done using different KENO values for the standard versus the samples.

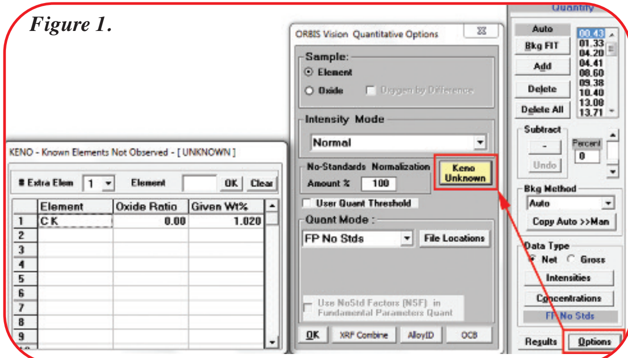


Figure 1.

In Figure 1, a stainless steel standard is measured, which contains a KENO value for carbon at 1.02 weight percent. When the KENO icon is highlighted yellow, it signifies that it is active.

The left-hand window in Figure 2 shows the initial No-Standards results, which includes the initial KENO value for carbon. As a result of using the KENO value, quantifying this spectrum with FP No-Standards normalizes the detected elements to a total of 98.8%. Note that the KENO window is marked "KENO-Unknown" thus far.

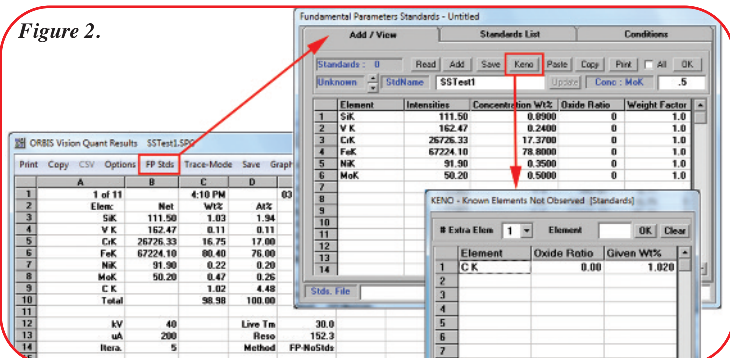


Figure 2.

This spectrum will now be used as a new calibration standard, and the known weight percent values are entered into the Concentration column of the FP window, as shown on the right-hand side of Figure 2. For any standards that have KENO elements, such as this example, the elements and values must be entered through the KENO dialog box from the FP window, as highlighted in red in Figure 2. Note that this KENO window is now marked "KENO-Standard." If the standard has no KENO elements, then this dialog must be cleared. Once the values are entered, click "Add" to apply this standard. It is possible to use multiple standards that each have different KENO elements and concentrations. Save the calibration file (.MSF) once all standards are added.

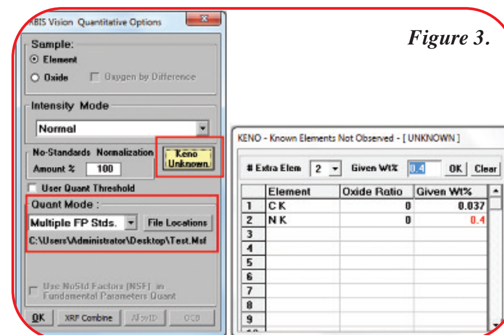
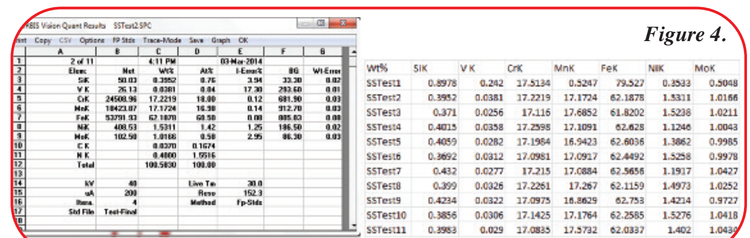


Figure 3.

Once the .MSF file is saved, it can be used to quantify other "unknown" stainless steels that have similar compositions. However, since different steels may have varying KENO values from the initial calibration standards, the user should open the unknown spectrum file and modify the KENO elements accordingly, as shown in Figure 3. In this sample, to reflect these changes in KENO values, carbon was changed to 0.037%, and a second KENO entry was made for nitrogen at 0.4%. These new values will be applied for all quantifications thereafter until the user deactivates or changes the KENO values. After exiting the KENO window, load the appropriate .MSF file from the File Locations menu, and select "Multiple FP Stds" from the Quant Mode dropdown, as indicated in red in Figure 3.

At this point, any spectra that are quantified will use the newer KENO values, and can be applied for both individual quantification or bulk data reprocessing. Figure 4 shows example results from each.



Wt%	SiK	V K	CrK	MnK	FeK	NiK	MoK
SSTest1	0.8978	0.242	17.5134	0.5247	79.527	0.3533	0.5048
SSTest2	0.3952	0.0381	17.2219	17.1724	62.1878	1.5311	1.0166
SSTest3	0.371	0.0256	17.116	17.6852	61.8202	1.5238	1.0211
SSTest4	0.4015	0.0358	17.2598	17.1091	62.628	1.1246	1.0043
SSTest5	0.4059	0.0282	17.1984	16.9423	62.6036	1.3862	0.9985
SSTest6	0.3692	0.0312	17.0981	17.0917	62.4492	1.5258	0.9978
SSTest7	0.432	0.0277	17.215	17.0884	62.5656	1.1917	1.0427
SSTest8	0.399	0.0326	17.2261	17.267	62.1159	1.4973	1.0252
SSTest9	0.4234	0.0322	17.0975	16.8629	62.753	1.4214	0.9727
SSTest10	0.3856	0.0306	17.1425	17.1764	62.2585	1.5276	1.0418
SSTest11	0.3983	0.029	17.0835	17.5732	62.0337	1.402	1.0434

Figure 4.

EDAX® Smart Insight

APPLICATION NOTE

EDAX Introduces New Pattern Region of Interest Analysis System (PRIAS)

EDAX is introducing an innovative new imaging technique named PRIAS, for Pattern Region of Interest Analysis System, as a key feature within the TEAM™ 4.1 Analysis System release. This technique enables enhanced microstructural imaging of traditional Electron Backscatter Diffraction (EBSD) materials such as metals, ceramics, semiconductors, and minerals, as well as new characterization opportunities for glasses and plastics.

Typical EBSD samples require a well-prepared surface to facilitate high-quality diffraction pattern collection. EBSD also requires the sample to be tilted to approximately 70° towards the EBSD detector to maximize pattern intensity upon the phosphor screen. These requirements make traditional SEM imaging more difficult, particularly for single-phase materials, as the topographic contrast detected by the Secondary Electron (SE) detector is minimized by the EBSD sample preparation and the atomic number contrast detected by the Backscatter Electron (BSE) detector is minimized by the tilting away from the traditional BSE detector positioning below the Scanning Electron Microscope (SEM) pole piece.

Traditionally, microstructural imaging of EBSD samples has been supplemented by a Forward Scatter Detector (FSD) system, which is a set of one or more solid-state diodes positioned around the perimeter of the EBSD detector phosphor screen that act as electron detectors. A typical geometry of the sample, SEM pole piece, SE and BSE detectors, EBSD phosphor screen, and FSD detector is shown in Figure 1. FSD micrographs can be obtained by mapping the variation of detected electron intensity as a function of beam position. Multiple contrasts can be detected using an FSD system by varying the position of the detector, either through insertion and retraction of the EBSD phosphor screen (and corresponding FSD), or through positioning multiple FSD detectors. Each FSD detector requires amplification and analog-to-digital conversion circuitry to turn the detected electron signal into a useable imaging signal. This requirement effectively limits the number of FSD detectors that can be deployed, as well as the number that can be imaged simultaneously.

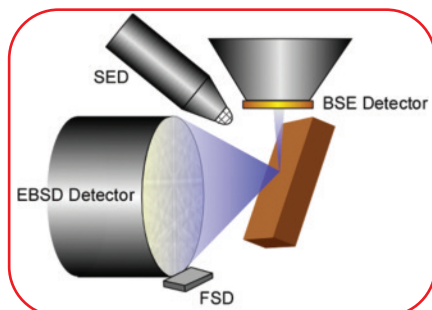


Figure 1. A typical geometry of an EBSD sample.

PRIAS provides an innovative new approach for microstructural imaging by synergistically using the EBSD detector system both as an EBSD pattern collection tool and as an array of positional electron detectors distributed across the phosphor screen. This approach takes advantage of the fact that the phosphor screen is positioned to collect the maximum intensity of the spatial distribution of backscattered electrons from the tilted EBSD sample. For PRIAS imaging, regions of interest (ROIs) are defined within the image of the phosphor screen and PRIAS micrographs can be obtained by mapping the variation of detected electron intensity with each ROI. One advantage of PRIAS imaging is that multiple ROIs can be measured simultaneously. In the current implementation, up to 25 positional ROIs can be analyzed.

Multiple image contrast mechanisms can be detected using the different PRIAS ROI detectors. These contrasts include orientation contrast (Figure 2), atomic number contrast (Figure 3), and topographic contrast (Figure 4). Images can also be created by weighted arithmetic processing and/or RGB color mixing of multiple detectors. This allows users to enhance or suppress the contrast mechanism of interest and highlight specific microstructural features within the sample. An example is shown in Figure 5 from a TiAl sample where three ROIs were selected for RGB coloring simultaneously with two different ROIs for gray scale shading. Differences in ROIs can also be used to determine grain boundary positions, as shown in Figure 6 for an Inconel 600 sample.

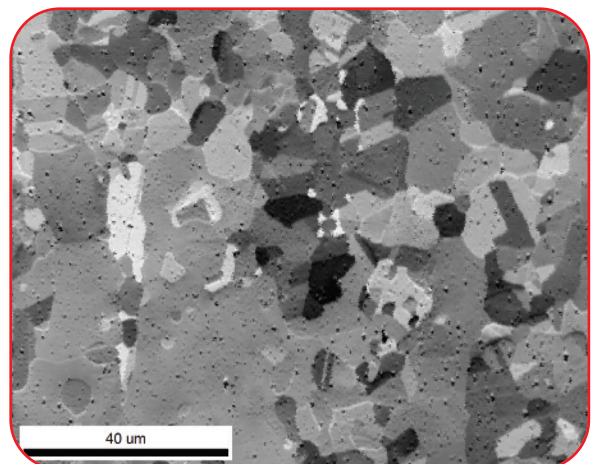


Figure 2. Orientation contrast from a Duplex 2205 steel.

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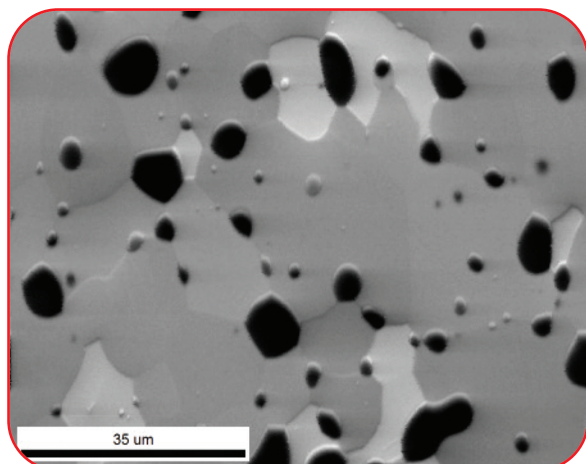

Figure 3. Atomic number contrast from a Mo-Si multi-phase sample.

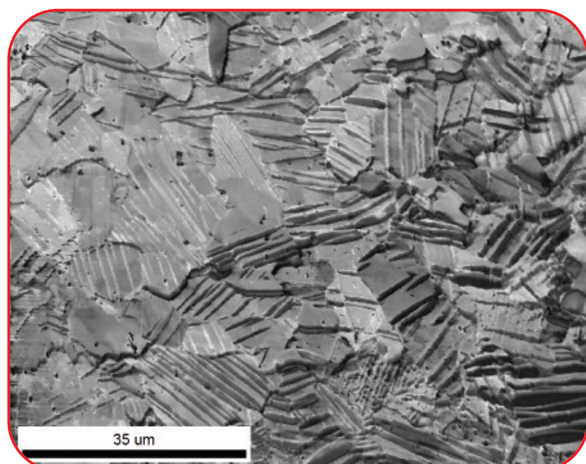

Figure 4. Topographical contrast from a Rh-W alloy.

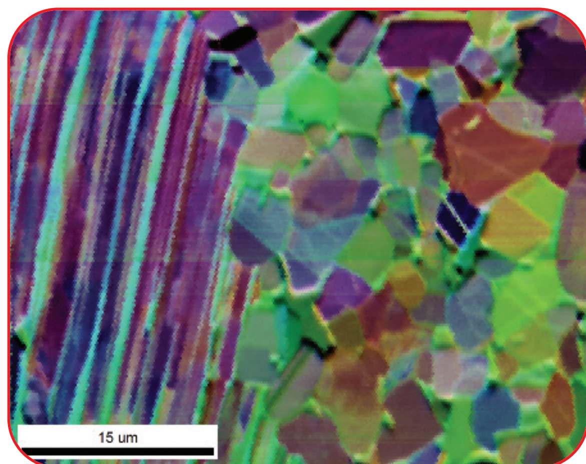

Figure 5. Three ROIs selected for RGB coloring simultaneously with two differenct ROIs for gray scale shading from a TiAl sample.

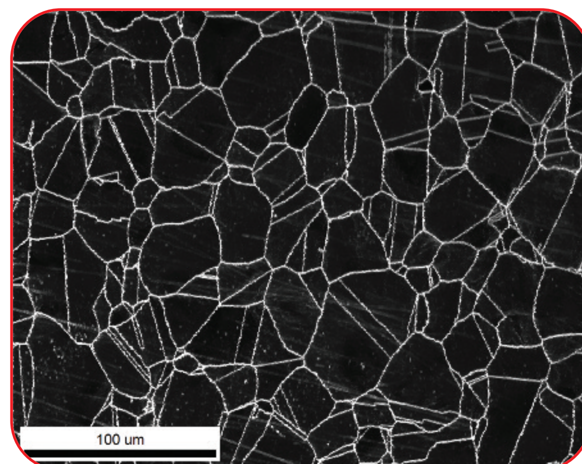

Figure 6. Differences in ROIs can be used to determine grain boundary positions from an Inconel 600 sample.

PRIAS technology has three modes of operation. PRIAS Live is an imaging technique independent from Orientation Imaging Microscopy (OIM™)/EBSD mapping. In this mode, the Hikari XP camera is binned to produce a low resolution, high frame rate image that does not contain enough diffraction information for accurate EBSD pattern analysis but can be used to detect intensity variations within the specified ROIs. It collects 25 images simultaneously and can be used with the TEAM™ software as an SEM image to guide further analysis. PRIAS Collection is a mode with predefined ROIs on the phosphor screen measured concurrently during OIM™ /EBSD mapping to provide PRIAS data automatically. These ROI signal channels are then available for subsequent visualization within the OIM™ Analysis software. PRIAS Analysis allows for the flexible positioning of ROIs on a set of EBSD patterns saved during OIM™ mapping. With this approach, ROI positioning and sizing can be varied to maximize the information extracted from the PRIAS data. With all three modes, the PRIAS contrast information can be directly correlated to the orientation and phase information collected during TEAM™ EBSD mapping.

PRIAS provides an exciting new way of visualizing microstructure, often without the need for full EBSD mapping. Additionally PRIAS data can be collected at low beam currents and voltages, providing a fast microstructural imaging technique for non-conductive and nanostructured materials. This technique extends the capability of the TEAM™ Analysis System to provide Smart Insight into materials characterization.

Cover shows various images captured with PRIAS.

Worldwide Events

April 1-4
Analytica — München, Germany

April 9
Microscopy Society of the Ohio River Valley — Dayton, OH

April 15-18
Analitika — Moscow, Russia

May 5-9
California Association of Criminalists (CAC) — San Diego, CA

May 11-13
Microscope Society Meeting — Chiba, Japan

May 19-22
CS Mantech — Denver, CO

May 19-23
International Symposium on Archaeometry — Los Angeles, CA

May 22-24
The Southeastern Microscopy Society (SEMS) — Greensville, SC

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

2014 Worldwide Training

To help our present and potential customers obtain the most from their equipment and to increase their expertise in EDS microanalysis, WDS microanalysis, EBSD/OIM™ , and Micro-XRF systems, we organize a number of Operator Courses at the EDAX facilities in North America; Tilburg, NL; Wiesbaden, Germany; Japan, and China.

EUROPE

EDS Microanalysis	
June 19-20 September 11-12	Tilburg* Tilburg*
TEAM™ EDS	
May 13-15 May 20-22 September 23-25	Wiesbaden# Tilburg* Tilburg*
Genesis	
April 14-16	Tilburg*
EBSD	
June 16-18 September 15-17	Tilburg* Tilburg*
TEAM™ Pegasus (EDS & EBSD)	
May 12-16	Wiesbaden#
TEAM™ Neptune (EDS & WDS)	
June 2-6	Wiesbaden#
TEAM™ WDS	
April 1-3	Tilburg*
Orbis: Course & Workshop	
May 6-8	Wiesbaden*

*Presented in English
#Presented in German

JAPAN

EDS Microanalysis	
Genesis	
April 10-11 October 9-10	Osaka Tokyo
TEAM™ EDS	
June 12-13 July 10-11	Tokyo Osaka

CHINA

EDS Microanalysis	
TEAM™ EDS	
May 8 June 9-12	Beijing Shanghai (ACES)
Genesis	
September 15-18	Shanghai (ACES)
EBSD OIM™ Academy	
April 14-17 October 20-23	Shanghai (ACES) Shanghai (ACES)

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
May 20-22	Mahwah, NJ
EBSD OIM™ Academy	
June 10-12	Mahwah, NJ
Micro-XRF	
April 8-10 October 7-9	Mahwah, NJ Mahwah, NJ

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EDAX's First TEAM™ WDS Workshop in Europe

On November 5, 2013, EDAX Europe held its first TEAM™ WDS Workshop. Nearly 40 participants traveled to Wiesbaden, Germany to listen to presentations by EDAX employees and WDS users from various industries.

- **Michaela Schleifer, EDAX Europe Application Manager**

Dr. Schleifer explained how WDS is a useful tool because it shows an improved energy resolution compared to EDS (eg. OKα in EDS ~ 60 eV resolution is four times better in WDS) and it also has better sensitivity. There are two types of WDS spectrometers on the market: the curved crystal spectrometer and the parallel beam spectrometer (PBS), which EDAX produces. In PBS X-ray optics are used to convert the divergent X-rays coming from the sample into a parallel beam. Since the beam is parallel, flat crystals can be used instead of curved ones. The X-ray optics operate on the principle of total reflection. Based on the better energy resolution of a WDS system, compared to EDS, it is possible to solve peak overlapping to show which elements are present in users' samples and also to improve quantitative results. WDS can improve results based on better energy resolution and detection limits especially for light element analytics and low kV analysis. An EDS system is not able to detect samples with low concentration of B (< 2 Wt%). With WDS, users can quantify concentrations in the ppm range of such elements.

- **Dr. Matthias Procop, Institute für Gerätebau, Berlin, Germany**

Dr. Procop's institute is one of the manufacturers of poly-capillary optics, which are used in micro-XRF systems (such as the Orbis) and also in PBS (such as the TEXS). He only explained a little bit about the manufacturing, as this is a secret. The quality of such an optic influences WDS resolution. The important criteria are the divergence of the exit radiation and the optic transmission. Spectrometer alignment becomes more and more critical with higher photon energy and decreased acceptance area. It is very important to take this into account for WDS mapping.

- **Dr. Peter Pölt, FELMI Institute Graz, Austria**

With over 30 years of WDS experience, Dr. Pölt explained that sputtering the surface of a sample always changes the specimen's structure and chemical composition. The depth of the damaged layer is around a few hundred Å and influences the WDS analysis. Peak shift, peak shape changes and satellite lines can provide information about the type of the chemical bonding in compounds. He also demonstrated the importance of selecting the correct standard for quantitative analysis in WDS. The standard should be similar in both composition and bonding to the unknown specimen. It is very difficult to measure carbon concentrations in steel with WDS because the analysis is influenced by different factors, leading to incorrect results.

Participants at the TEAM™ WDS Workshop in Wiesbaden, Germany on November 5, 2013.

- **Dr. Völksch, formerly of the University of Jena, Germany**

Dr. Völksch is an expert in glass chemistry. WDS is an important analytic tool in measuring B, N, O and F in glass chemistry. The concentration of such elements is often too low to detect with EDS. Typical overlays are Ba–Ti, Zn–Nd, P–Zr, which can be resolved by WDS. Glass is very sensitive for irradiation damage during a measurement. Therefore, it is very important that a PBS also works under low beam current conditions.

- **Tim Hattenberg, Aerospace Center NLR, The Netherlands**

Mr. Hattenberg described how an industrial company uses WDS in its day-to-day work. At NLR, WDS is an important supplementary technique to EDS. The company does a lot of quantification in combination with EDS and quantifying the trace elements (< 0.5 Wt%) with WDS. As there are often low concentrations of light elements, WDS gives NLR the possibility of doing a full quantification of the materials.

We would like to thank the speakers and all the participants at the workshop. If you are interested in the speakers' presentations you can find videos on the EDAX News YouTube channel. For more information about WDS and its integration into the EDAX TEAM™ platform or the workshop presentations, please contact Michaela Schleifer, at michaela.schleifer@ametek.de.

EDAX Smart Insight

Pat Camus (left) with the 8th Wisconsin Light Artillery.

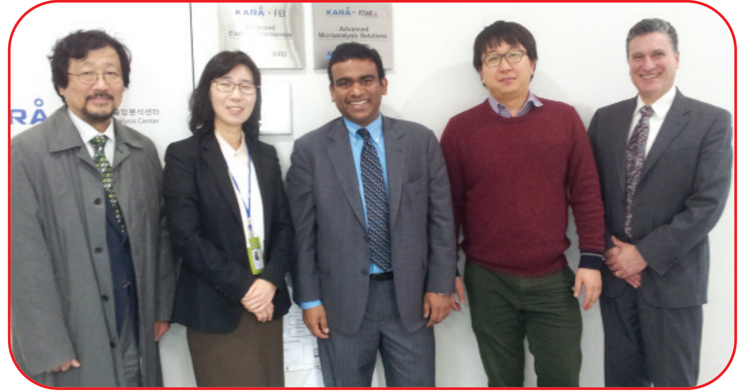
Pat Camus

In June 2013, Pat joined EDAX as a Principal Product Development Engineer on the Innovation Team in Engineering. Located in the Mahwah, NJ office, he is responsible for working with the engineers to resolve issues and test new products, as well as looking for potential new technologies.

Prior to EDAX, Pat spent 15 years at Thermo Fisher Scientific, dealing with electron beam microanalysis (EDS, EBSD and WDS). Previously, he performed Atom Probe Analysis at Oak Ridge National Lab (ORNL), the National Institute of Standards and Technology (NIST), and the University of Wisconsin-Madison for 18 years.

Pat earned a Bachelor of Science degree in Metallurgical Engineering and a Ph.D. in Materials Science and Engineering from the University of Pittsburgh in 1981 and 1986, respectively.

Pat and his wife Georgiann live in Pen Argyl, PA. They have two children, Julianne (25) and Philip (21). In his free time, Pat enjoys firearms marksmanship and miniatures gaming. He was a member of a cannon crew that shot an original 1853 bronze barreled Federal 6-pounder cannon.



(left to right): Mr. Yong Kim of Intec, EDAX distributor in Korea; Ms. Mi Ja Woo, General Manager of KARA; Narayan Vishwanathan, EDAX VP and Business Unit Manager; Mr. Taewoo Lee, Engineer at KARA; and Frank Cumbo, EDAX Director of Sales and Marketing.

Korean Advanced Institute of Science and Technology

The Korean Advanced Institute of Science and Technology (KAIST) unveiled its new Advanced Microanalysis Solution Center, a joint center sponsored by KAIST Research Analysis Center (KARA) and EDAX on February 14, 2014. This is a significant relationship that will foster a new generation of future scientists and engineering users of EDAX equipment in Korea and beyond. KAIST is a world renowned institute, consistently ranked in the top 100 institutions world wide, placing them in the same league as MIT, Stanford, Max Planck, NIMS, IIT Bombay and other leading institutions around the world. Korea is EDAX's fifth largest country in terms of sales volume and home to several of our key accounts, including Samsung, SK Innovation, LG and Posco Steel. We look forward to a long and fruitful relationship between KARA and EDAX.

EDAX Inc.
91 McKee Drive
Mahwah, NJ 07430
Phone (201) 529-4880
E-mail: info.edax@ametek.com
www.edax.com

Art and Layout
Jonathan McMenamin

Contributing Writers
Mike Coy
Laurie Krupa
Andrew Lee
Matt Nowell
Michaela Schleifer

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