EDAX FOCUS

Pileup Effects in Energy Dispersive Spectroscopy

Inside This Issue

Pileup Effects in Energy Dispersive Spectroscopy (EDS): Where do they originate and how are they fixed?

Pileup seems to be a topic of great interest these days. It causes distortions to the EDS spectrum at high count rates, the most troublesome being the sum peaks from the major element peaks. We will discuss the background on why pileup occurs, how to recognize it, and what can be done about it.

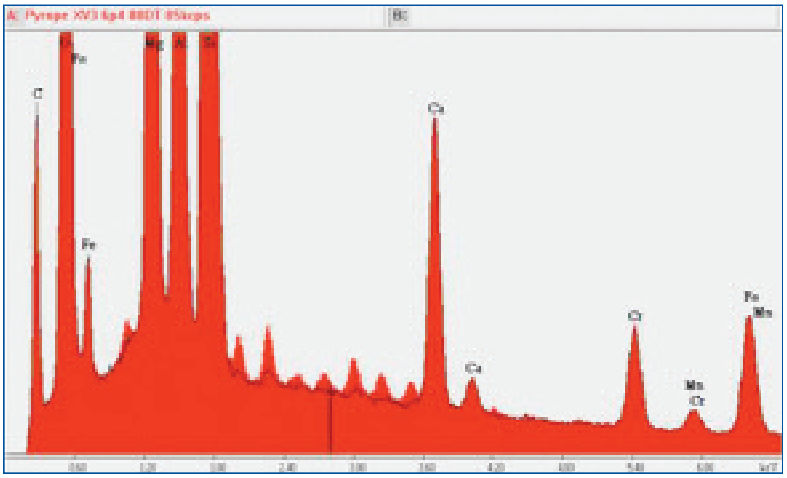


Figure 1: This is a spectrum of a pyrope mineral sample taken at high count rate (85,000 counts per second with 6.4 microseconds Amp Time, resulting in 80% dead time). The spectrum was taken with the Apollo XV SDD. Several pileup peaks are easily visible between the Si and Ca peaks. The black line is the same spectrum after software pileup correction. By comparing the corrected and uncorrected plots, it becomes clear which peaks are affected by pileup. Most of the small peaks are artifacts, but there may be some small amount of sulfur in the specimen.

Pileup originates from a combination of two things. The first is the random arrival times of X-rays. X-rays do not arrive at a regular pace like clock pulses, but rather at random and unpredictable times. This is the cause of the "counting statistics", or statistical error, since the number of X-rays that arrive in a given time varies slightly due to this randomness. The second reason pileup originates is that an energy-dispersive detector and pulse processor takes a finite amount of time to determine the energy of an X-ray once it arrives. The problem arises when a second X-ray arrives while the processor is still trying to determine the energy of the first one. When this happens, it is called pileup (since the two X-rays are "piled up" inside the detector and processor).

Pileup does not occur on a regular basis, it occurs with a certain probability. As with total counts, we can predict (on average) how many times it will happen in a given spectrum, but specific pileup events cannot be predicted.

To understand how pileup affects your spectrum, we need to look more closely at how the EDS detector determines the energy of an X-ray. When an X-ray arrives, it generates an electrical pulse whose amplitude is proportional to the X-ray energy. The pulse contains noise when it first leaves the detector, so it must be filtered before its amplitude can be measured accurately and converted to energy. The filtering is what takes the time, and the amount of time is determined by the Amp Time (shaping time). A longer Amp Time means more filtering and thus less noise and better energy resolution. It also means a greater chance that another X-ray will come along and generate another pulse. When two pulses are in the filter at the same time, it distorts the amplitude. The amount of distortion depends on how much later the second X-ray arrives. (Cont'd on Pg. 2)

Pileup Effects in EDS (Cont'd. from Pg. 1)

Three simple cases are shown in Figure 2. In the top case, the two X-rays arrive far enough apart that the filter (the triangular signal, as generated by the Digital Pulse Processor or DPP) is finished by the time the second X-ray arrives. The sketch of the spectrum at the right of the figure shows two separate counts, which is the desired result. Let's look at the bottom case, where the two X-rays arrive at exactly the same time. In this case, the energies of the two X-rays are added together to produce a sum peak. But X-rays seldom arrive at exactly the same time, as shown in the middle case. Here, the distortion from the second X-ray causes the energy to be higher, but not by as much as the sum of the two energies.

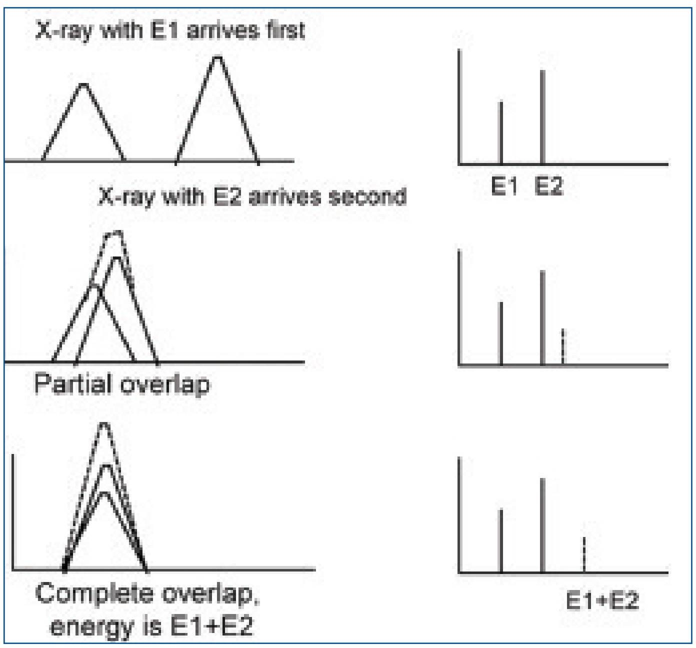


Figure 2: X-rays arrive at the detector at random times. If the time between X-rays is longer than the Amp Time, then the two X-rays are processed separately. However, sometimes a second X-ray arrives before the end of the Amp Time. In this case, the two X-rays overlap in the pulse processor and appear in the spectrum as a single count. The energy depends on the time between the X-rays and thus the degree of overlap. This figure shows well separated X-rays at the top, partially overlapped X-rays in the center, and fully overlapped X-rays at the bottom. On the left are the pulses vs. time, and on the right is the resulting spectrum. The bottom panel corresponds to the "sum peaks" that are very hard to remove with hardware.

The distorted energy depends on the time between the X-rays, the Amp Time, and the shape of the filter pulse. For a modern digital pulse processor, the pulse shape can be carefully controlled. The triangle shape shown is a simple case, but the relation between arrival times and energy can be calculated for any shape. The result is the "pileup spectrum", which covers the range from the larger energy to the sum. For a large peak in a spectrum, if there were no pileup removal, there will be pileup from the location of that peak all the way up to twice its energy.

How do we reduce pileup? The easiest and most effective way to detect pileup is to look at the arrival times of the X-rays. The pulse processor has additional filters called pileup inspection channels with shorter filter times than the Amp Time. They work by detecting the arrival times of the X-rays and sending a pileup reject signal when two (or more) X-rays arrive within the selected Amp Time.

The pileup reject signal causes the pileup count to be dropped. This simple but effective method removes almost the entire pileup from appearing in the spectra for Si(Li) detectors. However, there will always be some X-rays that arrive too close together for even the best pileup inspection to separate. This situation is why sum peaks are seen in a typical spectrum, since the unrejected pileup events are very close together and appear at nearly the sum energy.

Another method to reduce pileup effects in the spectrum is to use a software correction. The correction can remove the inevitable pileup peaks that remain after hardware pileup rejection. It is described in more detail below.

Silicon Drift Detectors (SDDs) can be operated at very short Amp Times and still achieve reasonable energy resolution. Shorter Amp Times help decrease pileup, because X-rays can arrive closer together without pileup occurring. If the count rate is increased to get data faster, as is often the case with SDDs, then pileup occurs more often. The result is that pileup occurs for both SDDs and Si(Li) detectors. The short Amp Time used with SDDs makes pileup inspection more difficult, since the pileup inspection channels must have filter times shorter than the Amp Time. Pileup inspection channels do not reject as great a fraction of the pileup events, and so more pileup appears in the spectrum. Pileup effects seem much worse for SDDs because we can see more of them.

Pileup Effects in EDS (Cont'd. from Pg. 2)

The final step that can be performed to reduce pileup is a software correction. As we discussed previously, the amount of pileup can be predicted on average. This prediction can be very specific, predicting the pileup from each channel in the spectrum and where it will appear in the higher channels. The prediction has two valuable uses: it can help determine what is pileup and what is real, and it can be used to correct the spectrum to further reduce the pileup effects. Note that the effects of the pileup inspection channels must be considered in this calculation, since even in the worst case they still remove the majority of the pileup events.

If the calculated pileup is subtracted from the spectrum, it reduces the pileup peaks to very small levels. This "software correction" is often nearly perfect – the imperfections are smaller than the noise. This is the case for the Basalt spectrum shown in the Figure 3, which is a complex spectrum taken at a very high-count rate. Note that there are a lot of pileup peaks, especially between 2 and 3.5 keV that are completely removed by this software correction method. Occasionally the calculated correction does not get the shape of the calculated sum peak precisely right, which causes the corrected spectrum to have small bumps and wiggles (see the arrow in Figure 3). Pileup is still reduced by a large amount, and which peaks are due to pileup is made very clear by the software correction.

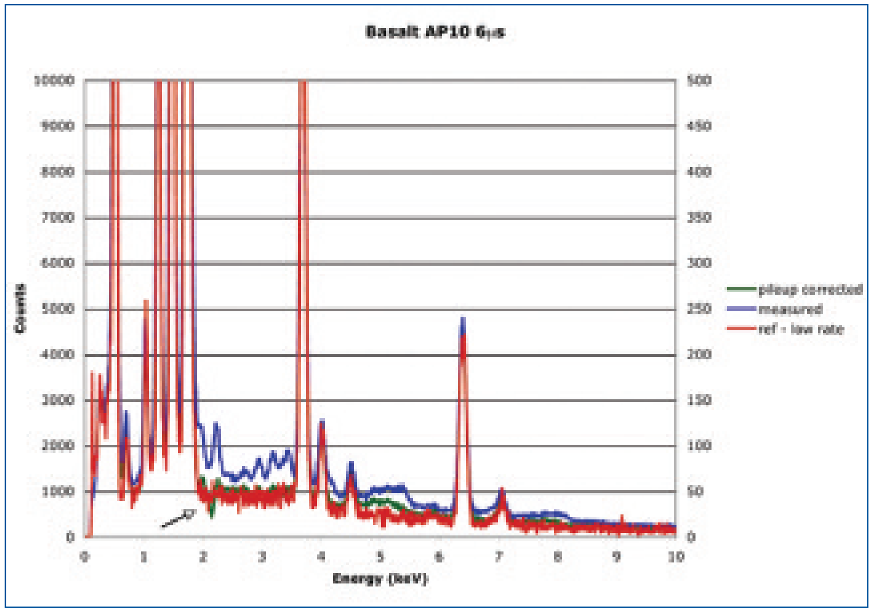


Figure 3: This is a spectrum of a basalt taken with an AP10 detector at 164,000 counts per second and a 6 microsecond Amp Time. The original spectrum is plotted in blue. Each of the main peaks (which are cut off in this figure) produces a pileup peak. The pileup peaks can be identified by comparing to the spectrum in red, taken at much lower count rate and free of pileup peaks. The hardware removes almost all of the pileup at the high count rate, but these sum peaks remain (the text explains why). The green plot is the blue spectrum after software pileup correction. Note that the pileup peaks are removed and the higher background due to continuum pileup is also reduced. The corrected spectrum agrees very well with the "true" spectrum. The arrow points to the one remaining pileup artifact where the software correction did not calculate quite the right peak shape.

As a final note, there is always one question that arises about pileup correction. Why can't the pileup events be added back to the spectrum, rather than rejected? This idea appears from time to time, as "lossless counting" or "loss-free counting". The problem goes back to the above discussion about pileup – we can only calculate the average number of pileup events, not specific events. If two X-rays arrive within the pulse pair resolving time, their individual energies are lost. How many times this occurs can be calculated on average and that average can be used as a correction, but it is an average based on the measured spectrum including its statistical error. Adding the calculated pileup counts back into the spectrum will increase the number of counts, but will NOT decrease the statistical error. Therefore, no improvement is made.

The conclusion is that pileup rejection is best done in the pulse processor, where the X-ray arrival times can be individually measured and pileup counts reliably rejected. Digital pulse processors can have several fast pileup inspection channels and do an excellent job of pileup rejection, even at short Amp Times and high-count rates. Just as importantly, digital pulse processors have a reliable filter shape that facilitates accurate calculation of the inevitable pileup counts that will always get past the inspection channels and cause sum peaks. This calculated pileup spectrum could be used to determine which peaks are due to pileup and which are real, and to further correct the spectrum and reduce pileup.

TIPS & Tricks

Using Templates in OIM™ Analysis

Templates in OIM™ Analysis are definitions of specific analyses that can be used to quickly generate a predefined map, plot, or chart. A number of such templates are already pre-programmed in the "Quick Gen" toolbar.

Figure 1: Quick Generate toolbar.

The buttons are designed to give the user quick access to a number of standard analysis operations. When a Quick Gen template button is selected, it will only be applied to the active partition as indicated by the pull down menu (the "All data" partition of "sample 1" dataset is present).

The buttons that are greyed out can be used to activate a custom prepared template. Defining an analysis can generate a custom template, e.g. the blended EDS map with different types of boundaries below, and selecting the "export template" function in the contextual menu (right-click on the map).

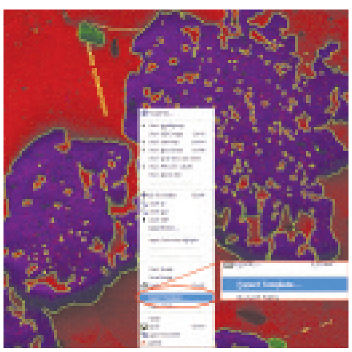


Figure 2: Generating a template for a map.

Templates that are generated this way may be stored anywhere on the hard disk, but a template folder that already contains a number of useful templates is provided in the OIM™ Analysis folder (C:\Program Files\TexSEM\OIM Analysis 5\Templates).

To apply a custom template to a single partition only, right click on the red partition icon and select "Apply Template". You must specify which type of template you wish to use. If you want to apply a template simultaneously to all datasets or partitions in an analysis project, go to the project icon and select the apply template function there.

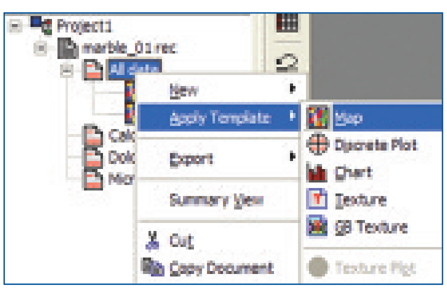


Figure 3: Template function.

Alternatively, if you wish to use a template often, you may program it behind one of the greyed-out buttons in the Quick Gen toolbar. In the settings _ preferences dialog is a section where up to eight templates may be selected, two for maps, plots, charts, and partitions.

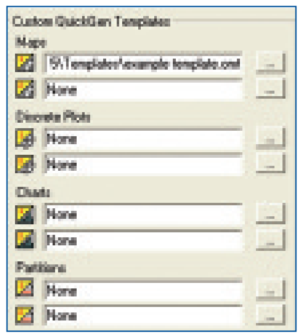


Figure 4: Custom Quick-Gen template section in the OIM™ Analysis preferences.

If you only want to apply a defined map, plot, or chart to another partition within the same project, there is no need to create a template first. You can simply select the copy document function in the contextual menu of a map, plot, chart, or texture, and paste the selected analysis into another partition. Similarly you can copy an entire partition, including any partition formulas and analyses, into another dataset.

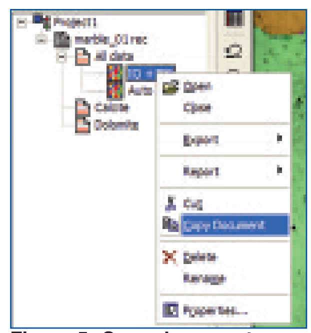


Figure 5: Copy document dialog in the OIM™ Analysis project tree.

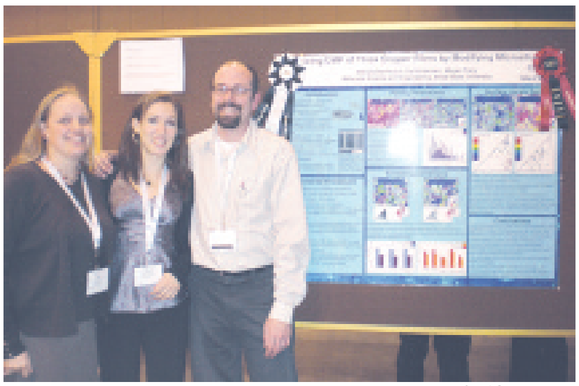
Templates can also be used to automate the analysis of a number of scan datasets using the OIM™ Analysis batch processor.

Figure 6: Batch processor button.

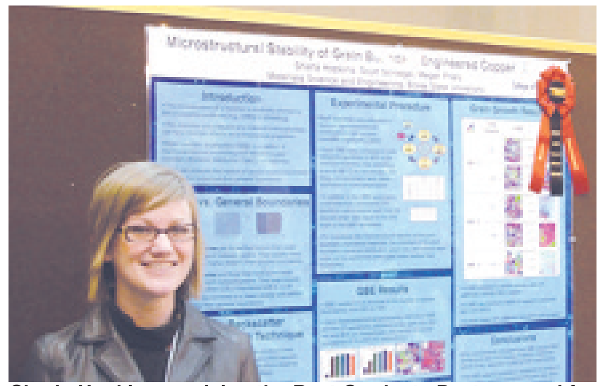
The batch processor allows the user to perform cleanup and rotate operations to selected OIM™ scan files, followed by automatic generation of OIM™ Analysis project files for each dataset. For each project file, all the analyses, as defined by a dataset template, can also be exported as images to the hard disk for easy report generation.

Award Winning Research

Congratulations to Megan Frary's group at Boise State University located in Boise, ID for their award winning OIM related research. Sharla Hopkins won the Best Undergraduate Poster award from the MPM Division and Mariela Bentancur won both the Best Undergraduate Poster award from the EMPM Division and Best of Show at the TMS meeting and exhibition in New Orleans, LA in March 2008. Dr. Frary has also received a teaching award from ASM International as well as the National Science Foundation's (NSF) award for early CAREER faculty. These awards are well deserved and recognize the hard work of Dr. Frary's students.

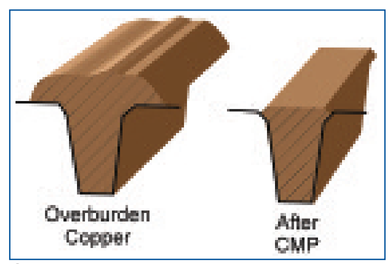


Mariela Bentancur with her co-authors M. Frary (left) and P. Andersen (right) receiving the Best of Show and Best Undergraduate awards for her poster entitled "Optimizing CMP of Thick Copper Films by Modifying Microstrucure".



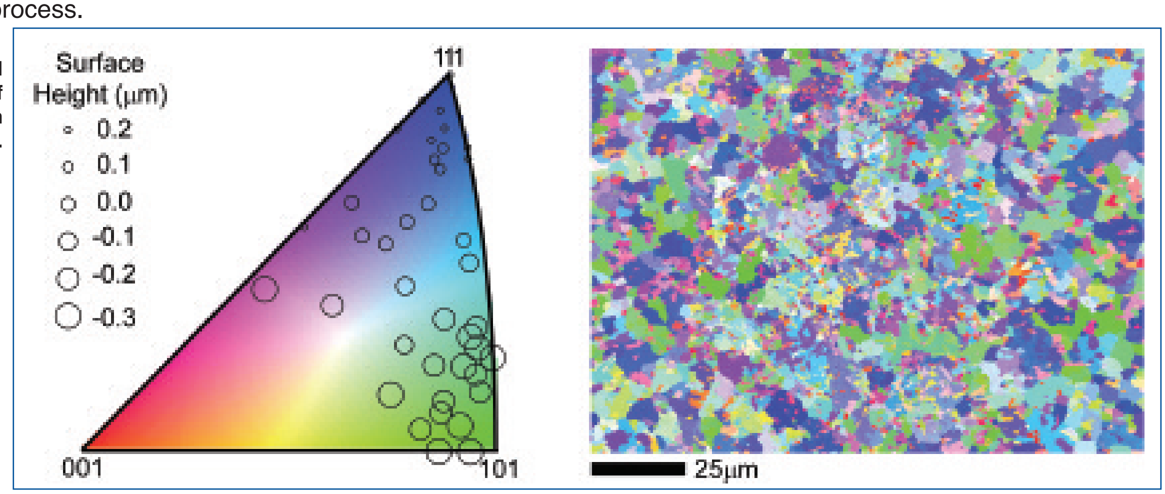
Sharla Hopkins receiving the Best Graduate Poster award for her poster entitled "Microstructural Stability of Grain Boundry Engineered Copper" co-authored by S. Schlegel and M. Frary.

Mariela's work focused on understanding the impact of chemical mechanical planarization (CMP) on electroplated copper microstructures. CMP is a critical step in the damascene process used in the production of copper interconnect lines in multilayered microelectronic structures. In this process, silicon oxide is patterned with open trenches. The patterned insulating underlayer is coated with copper, which overfills the trenches. CMP removes the overburden copper to the level of the top of the insulating layer. The copper in the trenches becomes the interconnects. Mariela's work showed the impact of crystal orientation on CMP. After CMP the height of the individual grains is measured using an optical profiler and orientation by OIM. It was observed that when a polarizer is added during the plating process, the <111> oriented grains were consistently "taller" indicating their resistance to the CMP process.



Copper Damascene Process

Orientation map and optical profiler results after CMP of electroplated copper with an added polarizer.



TRAINING AND EVENTS

World-Wide Events

February 16-18	TMS (Minerals, Metals & Materials Society Meeting)	San Francisco, CA
February 16-21	AAFS (American Academy of Forensic Sciences)	Denver, CO
March 12	AIMS (Arizona Imaging & Microanalysis Society)	Arizona
March 9-13	Pittcon	Chicago, IL
March 18-20	SEMPA (FEI SEM Users)	Grenoble, France
March 30-31	EBSD Meeting	Swansea Wales, UK
April 20-23	MAS Spring 2009 (Microanalysis of Particles II)	Westmont, IL
May 1-2	NESM (New England Society for Microscopy)	Woods Hole, MA
May 4-7	Scanning	Monterey, CA
May 10-14	EMAS 2009	Gdansk, Poland
May 28-29	(SEMS) The Southeastern Microscopy Society	Athens, GA
May 31 - June 12	Lehigh School	Bethlehem, PA
June 8-10	Scandem	Reykjavik, Iceland
July 27-30	M&M (Microscopy & Microanalysis)	Richmond, VA

***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):

Microanalysis Courses: 3-4 Day
- January 27-30 (W)
- February 2-5 (T)
- June 22-25 (T)
- September 28 - October 1 (T)
- November 17-26 (W)
- November 23-26 (T)

2 Day
- March 12-13 (T)
- June 18-19 (T)
- September 17-18 (T)
- November 12-13 (T)

LEX Course: 3 Day
- March 31- April 2 (T)
- October 13-15 (T)

TEX Course: 2 Day
- June 23-24 (W)

Pegasus Courses:
- February 9-13 (W)
- March 9-13 (T)
- May 11-15 (W)
- June 15-19 (T)
- September 14-18 (T)
- October 19-23 (W)

OIM™ (EBSD) Courses:
- January 26-28 (T)
- March 9-11 (T)
- June 15-17 (T)
- September 14-16 (T)

Japan

Microanalysis Courses:

February 4-6	Tokyo
April 8-10	Osaka
June 3-5	Tokyo
July 1-3	Osaka
October 7-9	Tokyo
November 11-13	Osaka

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

North America

Microanalysis Courses:

March 3-5	Draper, UT
March 16-20	Mahwah, NJ
April 27 - May 1	Mahwah, NJ
June 22-26	Mahwah, NJ
July 14-16	Draper, UT
August 10-14	Mahwah, NJ
September 14-18	Mahwah, NJ
October 19-23	Mahwah, NJ
December 7-11	Mahwah, NJ

Particle Course:

May 12-14	Mahwah, NJ
November 10-12	Mahwah, NJ

EBSD OIM™ Academy Course:

February 3-5	Draper, UT
April 28-30	Mahwah, NJ
September 1-3	Mahwah, NJ
October 13-15	Draper, UT

Micro-XRF Course: Mahwah, NJ
- April 14-16 (Eagle/Orbis)
- October 6-8 (Orbis only)

Debora DeRosa began her career with EDAX in September 1996 as a mail clerk for Philips Electronics Instruments (P.E.I.) in Mahwah, NJ. In 1998, she joined the Electronic assembly group. Debora's hard work, quality workmanship and dedication allowed her to move into the Detecting Unit (DU) Production department as a DU Specialist in 1999. In 2004, Debora was promoted to DU Externals Specialist where she excels in her position to this day.

In her current position, Debora has become the expert for all vital information pertaining to detectors in process and the history of all shipped detectors. Throughout the organization, departments rely on Debora for specific information relating to a customer's order (model numbers, shipping dates, accessories, etc.). Debora is also the chief assembler of the Wavelength Dispersive Spectrometer (WDS) product and comes in contact with nearly all the detecting units that ship from this facility.

Over the course of her career Debora has received many accolades, including Instant Quality Recognition Awards. In 2003, Debora was the winner of the Presidents Award for Excellence, nominated by her peers throughout the organization.

Debora is the proud parent of her daughter Deanna, who has just turned sixteen. In her spare time she likes exercising, power walking, and dancing. Debora also enjoys doing home improvements on her own. She recently upgraded a bathroom and kitchen, doing the tiling and cabinets herself.

Yoshiko Koda joined the Philips IE Division in Japan in April 1989. During this time EDAX was part of Philips, and she was assigned to EDAX. Prior to joining EDAX, Koda-san had received a degree in Cultural Science at Seikei University.

Koda-san has been in the Service Department throughout her career with EDAX. Thanks to Koda-san and her loyalty to both EDAX and its customers, Japan EDAX has the reputation of having the very best customer support in the business.

Koda-san handles various tasks and is truly multi-talented. Her people skills are excellent and she is therefore very much liked by both our customers and her colleagues within the EDAX Japan team. Koda-san ensures the service engineers are prepared when making customer calls and also handles service invoicing and contracts, parts requirements, as well as bookkeeping. For many end users, as well as, electron microscope manufacturers, she is the "voice of EDAX" when they call in for support. In Japan, EDAX has over 1,500 customers. All of them will eventually speak with Koda-san. As anyone can imagine, she spends a lot of time on the telephone. Each call is given Koda-san's undivided attention.

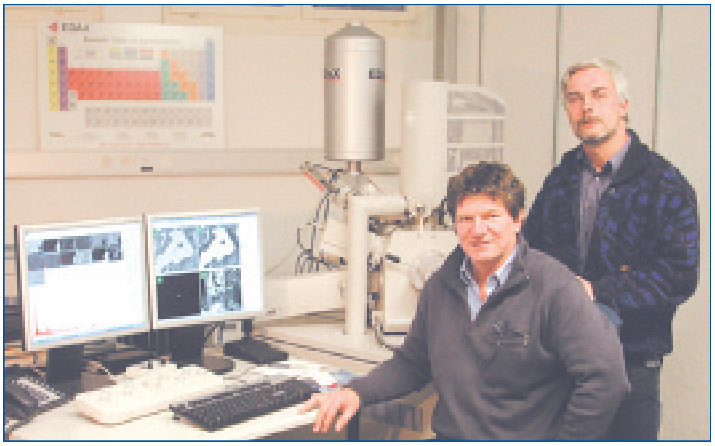
Outside of work, Koda-san enjoys skiing and racing in her new Audi Quatro, a recent present from her husband.

Electron Microscopy ETH Zurich, Switzerland

The Eidgenössische Technische Hochschule (ETH) Zürich is the Swiss Federal Institute of Technology Zurich and one of the two federal science and technology universities in Switzerland. As an internationally oriented institution of higher education ETH Zurich provides the study, research and the work place of 18,000 people from 80 nations.

The federal institute runs the Electron Microscopy ETH Zurich (EMEZ). This center provides research facilities and scientific and technical support in electron microscopy to researchers in departments and laboratories across all disciplines at ETH and its partner institutions. EMEZ currently has more than 120 trained EM users. Their equipment consists of six TEM, five SEM, and one FIB-SEM. Material scientists make intensive use of the two 300kV TEM for investigations on nanoparticles, semiconductors, ceramics, catalysts etc. Research in life science is focused on developments and applications of cryo-preparation and cryo-microscopy covering the cellular and the molecular scales. Dr. Roger Wepf, head of EMEZ, sees great potential for better understanding complex biological systems in combining information obtained at different scales and imaging modes. He uses integrated approaches for correlative microscopy as well as for enhancing synergies between different disciplines to satisfy their EM requirements. For additional information visit www.emez.ethz.ch.

One TEM (F30 Tecnai), two SEM (Quanta 200F and CamScan CS44LB), as well as one FIB (NVision 40) are all equipped with EDAX systems for EDX and/or EBSD analysis, mostly applied to study elemental and microstructural distributions in a diversity of materials including metals, ceramics, rocks, concrete or identifying nano-particles in man-made respectively biological material. “It has been a durable partnership with EDAX”, Dr. Karsten Kunze, Senior Researcher at EMEZ, states. He very much appreciates the professionalism and individual support by EDAX engineers and the upgrade policy at EDAX, which helps the center to maintain equivalent software levels on the various systems, thus simplifying user training and servicing significantly. Karsten’s ties, with what is now part of EDAX, go back to his years as a post-doc in the research team of Professor Brent Adams and Dr. Stuart Wright, who developed the first fully automated system for Orientation Imaging Microscopy (OIM™). Repeated mutual visits with exchange of feedback and ideas have helped incorporate further developments and improvements particularly to the OIM™ suite of products. Current research projects require particularly fast orientation mapping for studying crystallographic control of crack propagation, high resolution mapping with minimum drift to analyze textures in thin nanocrystalline films, FIB slice and map to identify deformation mechanisms in compressed nanopillars, and close integration of EDX with EBSD for comprehensive fabric characterization of polymineralic rocks recording processes of deformation, recrystallization and mineral reactions.



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