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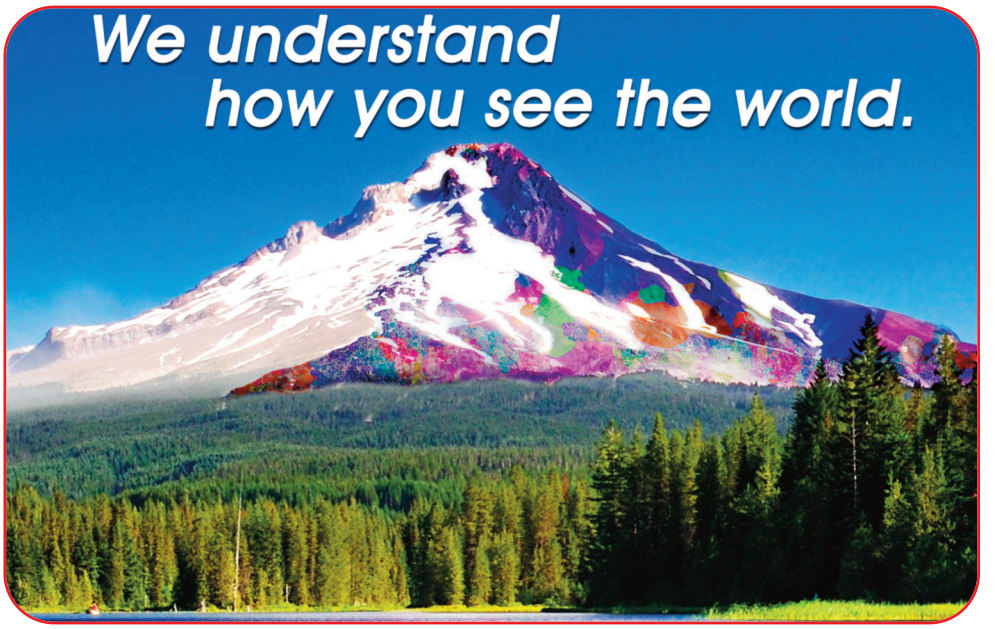

EDAX NEWS

Introducing the XLNCE SMX-ILH X-ray Fluorescence (XRF) Analyzer

The SMX-ILH System is the second XRF analyzer to be introduced in the XLNCE product series, after the SMX-BEN benchtop unit. The SMX-ILH system is an in-line process metrology tool capable of integration into a production conveyor system or manual loading. As with the SMX-BEN, the SMX-ILH analyzer measures thickness and composition of simple to complex multi-layer coatings and metal treatments, as well as composition of bulk materials. Panel substrates as large as 1.1 by 1.4 m can be loaded into the system or coiled substrates as wide as 1 m can be streamed through the system. The system can be configured with either side to side entry/exit ports or with a single entry/exit port as dictated by the floorplan of the installation site.

Figure 1. XLNCE SMX-ILH XRF Analyzer.

The X-ray measuring head of the benchtop unit is also incorporated into the SMX-ILH system. This provides motorized collimators and primary beam filters as needed to measure many types of coatings and materials. The head includes constant view video optics, allowing "what you see is what you get" imaging of the material being analyzed. Process materials up to 300°C can be measured when the measuring head is outfitted with a patented heat shield to prevent measurement drift due to the heat emanating from the hot material. Laser auto-positioning is used to maintain distance to the sample.

(Continued from Page 1)

For streaming substrates where the target position may fluctuate with the underlying roller, a spectral ratioing method can be used. In the plot in Figure 2, the SMX-ILH was used to measure two different Ni plating thicknesses. The measuring head moves back and forth between the two different parts, sets the measuring distance using the laser auto-positioner and makes the measurement. Measurements were made over the course of 60 hours with precision better than 0.3% relative standard deviation (RSD).

The SMX-ILH and SMX-BEN analyzers have been used to measure a wide variety of coatings and materials. XRF is typically used to measure non-organic coatings. This would include CIGS and CdTe photovoltaic materials, Zn-coated steel and a wide variety of oxide coatings (Ti, Zr, etc.) on aluminum. The SMX-ILH has even been used to measure silicon coatings on advanced battery anodes at normal atmospheric pressure. Coatings are typically measured by direct detection of the elements in the composition. However, the advanced coating software used in SMX-ILH systems also allows for measuring Al coatings in air by measuring the absorption of the underlying substrate signal and more complex aluminum treatments by stoichiometry of the detected elements (e.g. Ti) to the undetected elements (e.g. elements lighter than Na).

In a process metrology environment, the software GUI must allow simple operation, while securing various system setups and calibrations from unauthorized changes. The SMX-ILH software GUI, known as Mira, is a recipe driven instrument control package. Mira software provides three different password protected levels, Operator, Supervisor and Maintenance, to prevent less experienced users from changing existing measurement routines. The Supervisor can create and calibrate measurement applications, as well as create tables of measurement positions to build a full recipe. The general Operator can load measurement recipes and collect data. The Maintenance level provides access to various additional system diagnostics and setups. In addition, the SMX-ILH offers remote operation by a programmable logic controller (PLC) managed interface or open platform communications (OPC) standard protocols. All measurement data are available for report generation in the Mira software, as well as being output to a structured query language (SQL) database, which is then accessible to the production facility's manufacturing execution system (MES).

The SMX-ILH process metrology system offers flexibility for integration into production environments, as well as powerful analytical software capable of measuring a wide variety of coating systems, treatments and materials. The mechanical design allows for manual loading, streaming coiled substrates or integration into a production conveyor system. The SMX-ILH can be operated by a human operator or controlled via the factory's MES system. The control interface is recipe driven to facilitate simple operation and is accessible via password protected levels to prevent unapproved changes and operator error. All of these features make the SMX-ILH System one of the most versatile XRF process metrology tools available on the market.

Click here to find out more information about the EDAX XLNCE SMX-ILH In-Line Analyzer.

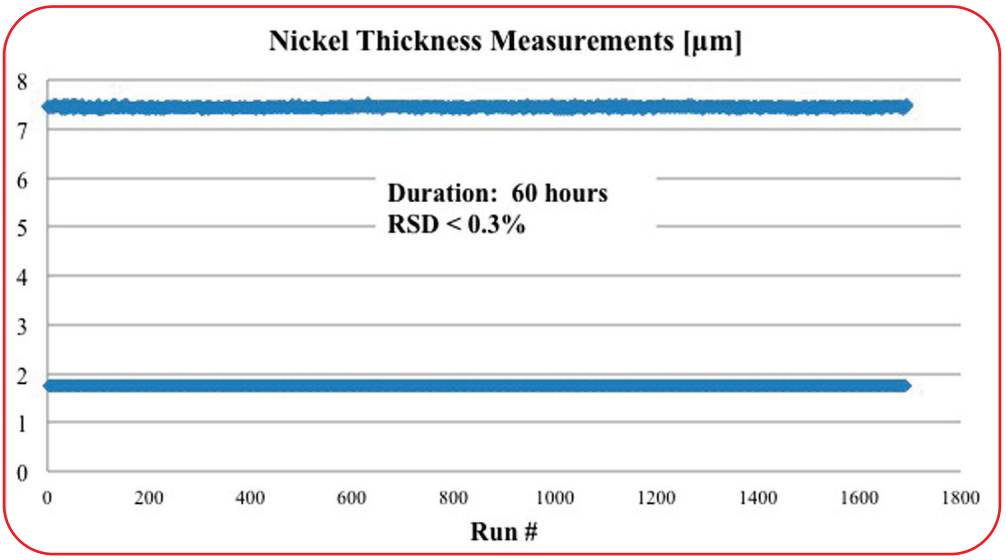

Figure 2. Measurements of two different Ni plating thicknesses using the SMX-ILH over the course of 60 hours. The plot shows precision better than 0.3% RSD.

Energy Dispersive Spectroscopy (EDS) Calibration in TEAM™

When environmental conditions (e.g. temperature, humidity) are controlled within narrow limits, EDS detectors are very robust and calibration hardly shifts over very long time periods.

Calibration of an EDAX EDS detector is done to adjust the energy scale for the EDS spectrum, making sure PeakID does a perfect job identifying elements correctly. This is the only calibration that a customer might have to do. No calibration is needed for EDS quantification, as this is done standardless by default.

Calibration is needed when either PeakID is incorrect (for instance, if FeKα is misidentified as CoKα) or when the fitted spectrum (Halographic Peak Deconvolution (HPD) trace) does not exactly match the peaks. In some specific cases, there might be a little shift, which is caused by elements overlapping in EDS (Figures 1 and 2).

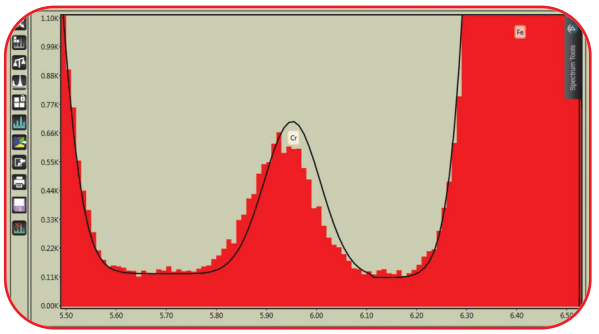

Figure 1. A steel sample with only Cr identified (HPD misfit).

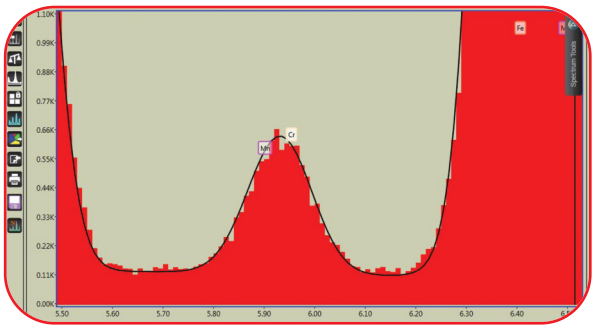

Figure 2. Same steel sample with CrKβ and MnKα (overlapping) identified (perfect HPD fit).

Calibration is done using an Al+Cu sample. This is because copper tape is usually present in an electron microscopy lab and the stub material is Al. The big advantage of using Al and Cu is that the AlKα and CuKα peaks are far apart and will give a very accurate calibration.

Procedure

- Put the Al+Cu sample in the microscope and pump down.
- Set the high voltage to 25 or 30 kV.
- Use > 500x magnification and spot size and/or aperture to get count rates around 10,000-20,000 cps.
- Make AlKα and CuKα peaks roughly the same height.
- Go to Advanced Properties and select Spectrum Calibration. Check that Peak 1 (AlKα) is set to 1487 eV and Peak 2 (CuKα) to 8041 eV.
- Set the number of counts to 10,000-20,000.
- Choose the amp time and press Start (Figure 3).

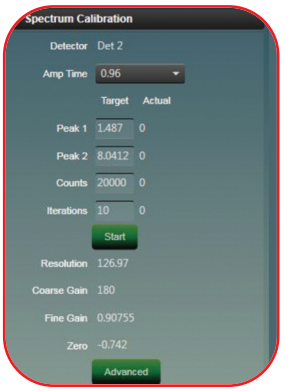

Figure 3. Calibration panel in TEAM™.

Calibration should typically be finished using 2-3 iterations. Do this for all seven amp times. If it is not calibrated after 10 iterations, another 10 can be run.

Resolution at MnKα (peak width at half of peak height, called the Full Width Half Max (FWHM) value) is calculated from AlKα and CuKα peak width values. When checked by measuring MnKα, one often sees resolution is even a bit better than this calculated value.

Calibrations done in TEAM™ are saved in a logging file under c:/ProgramData/EDAX called calibrat.csv. It is good to check calibration every few weeks.

When a peak is seen at the very beginning of an EDS spectrum (often misidentified as BKα), it means the BLM (noise level) has not been adjusted properly. To solve this, go to the calibration section and click on the advanced button. Increase the BLM using small steps (increments of 1-2) until this peak is gone.

Figure 4 shows the BLM set incorrectly (at 18, in cyan) and the BLM being set correctly (at 23, in red). Note that the BLM is different for every amp time.

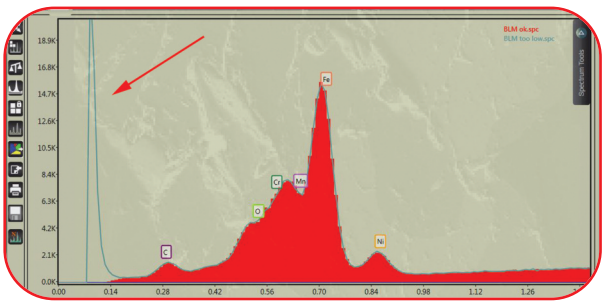

Figure 4. BLM set incorrectly (cyan) and set correctly (red).

EDAX Smart Insight

APPLICATION NOTE

Analyzing Absorption of Wood Preservatives Using Micro X-ray Fluorescence (Micro-XRF)

Figure 1. Example of untreated wood with "dry rot".

Wood is often chemically treated to preserve it from physical degradation and maintain its structural integrity against natural elements like fungus, insects, and moisture. The concept and practice of lumber treatment has been used for millennia. Early examples include the ancient Greeks soaking wood in olive oil, and the Romans brushing wood with tar to protect and prolong the life of the material. In modern lumber treatments, there are numerous processes and preservative chemicals that can be used, which are typically characterized by the solvent used to carry the preservative into the wood, i.e. water, oil, or light-organic solvents. Many preservatives, particularly those which are water-borne, utilize copper as a key constituent. They are usually dissolved into a solution using chemical reactions, but have also recently been utilized by suspending micronized copper particles in an aqueous solution. These various preservatives can be applied to the wood using a number of different processes, such as steeping, brushing, pressure-assisted methods, or even integrating the preservative into the live plant's sap stream. The end goal of these processes is to create a deep and uniform absorption of the preservatives into the wood to ensure the effectiveness is maximized.[1]

In this example, a sample of 4" x 4" wood was treated with a copper-based chemical on its surface (Figure 1). The sample was then cross-sectioned against the grain into a slice approximately one-half inch thick. The goal of this analysis was to assess how deep the copper-based solution had been absorbed into the wood by measuring copper using Micro-XRF. Because the treatment is not visible to the naked eye (i.e. there is no discoloration), spectroscopic methods relying on the visible light spectrum are not useful. The absorption depth can be easily determined by Micro-XRF performing an elemental spectrum map of the cross-section, looking specifically for copper. Micro-XRF utilizes a micro-focused X-ray beam to generate characteristic X-ray energy lines, similar to that used in Energy Dispersive Spectroscopy (EDS). However, Micro-XRF is a non-destructive measurement technique with superior sensitivity for higher-energy elements such as copper. Detection limits for copper are nominally < 10 ppm for a single point measurement. In addition, Micro-XRF generally requires very minimal sample preparation and operates under low vacuum, whereas other techniques may require extensive sample preparation, particularly for organic or biological materials.

For this analysis, the EDAX Orbis PC Analyzer was used. The Orbis PC was ideal for this sample characterization because the X-rays were focused to nominally 30 μm in diameter using a mono-lithic hollow glass fiber bundle (a.k.a. poly-capillary optic). The scale of features on a 4" x 4" cross-section of wood cannot be fully imaged using EDS, as the beam diameter is too small to cover such a large area. Conversely, some "bulk" XRF systems have smaller apertures on the order of a few millimeters, which is too large and not capable of generating higher resolution images. The 30 μm beam diameter on the Orbis was the right size to get high image resolution, while also being capable of mapping a larger area in a relatively short period of time.

To optimize the mapping collection parameters, it is important to factor in the size of the mapping area, the beam diameter, and the desired beam spacing. For this sample, the area being mapped is shown in a red outline in Figure 2, approximately 50.4 x 3.5 mm.

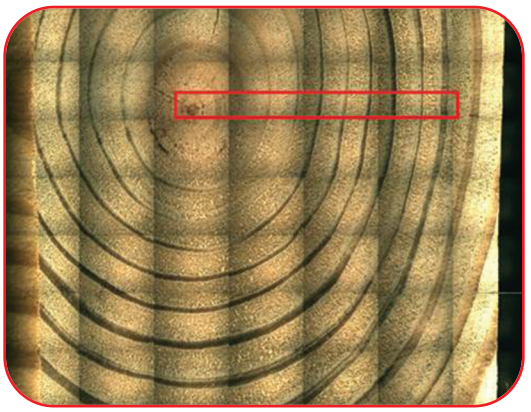

Figure 2. Montage image of wood cross-section sample, with mapping area highlighted in red. The wood sample has been treated with a copper micro-particle preservative.

(Continued from Page 4)

The narrow area minimizes extraneous mapping time, but will still give a clear profile of the copper signal as a function of distance. The X/Y matrix, or the number of points collected in each axis, was 420 x 35 points, giving approximately one beam space (~30 μm) in between each collection point. Dwell time (per point) should be determined by the composition of the elements of interest, as trace elements require longer dwell times than major elements. However, it is important to remember that overall sensitivity degrades substantially when mapping because the acquisition time is much shorter compared to a longer single-point analysis. Using a dwell time of 500 msec per point, the total collection time was approximately two hours.

The resulting images show the video image of the mapped area (Figure 3a), along with the imaged Cu (K) intensities displayed with thermal color scaling (Figure 3b). As expected, the Cu (K) was most intense near the outer edge of the wood with a maximum intensity of nominally 21,000 counts per second.

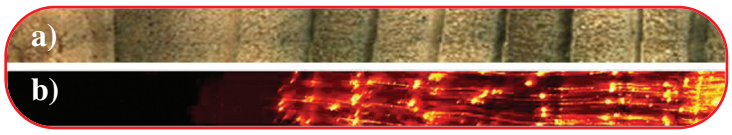

Figure 3. (a) Video image of the mapped area and (b) the Cu (K) spectral map in thermal scaling.

The distribution of copper was interesting because it did not show a smooth uniform distribution throughout the wood. Instead, Cu (K) "hot spots" were clearly evident, along with streaking normal to the rings of the wood. The hot spots tended to form right before (to the left) of the next tree ring, which is best shown in the total counts map in Figure 4.

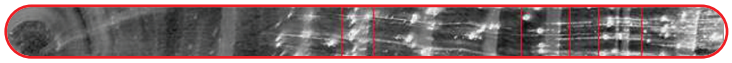

Figure 4. The total counts map does not separate maps by energy, but instead represents the total countrate at any given point. This clearly shows that the copper hot spots do not directly correlate with the tree rings.

The streaking patterns all appeared to be against the grain, and instead of gradually decreasing towards the center, there was a relatively abrupt drop in Cu (K) intensities near the third and fourth ring from center. Beyond that point, the copper drops to trace levels, and at that point became dependent on the sensitivity of the instrument. Overall, it appeared that the absorption of copper was relatively deep, but not uniform. Absorption appeared to have been successful through the outer eight tree rings (out of about eleven), or approximately 2.8 mm.

The Orbis PC Micro-XRF Analyzer was used to provide valuable information on the distribution of copper micro-particles absorbed into an unprocessed piece of wood. The copper preservative was absorbed relatively deep into the wood showing a pattern of absorption which appears to be dependent on the structure of the wood. Measurements using the Orbis PC Micro-XRF Analyzer were non-destructive, which preserved the sample for measurements using other techniques, and required minimal sample preparation. Other elemental measurement techniques typically require much more work to prepare the sample for analysis, particularly for biological and organic samples.

Reference

1 Quarles, S., Kobzina, J., Geisel, P., "Selecting Lumber and Lumber Substitutes For Outdoor Exposures." University of California, Division of Agriculture and Natural Resources, retrieved Jan. 11, 2016. http://anrcatalog.ucanr.edu/pdf/8144.pdf

EVENTS AND TRAINING

2016 Worldwide Events

April 19-20		**May 23-27**	
Forensics Europe Expo	London, United Kingdom	Asia-Pacific Microscopy Conference	Phuket, Thailand
April 26-29		**June 5-10**	
Control	Stuttgart, Germany	Lehigh School	Bethlehem, PA
April 29		**June 7-10**	
Minnesota Microscopy Society Spring Symposium	St. Paul, MN	Scandem	Trondheim, Norway
May 10-13		**June 14-16**	
Analytica	Münich, Germany	The Japanese Society of Microscopy	Sendai, Japan

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2016 Worldwide Training

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

EUROPE

EDS Microanalysis	
TEAM™ EDS	
April 11-13 May 31-June 2 June 13-15	Weiterstadt# Tilburg* Weiterstadt#
Microanalysis	
June 9-10	Tilburg*
Genesis	
June 27-29	Weiterstadt#
TEAM™ EBSD	
April 13-15 June 13-15	Weiterstadt# Tilburg*
TEAM™ Neptune (EDS & WDS)	
June 13-17	Weiterstadt#
TEAM™ Pegasus (EDS & EBSD)	
April 11-15	Weiterstadt#
TEAM™ WDS	
June 15-17	Weiterstadt#
XRF	
April 5-7	Tilburg*

*Presented in English
#Presented in German

JAPAN

EDS Microanalysis	
Genesis	
April 7-8	Osaka
TEAM™ EDS	
June 2-3 July 7-8	Tokyo Osaka
OIM™ School	
Basic	
June 21-22 July 14-15	Tokyo Osaka
Entry	
May 10-11 June 6-7	Tokyo Osaka
Practice	
April 21-22	Tokyo

CHINA

EDS Microanalysis	
TEAM™ EDS	
June 14-16	Shanghai

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
May 16-20 July 12-13	Mahwah, NJ Draper, UT
TEAM™ EBSD	
June 21-23	Mahwah, NJ
XRF	
April 5-7	Mahwah, NJ

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EMPLOYEE SPOTLIGHT

(left to right): Rudolf, Lucie, Beata and Adam Krentik.

Rudolf Krentik

Rudolf joined EDAX in December 2015. Based in the Czech Republic near Budweis, he is the key account manager for Central and Eastern Europe. Rudolf is responsible for direct sales and distributors in the region. He also handles EDAX's two largest key accounts in the Czech Republic, TESCAN and FEI.

Prior to EDAX, Rudolf was a sales representative for light and electron microscopy applications at Carl Zeiss Microscopy in the Czech Republic for almost 10 years. After high school, he moved to Prague to study mechanical engineering at Czech Technical University. His studies focused on biomaterials and their mechanical properties. Rudolf earned his master's degree in mechanical engineering in 2006.

Rudolf and his wife, Lucie have been married since 2011 and currently live in Pisek, Czech Republic. They have a daughter, Beata (four years) and a son, Adam (six months). In his spare time, Rudolf does a lot of work around the house. His last project was a wooden house and terrace on the garage. He enjoys road and mountain biking, snowboarding and sports in general. Rudolf likes exploring nature with his family and teaching his daughter how to ski and swim.

(front to back): Kaori, Keita, Fumiya and Takashi Matsumoto.

Takashi Matsumoto

Takashi joined EDAX, then Philips, in 1991. For the past 25 years, he has served as a field service engineer based in the Tokyo, Japan office. Takashi supports customers by performing installations, relocations, repairs, demos and customer training.

Takashi and his wife, Kaori have two boys, Fumiya (17) and Keita (13). Both of the boys play club basketball. In his spare time, Takashi enjoys competing in motorcycle gymkhana events in which riders compete for the best time on a paved course restricted by traffic cones or other obstacles. He also likes shopping in the DIY store and doing metal and wood machine work around his house, including milling, drilling, shaping by lathe, and welding.

At the end of 2015, Takashi performed a gunshot residue (GSR) demo at the Hitachi lab for the Tokyo metropolitan police scientific laboratory. The potential customer mentioned a special sample holder that they were currently using. Their common samples were 10 mm x 10 mm square long rubber sticks. They pasted double stick tape on the edge during sample collection. Afterwards, the customer cut the rubber to a reasonable size (about 2-3 mm thickness), then mounted it on a special holder that was provided by JEOL. The reason for the special holder was that the surface was tilted a little bit when a normal holder was used.

The customer said the special holder was not working perfectly and the surface was still a bit tilted. Takashi decided to take on the challenge of creating a new holder in his garage for the customer demo. With the new holder (Figure 1), he then tested the sample and confirmed that the tilted surface was no longer an issue after the sample was mounted. He is still brushing it up to get more accuracy in hopes of getting the order.

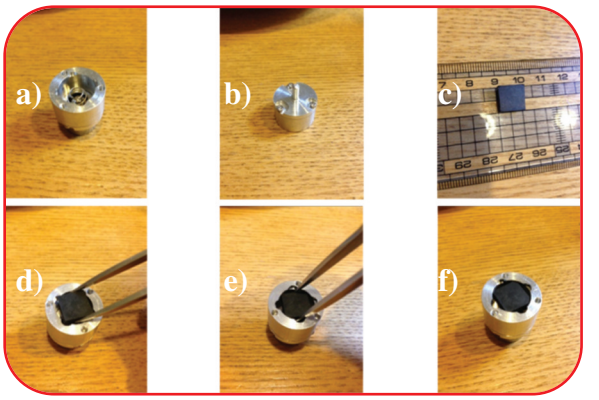

Figure 1. The holder created by Takashi. (a) Top, (b) bottom, (c) the sample, (d) placing the sample in the holder, (e) rotating the sample into place, and (f) the finished mounted sample.

EDAX Smart Insight

CUSTOMER NEWS

Materials Engineering Department at KU Leuven Belgium

The materials engineering department at KU Leuven performs research on the structure, properties, and processing of different types of materials, including: metals and alloys, ceramics, biomaterials, polymers, and composite materials. The lab uses electron microscopy to gain detailed structural and chemical information about materials. The department also offers scientific support for researchers in the materials science, mechanics, electronics, chemistry, physics, bioengineering, dentistry, and geology fields.

The department investigates complex materials systems, such as new metal- or polymer-composite materials incorporating nano-materials or new metal alloys. The laboratory is also interested in finding out about 3D structures produced from fine powders through laser melting, materials with grain structures reaching nanometer-scale, and heavily deformed materials.

Detailed microstructure and phase distribution information is needed to correlate the microstructural characteristics with the physical attributes of these materials to understand their mechanical, thermal, and electrical properties. The department has a significant modeling effort to predict these properties. The data obtained from the Scanning Electron Microscope (SEM), Energy Dispersive Spectroscopy (EDS) and Electron Backscatter Diffraction (EBSD) analysis represent the key parameters to validate these models.

The KU Leuven materials engineering department is currently using three TEAM™ Pegasus Analysis Systems to perform its analyses. The laboratory has a mix of Hikari and DigiView EBSD cameras and Octane and Apollo Silicon Drift Detectors (SDDs) for EDS analysis.

"We have been using analysis systems from EDAX for many years. , In our opinion they are very powerful, as well as user-friendly," said Tom Van der Donck, Core Facility Engineer Electron Microscopy.

Figure 1. Tom Van der Donck (left) and Gokula Krishna Muralidharan (right) in the laboratory next to a Nova Nanosem 450 with EDAX TEAM™ Pegasus Analysis System.

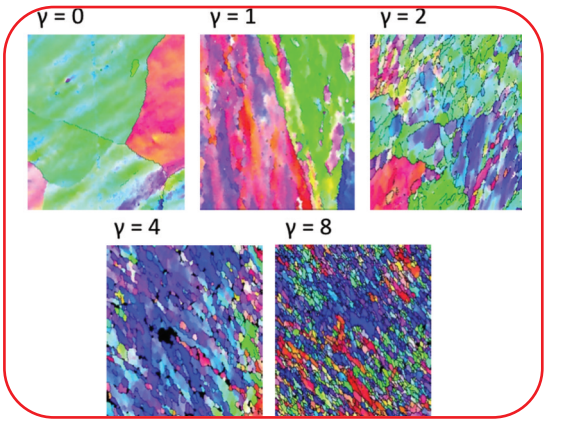

Figure 2. Microstructure of Al after different shear strains processed by ECAP.

"EBSD is one of our key tools. The EDAX EBSD performance played a key role in our system choice. The good communication, support, and service of the EDAX Tilburg office is also an important asset."

One of the biggest areas of interest for the department is studying deformation behavior of aluminum and other metals under severe processing conditions. The grain refinement is monitored after different levels of strain and the effect of the deformation mode and grain orientation on the microstructures and the refinement mechanisms are studied. The images in Figure 2 show the step-by-step evolution of fine grains in commercial pure Al from the annealed state up to shear of 800%, processed by Equal Channel Angular Pressing (ECAP). The grain size is reduced from an average of 50 μm to < 700 nm. The grains are initially elongated along the shear direction and later evolved into a more equiaxed structure at higher strains, aligning along the shear direction. Deformation behavior of metals at extreme strains is very unconventional with several non-equilibrium features contributing to the microstructural transformations. Several factors, such as crystal structure, orientation, alloying elements, etc. play an influential role in the evolution of the microstructures under such heavy deformation conditions.

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
Andrew Lee
Bruce Scruggs
Tom Van der Donck
Harry Verhulst