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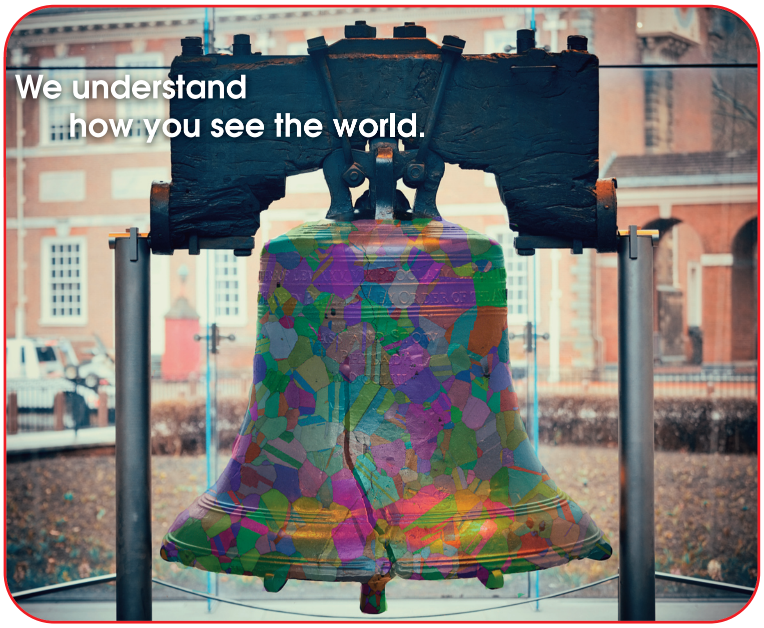
INSIDE



EDAX NEWS

Octane Elite and Monarc Come Together to Capture EDS and CL Simultaneously

Energy Dispersive Spectroscopy (EDS) in the Scanning Electron Microscope (SEM) is a key technique in analytical sciences used to determine the elemental composition of a sample with microscopic spatial resolution. EDS analyzes the energy distribution of X-rays emitted from a sample, from which the distribution of elements may be determined. X-rays form part of the electromagnetic spectrum, and in the SEM, a wide range of radiations from the electromagnetic spectrum may be emitted, including light—termed cathodoluminescence (CL)—at visible, ultraviolet, and infrared wavelengths (Figure 1).

Although the techniques of EDS and CL both measure photons from the electromagnetic spectrum, the information provided by each technique is distinct and complementary. EDS determines the elemental composition, whereas CL reveals crystal properties, such as optical and electronic properties. For example, CL has been used to reveal trace element (parts per million) distributions in minerals and cultural heritage items to help understand mineral formation and alteration processes and has been used extensively in compound semiconductor research (academic and industrial).

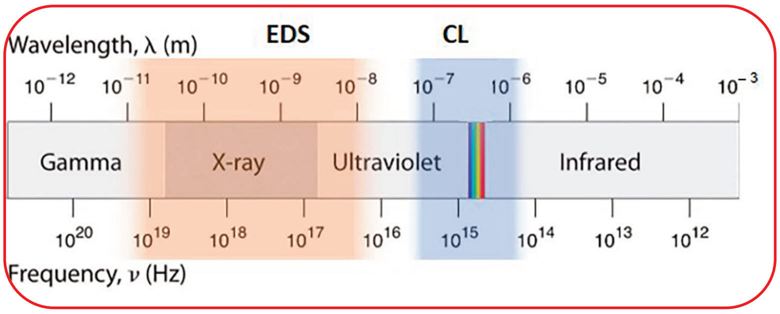


Figure 1. Electromagnetic spectrum highlighting the regions relevant to EDS and CL.

There are clear advantages to using both spectroscopic techniques to characterize a specimen. However, historically this has required EDS and CL signals to be captured sequentially due to the incompatibility of the detection hardware leading to extended data collection times, image registration complexity, and increased electron dose.

(Continued from Page 1)

However, this barrier to a streamlined workflow has been broken thanks to the integration of the Gatan Monarc® CL detector and EDAX EDS detectors, enabling the EDS, CL, and SEM signals to be captured simultaneously within the Gatan Microscopy Suite® software.

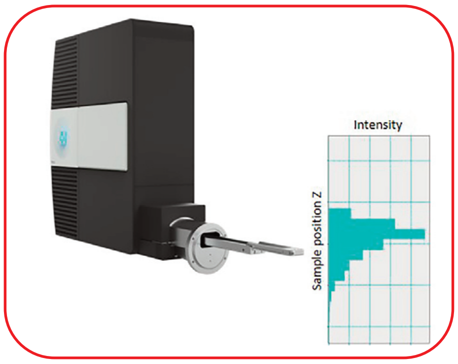


Figure 2. Monarc optimization of sample position.

Gatan's latest CL detector, Monarc, redefines what is possible in CL microscopy, boasting unsurpassed sensitivity for the sharpest CL maps and spectra in the shortest time and providing the most complete analysis of the emitted light (including by intensity, wavelength, polarization, and angle/direction—model dependent). Perhaps more importantly, it allows for the most accurate correlation with other signals, detecting multiple signals for morphology and composition matching, simultaneously. To collect the CL signal (light), a proprietary mirror (solid angle > 1.4π sr) is inserted between the specimen and the final electron lens of the SEM column collecting > 80 % of the signal emitted from a typical specimen. The objective is designed for use at the analytical working distance and can be configured to provide a line of sight to an EDS detector. The specimen's position beneath the CL collection mirror is paramount to optimizing light collection efficiency (Figure 2); Monarc includes ground-breaking optimization routines to automatically co-align the sample and optical system to the SEM's focus, guaranteeing optimal results.

To highlight the complementary nature of the CL and EDS data with a streamlined workflow, an example dataset was captured from a kyanite (Al_2SiO_5) thin-section. This was done using an Octane Elite EDS detector and Monarc CL detector with experimental control and data analysis performed using Gatan Microscopy Suite software. Kyanite was chosen due to its importance as a resource for the refractory industry and the ambiguity over the role that minor and trace elements play in determining the refractoriness. The EDS maps revealed a predominantly coarse-grained quartz-kyanite segregation with small amounts of staurolite (Figure 3). In the CL map, the kyanite grains exhibit significantly stronger luminescence than the quartz and staurolite grains, as well as reveal distinct intragrain zonation. CL spectra captured from the kyanite grains (Figure 4) exhibit an intense red luminescence that consists of a broad peak centered at 768 nm with narrow bands at 701 and 718 nm and weaker violet luminescence at 330 nm (outside the range shown in Figure 4). The red and violet luminescence peaks can be ascribed to trace levels of chromium and titanium respectively.

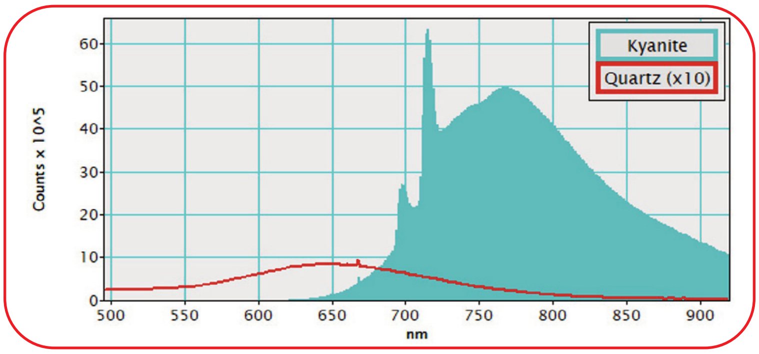


Figure 4. CL spectra extracted from kyanite and quartz regions. The quartz spectrum increased by a factor of 10 for comparison.

Through the combination of the EDS and CL signals, we are able to collect results that cannot be obtained by the use of each technique in isolation. We revealed three distinct mineral phases and discovered the distribution of Cr and Ti trace impurities within kyanite. The techniques of CL and EDS clearly complement one another, and the demonstration of simultaneous acquisition represents a remarkable benefit for specimen analysis. This is especially true in the case of electron beam-sensitive materials, where it is imperative to minimize exposure by capturing as much information as possible in parallel.

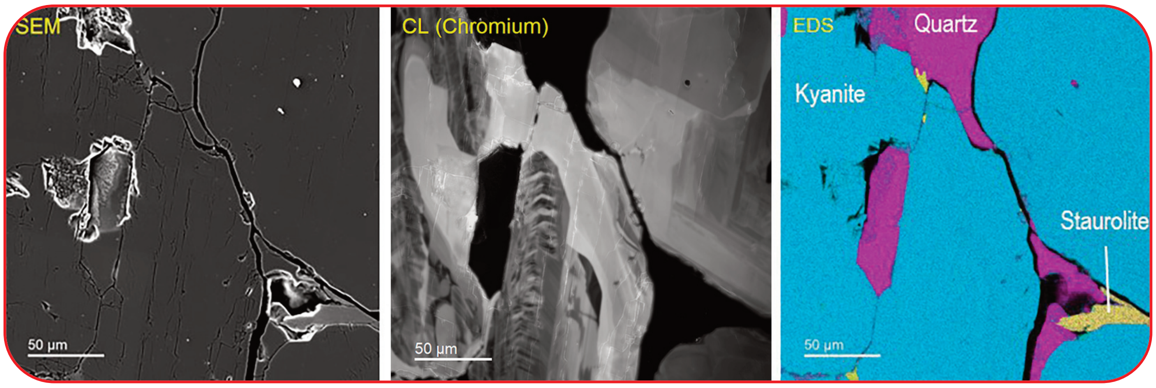


Figure 3. Co-acquired SEM (left), CL (center), and EDS (right) maps. EDS map indicates materials: quartz (magenta), kyanite (cyan), and staurolite (yellow).

TIPS AND TRICKS

Low Overvoltage Quantification with SCC

Energy Dispersive Spectroscopy (EDS) analysis of small structures or surface contaminants can be exceedingly challenging due to the X-ray signal generation volume, which is typically orders of magnitude larger than the resolution seen in the electron image. One way to reduce the X-ray volume is to reduce the acceleration voltage, however, this limits the number of X-ray lines available and makes quantification very challenging due to the large absorption corrections. Another approach is to use low overvoltage. Overvoltage is the ratio of the electron energy to the critical ionization energy of a given absorption line. A simulation of the signal depth for Si and Fe can be seen in Figure 1.

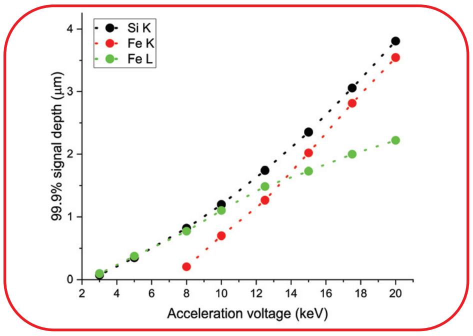


Figure 1: Simulation of signal depth as a function of acceleration voltage for Si K, Fe K, and Fe L lines using NIST DTSA-II[1]. The figure shows that the X-ray depth resolution is better using the Fe K line relative to the low energy Fe L line from roughly 13 kV acceleration voltage down to the excitation energy of 7.113 keV.

While low overvoltage measurements limit the X-ray generation volume, quantification is exceedingly challenging since the ionization cross-section changes dramatically as the acceleration voltage approaches the critical ionization energy (Figure 2). Significant deviations from theoretical models are to be expected[2] and consequently, standard-less models frequently require an overvoltage of at least 1.5 before a given energy line can be used for quantification.

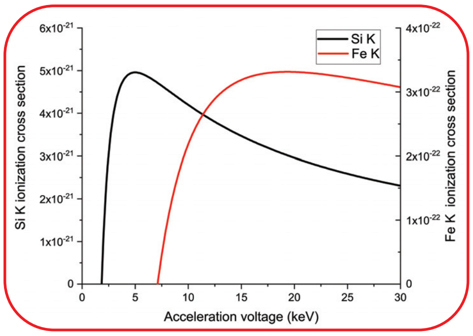
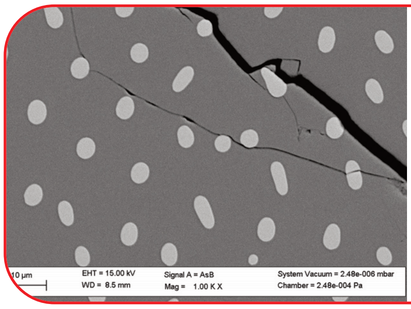


Figure 2: Calculated ionization cross-section of Si K with an ionization energy of 1.839 keV and Fe K with an ionization energy of 7.113 keV[3].

However, by using the Standard Customized Coefficients (SCC) option available in the APEX™ 2.0 software, it is possible to measure and compensate for deviations between the implemented model and the actual ionization cross-section for a given element and voltage combination.

To illustrate the improvements with SCC, an iron silicide standard with two distinct phases was examined (Figure 3). A pure Si sample was used as a reference sample at different voltages, while a pure Fe sample was used to determine the Fe K SCC values. Spectra were collected in a range from 9 - 20 kV corresponding to an overvoltage range of 1.1 to 2.8.



	Si	Fe
Main matrix	53.34	46.66
Bright spots	33.68	66.32

Figure 3: Backscatter image of an iron silicide standard with composition in weight %.

The quantification results in Figure 4 show a significant overestimation of Fe as the acceleration voltage approaches the Fe K ionization energy. By applying SCC measurements from a pure Fe sample, the deviations from implemented models can be compensated for and satisfactory quantification results can be achieved.

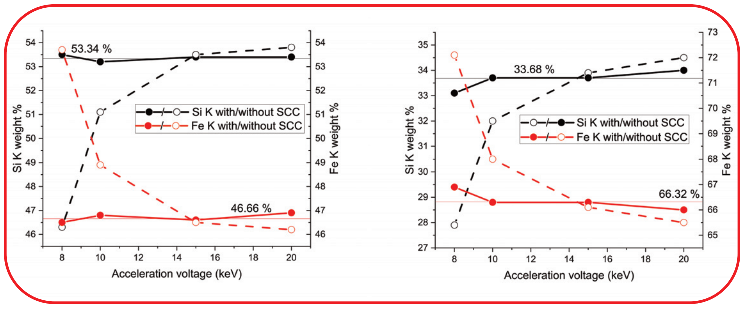


Figure 4: Quantification results with/without SCC for the main matrix (left) and bright spots (right) shown in Figure 3. The certified compositions are indicated by black/red lines for Si/Fe.

The SCC adjustments allow the user to optimize the quantification routine and compensate for model deviations in specific samples or uncommon conditions. The example presented here demonstrates optimization using simple, pure element samples. For effects with no significant inter-element influence, the SCC measurements for a given element can be applied irrespective of composition.

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[1] Ritchie, N.W.M. (2019). NIST DTSA-II software, including tutorials. Available at https://cstl.nist.gov/div837/837.02/epq/dtsa2/

[2] Llovet et al., Journal of Physical and Chemical Reference Data 43, 013102 (2014)

[3] Goldstein et al., Scanning Electron Microscopy and X-Ray Microanalysis (2018). ISBN: 978-1-4939-6674-5. 10.1007/978-1-4939-6676-9.

APPLICATION NOTE

How Ion Milling Improves EBSD Specimen Preparation

The quality of Electron Backscatter Diffraction (EBSD) analysis largely depends on the characteristics of the detector system in combination with the pattern processing and indexing capabilities. However, sample preparation may be even more important to get good EBSD patterns. EBSD is critically dependent on the surface of the specimen under investigation. The EBSD signal needs a crystalline material where the electron beam interacts with the atomic arrangement to generate the diffraction patterns that we need to identify the phase and orientation of the crystals. This requirement puts a high demand on the specimen preparation, as we can only get a reliable measurement if the true crystal structure of the sample is continuous all the way to the surface. There should not be any distortion or damage to the crystal or a contamination layer on the surface that scatters the diffracted electrons to weaken or even destroy the diffraction patterns.

There are multiple methods to prepare EBSD specimens. In most cases, preparation begins with mechanically polishing the sample on a wheel, which is often followed by a vibratory polishing step. The goal of the polishing is to remove any distortion introduced during earlier steps in the preparation (sectioning/mounting/grinding). It is crucial to remove this distorted layer completely; otherwise, the EBSD analysis surface may show an artificial microstructure generated during cutting and grinding and not the true structure of the sample. This involves multiple grinding and polishing steps with successively smaller grain sizes until the undeformed region is exposed at the surface (Figure 1).

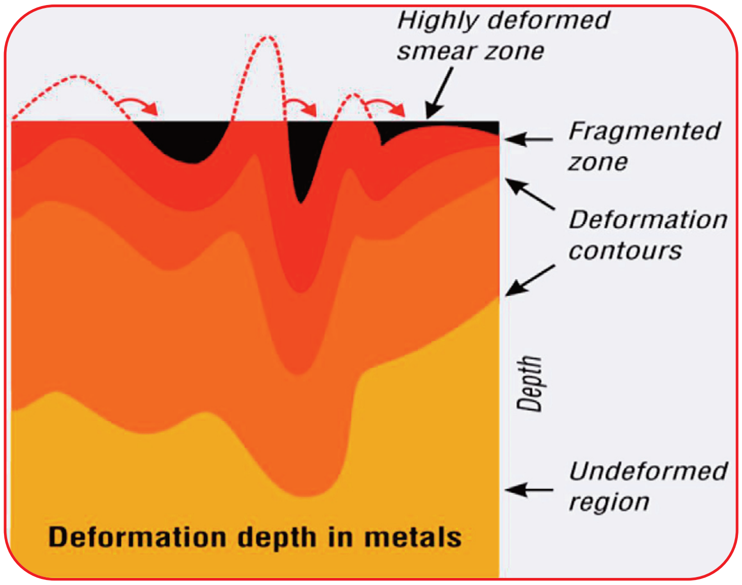

Figure 1. Diagram of the deformation depth required for EBSD analysis.

Alternative methods include electropolishing and chemical etching. Each method has its advantages and disadvantages, plus different materials might require a specific tool for optimum preparation for EBSD. But with all of these techniques, difficulties may arise with multiphase samples, where the individual phases respond very differently to the polishing technique. In other cases, samples may be so small, soft, or fragile that it is not possible to fully exclude any polishing artifacts from the final sample surface.

These issues can be overcome by ion milling with the Gatan PECS™ II for EBSD specimen preparation (Figure 2). The PECS II is a fully automated argon ion polishing system for SEM samples to prepare damage-free surfaces, cross-sections, and has the capability to deposit coatings to protect the surface or eliminate charging.

Figure 2. Gatan PECS II.

In the PECS II, a broad beam of Ar (or other) ions can be directed at the sample using a low incidence angle. The ions then sputter away the atoms at the surface of the EBSD sample to reveal the crystal structure underneath, while causing minimal topography (Figure 3). An important advantage of ion milling is that there is no mechanical interaction with the sample, so even extremely soft or fragile materials can be prepared.

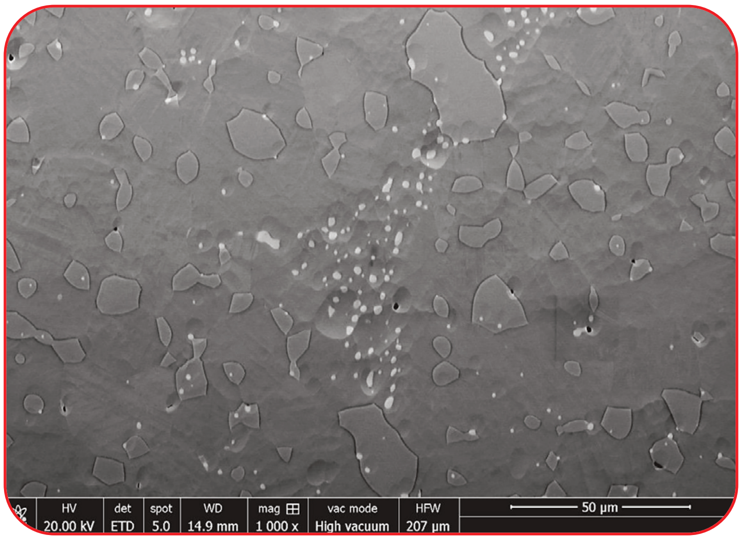

Figure 3. CoCrRe alloy sample after PECS surface preparation. Surface distortion is fully removed and minor surface topography does not coincide with grain boundaries, minimizing shadowing artifacts and allowing detailed EBSD grain boundary analysis.

(Continued from Page 4)

Polishing induced damage is often observed in mechanically polished Al alloys. The IPF map in Figure 4 shows intragranular deformation structures that were not fully removed by the grinding and polishing process. After ion milling, the true grain structure is visible.

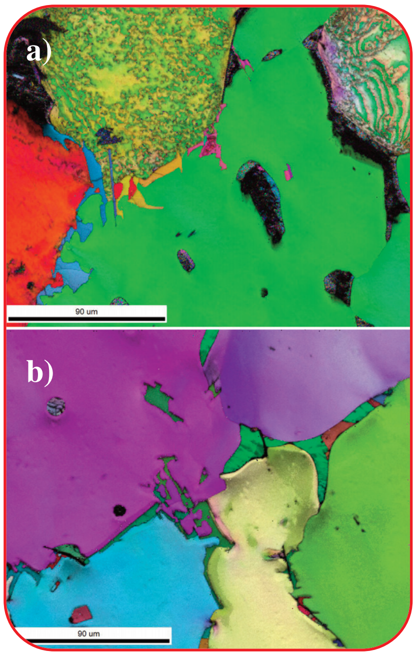

Figure 4. Al alloy EBSD maps a) after mechanical polishing and b) ion-milling collected with the Velocity Super EBSD camera.

You can adjust the sputtering characteristics of the ion mill by modifying the ion energy, current, and incident angle. As shown in Figure 5, the highest material-removing rates are achievable with incident angles of 4 – 10°. Using such a low incident angle ensures minimum ion implantation and damage to the crystal lattice.

This not only allows the preparation of soft or multiphase materials with very different polishing properties; it also allows quick removal of oxidation layers on, for example Mg and Zn alloys (Figure 6a - c).

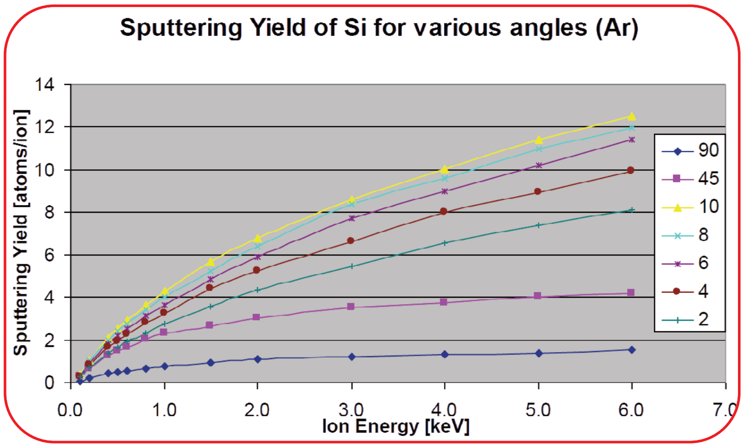

Figure 5. Chart showing the sputtering yield of silicon at various angles.

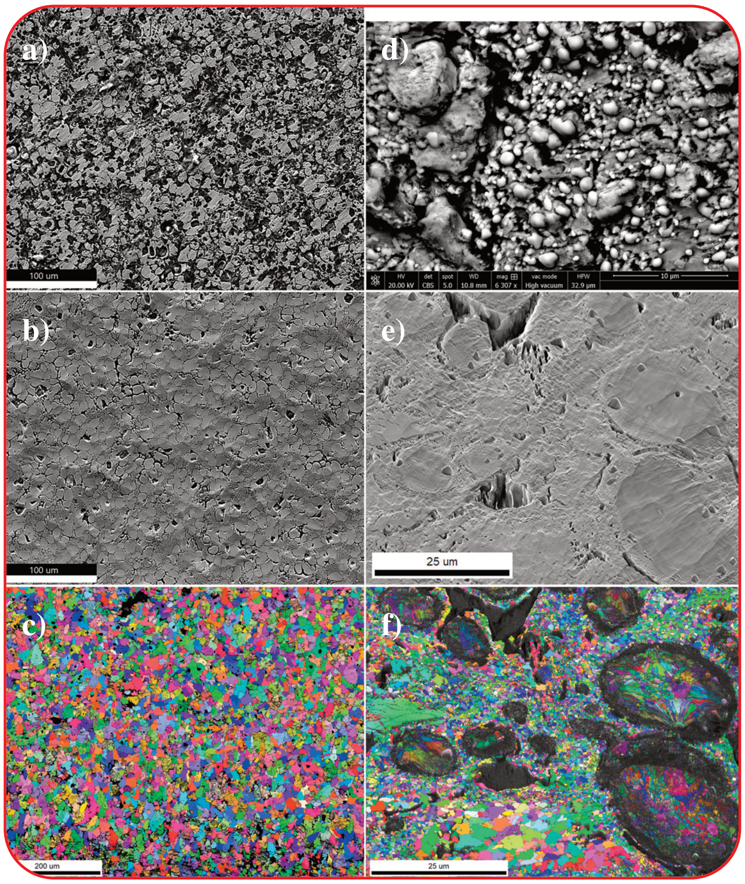

Figure 6. SEM images of an Al-Zn alloy a) before and b) after PECS milling. c) PECS milling fully removed the oxidation layer and enabled successful EBSD mapping of the entire surface area with the Velocity Super EBSD camera. SEM images of an AgHg alloy d) before and e) after PECS milling. f) IPF on IQ map of the milled area collected with the Hikari Super EBSD camera.

The example in Figure 6d - f shows the milling result on a dental amalgam. This is an AgHg alloy with added Cu and Sn. The initial surface is rough and very inhomogeneous. Preserving the phase distribution and phase contacts is crucial for the description of the microstructure. Ion milling has removed the irregular surface without introducing any mechanical smearing, and now the microstructure can be analyzed successfully.

As shown with the examples presented here, in addition to having the proper EBSD camera, sample preparation plays a huge role in getting optimal EBSD data. The broad beam ion mill is a versatile instrument to effectively remove oxidized, distorted, and irregular surfaces on all types of crystalline materials to prepare artifact-free EBSD analysis surfaces.

2020 Worldwide Events

October 19-22	
Analytica	Virtual
November 11-13	
Japan Analytical & Scientific Instruments Show	Chiba, Japan
November 14-19	
ISTFA 2020	Pasadena, CA
November 29-December 4	
Materials Research Society (MRS) Fall Meeting	Virtual

Note: We are continuing to monitor events affected by the spread of COVID-19.
Please visit http://www.edax.com/news-events/conferences-tradeshows for the most up-to-date information about our conferences and trade shows.

2020 Worldwide Training

To help researchers 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 following EDAX facilities.

EUROPE

EDS Microanalysis (APEX™ EDS)	
November 9-11	Weiterstadt*
November 30-December 2	Weiterstadt#
EBSD OIM Academy	
November 2-5	Weiterstadt*

JAPAN

APEX™ EDS	
October 15	Virtual
November 19	Virtual

CHINA

EDS Microanalysis	
December 1-3	Shanghai (ACES)
EBSD OIM Academy	
December 8-10	Shanghai (ACES)

*Presented in English
#Presented in German

Note: We are closely monitoring developments relating to COVID-19.
Please visit http://www.edax.com/support/training-schools for the most up-to-date information on our training courses.

Other EDAX Resources

EDAX has many resources available online for you to access at any time. Here are a few:

- Videos - EDAXNew YouTube Channel
- Blog - EDAX Blog
- Webinars - Available on-demand

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EMPLOYEE SPOTLIGHT

Marek Hradisky.

Marek Hradisky

Marek joined EDAX as a Field Service Engineer in April 2020. He is responsible for the installation, service, and maintenance of Energy Dispersive Spectroscopy (EDS) and Electron Backscatter Diffraction (EBSD) systems in Central and Eastern Europe. Marek is also available to support the rest of Europe.

Prior to EDAX, Marek worked at FEI (now Thermo Fisher Scientific) in Brno, Czech Republic. He began as a System Tester for Transmission Electron Microscopes in the factory. After gaining experience, he was promoted to Troubleshooter and eventually worked on a project involving X-ray spectroscopes. In 2016, Marek earned a Master of Engineering in Microelectronics from the University of Technology in Brno.

In his spare time, Marek enjoys hiking, skiing, and traveling. He also likes to race his professional radio-controlled cars on a small racing circuit.

(left to right): Manabu, Mio, Kazuki, Kaoru, and Mao Yamada.

Manabu Yamada

Manabu started at EDAX in 2008. As an Account Manager based in Tokyo, Japan, he provides sales and customer support to the eastern part of Japan.

Prior to EDAX, Manabu worked at Seiko EG&G in Tokyo. As an Account Manager, he was involved in the sale and support of Ortec products, an AMETEK company that manufactures Ge radiation detectors. Manabu studied system administration for three years at Tohoku Computer College in Miyagi, Japan. He learned how to build an entire information technology system.

Manabu and his wife, Kaoru, have a son, Kazuki (nine), and two daughters, Mio (eight) and Mao (four). In his spare time, Manabu enjoys watching movies and going out to eat with his family. He also loves to watch Mio dance to TWICE, a Korean idol group she watches on YouTube. Manabu likes playing with radio-controlled cars, but his children do not. Unfortunately, he has not been able to do everything he likes doing with the current restrictions in place due to COVID-19, but he is thankful for his family and enjoying life.

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Institute of Metals and Technology, Ljubljana, Slovenia

The Institute of Metals and Technology (IMT) was established in 1984 to provide support for the steel industry and became a public research institute in 1997. The bulk of the Institute's activities are focused on the research of metallic materials, mainly aluminum and steel. They also collaborate with major steel and aluminum producers in Slovenia.

The Scanning Electron Microscope (SEM) lab at IMT is part of the metallography lab, where scientists perform research on materials and their microstructures. This includes sample preparation, electron microscopy, microanalysis, and crystallography. The Zeiss Crossbeam 550 in the lab is equipped with an EDAX Pegasus Analysis with Octane Elite Super silicon drift detector and Hikari Super EBSD camera.

"We wanted a system that would allow us to use the microscope at its full potential, and we were especially looking for a combination of Energy Dispersive Spectroscopy (EDS) and EBSD analysis," stated Dr. Barbara Šetina Batič. "But data acquisition was not the only thing that was important. We also wanted a reliable system with good data processing software for EBSD. OIM Analysis™ was the most convincing of all the systems we tried."

The IMT uses its EDAX system to solve many problems. The scientists at the lab study steel cleanliness and to know the type, distribution, size, and composition of inclusions in steel. In order to accomplish this, they use particle analysis for statistical analysis of morphology, distribution, and chemical composition, EDS mapping and spot analysis for details on individual inclusions, and EBSD to determine the type of inclusion. This analysis helps improve the mechanical properties of steel.

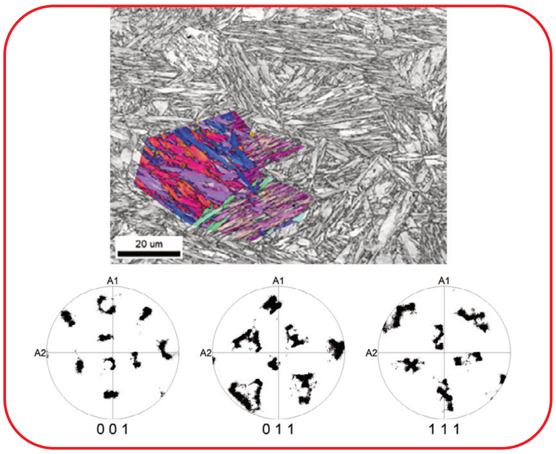

Figure 1. A Martensitic steel sample. The highlighted area belongs to the same prior austenite grain, as is seen from the pole figure.

Another example involved the study of carbides. The lab has used EDS and EBSD in the study of deep cryogenic treatment of steels. In particular, the scientists are interested in where the carbides are, what type of carbides are present, and how they are distributed. With that information, the team hopes to understand how cryogenic treatment works and how it influences the mechanical properties of the steels.

Figure 2. This is an EBSD IPF-Z image of SLM AM stainless steel.

The IMT is also interested in analyzing metals produced through additive manufacturing. The lab recently purchased a laser powder fusion bed and is researching the use of additive manufacturing of biocompatible alloys used for implant materials. Since microstructure information for mechanical and magnetic properties is so important for these materials, EDS and EBSD have been vital in developing the right processing parameters for the lasers.

To learn more about the IMT or to see how they are trying to help develop new materials with better properties that can make people's lives easier, please visit https://www.imt.si/.

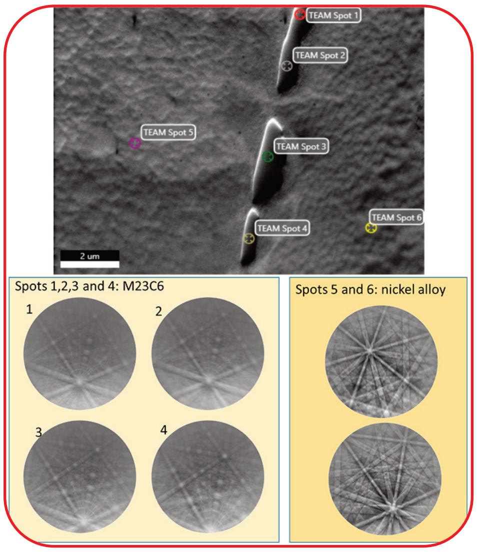

Figure 3. This is a study of carbide precipitates on grain boundary of nickel superalloy.