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EDAX NEWS

EDAX Propels Technology With The Latest TEAM™ Release

The EDAX TEAM™ software version 4.3 brings new, high performance features to our Energy Dispersive Spectroscopy (EDS), Electron Backscatter Diffraction (EBSD), and Wavelength Dispersive Spectrometry (WDS) products. It includes over 40 new features and workflow improvements to benefit users of all levels.

Several new EDS and EBSD routines are designed to speed data collection and analysis. An important EDS mapping update gives the user the ability to modify the EDS element list during the collection of a map set. This means that any changes can be accomplished in live time without having to stop the map, change the elements, and restart. New elements can be added while the collection continues, allowing the map data to be acquired during the investigation of elemental distribution. Even low concentration elements can be uncovered while the analysis is underway. Since all of the elements are thoroughly searched during the collection, as soon as the data collection is complete, the map set can quickly be reported, exported, and shared.

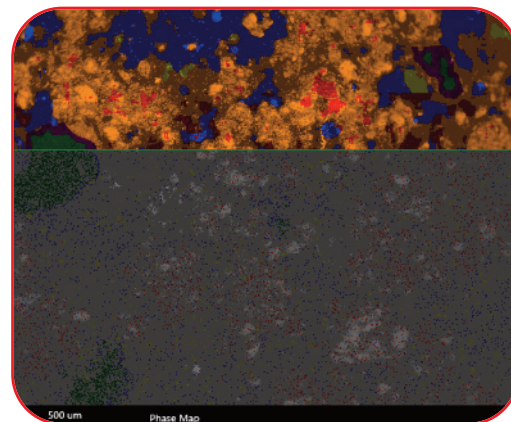


Figure 1. Displays the difference in phase assignments with the new fast phase mapping turned on (top) and not yet activated (bottom section). The increased assignment of phases is visible in the very first pass.

Fast phase mapping is a new patent-pending spectrum aggregate mapping technique, which further improves EDS maps. TEAM™ EDS has always used spectrum peak association as the primary mode for determining phases. This has a wealth of benefits over phase

(Continued from Page 1)

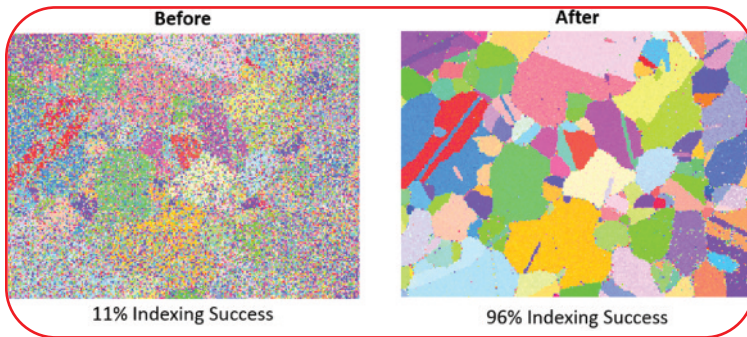


Figure 2. Compares the indexing success of the same low signal, high noise dataset before and after the NPAR routine.

identification by element map image association. This new routine further exploits this method by grouping neighboring like-spectra together and using the augmented sparse data to solve complex compounds with spectrum peak intensity ratios. As a result, all pixels in a map can be assigned to phases even on the first few passes (Figure 1). This means that there is the potential for 100% allocation of each data point to a phase within one minute. It also revolutionizes phase images to be another live-time imaging technique, with the benefit of full chemical characterization of the field of view. With increased collection time, higher resolution is possible, and the Auto routine will adjust the resolution to a higher level once an ample sized spectral data threshold is reached. This ensures the highest quality data display, with the best phase assignments.

We also have an EBSD technology breakthrough named Neighbor Pattern Averaging Rescan (NPAR), which evaluates and averages the band patterns of neighboring data points. With the intensified band pattern, orientations and structures can be solved with high confidence, even with low signal or during extremely fast frame rates. It enables analysts to max out their data collection speeds, while still achieving scans with high quality orientation solutions. This routine boosts the data from low indexing success to A-game top performing indexing success rates over 90% (Figure 2). NPAR indexing success is also possible with low signal, high noise patterns, which may be collected at low beam currents, consistent with high resolution Scanning Electron Microscope (SEM) imaging (Figure 3).

The Pattern Region of Interest Analysis System (PRIAS) feature has also been updated. It now determines grains by PRIAS images prior to the start of a scan. With PRIAS the number of redundant scanning points is minimized, the number of patterns is reduced, and the collection times are up to 100 times faster. The resulting dataset can be used to generate orientation and grain boundary maps used from metals to geological applications.

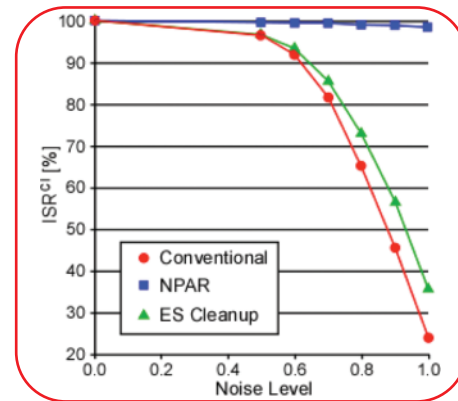


Figure 3. This chart compares various raw and data processed steps as a function of noise and indexing success rate (ISR). There is a tremendous extended range for high quality data even at low signals encountered in low beam current and fast frame rate conditions.

EBSD users have relied upon the power and practicality of OIM™ Comboscan to cover large orientation mapping areas to generate high volume micro-sized data on a macro scale. This feature is now available in the TEAM™ software. Users can quickly set up their intended area of analysis and the TEAM™ software will guide the best setup and collection parameters. The acquisition then progresses through many fields with no further user input, allowing for unattended collection with automatically combined images that are viewable during the collection.

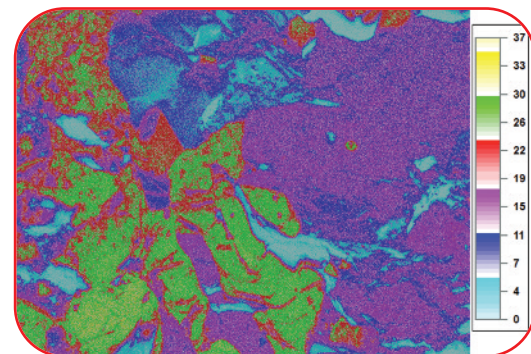


Figure 4. Silicon Region of Interest (ROI) map at 100x magnification, which shows signal intensity scaled by unique, user defined color.

Finally, element map images are now scalable to user specified or manually scaled color intensity threshold, which maximizes the visual appeal and minimizes the need for data interpretation using other methods. When all maps are scaled to the same values, any analyst can quickly and easily compare the map data to see where element variations occur. With a quick visual comparison, there is less need to understand the finer points of quantification. Yet, for those skilled in higher levels of interpretation, there is also a user defined color palette, which will show a scaling of the element intensity by peak intensity (Figure 4) or by quant value.

Analysis Depths for Micro-XRF

When measuring thin-film coated samples with Micro X-ray Fluorescence (Micro-XRF), one of the most common questions asked is “how thick can the coating be?” Also referred to as critical depth, or infinite thickness, this indicates the maximum sampling depth from which an analyte photon can be detected. It is important for thin-film applications because it defines the measurable thickness range for a given layer. However, because analysis depth is matrix-dependent, knowing the order of the layers and their composition is important in determining this value.

It is imperative to first understand the fundamental difference between “thin” and “bulk” thicknesses for Micro-XRF. Bulk thickness is approximated when 99% or more of the incoming X-ray signal undergoes photoelectric absorption or scattering, and less than 1% of the original signal is detected in the direction of the measuring system. In this case, the bulk sample has reached saturation of the intensity, where increasing the thickness will no longer increase the fluorescence signal. However, if more than 1% of the X-ray is detected (or less than 99% loss) it can be treated as a thin sample. Unlike bulk samples, the measured intensity of a thin sample is a direct function of its thickness.

Figure 1 below illustrates the incident and emergent beam, and their respective paths. The incoming X-ray reaches a critical depth d , and the emitted fluorescence signal emerges towards the measuring system over path length x . If 99% or more of the initial X-ray signal is absorbed over path length x , it is considered a bulk sample.

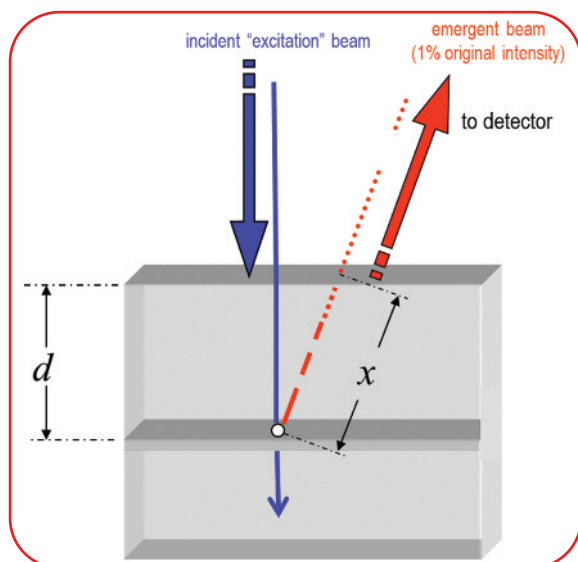


Figure 1. Illustration of incoming and outgoing X-ray paths, with x defined as the critical analysis depth.

Analysis depth x can be derived from the Beer-Lambert formula, shown below. This also factors in the density (ρ) and mass attenuation coefficient (μ) of the sample. I_x represents the measured intensity through x , and I_0 is the initial incoming X-ray intensity.

$$I_x = I_0 \exp(-\mu \rho x)$$

Since the criteria for bulk samples is at least 99% attenuation of the initial signal, the ratio of I_x/I_0 can be set to 1%, or 1/100. Now analysis depth x can be solved by simplifying the equation to:

$$x = 4.605 / (\mu \rho)$$

Using this formula, analysis depth can be calculated for any sample with known density and mass attenuation coefficients. The table below shows examples of nominal measured analysis depths for various pure elements.

Pure Element Analysis Depths (um) For Micro-XRF			
Element	K	L	M
Ti	20		
Ni	25		
Zn	35		
Mo	60	1.5	
Pd	70	2.5	
Ag	75	2.5	
Sn	100	3	
Pt		6.5	0.3
Au		8	0.5
Pb		10	0.75

Table 1. Nominal pure element analysis depths in microns, for K, L, and M series.

Because the mass attenuation coefficient decreases with increasing energy, the general trend in Table 1 shows increasing analysis depths as the atomic number increases. This is expected because as the atomic number increases, the energy of the emitted photon increases as well, allowing it to “escape” the sample from greater depths. When switching to lower-energy transition series, for example from K-series lines to L-series lines, then the analysis depth is significantly decreased.

Atom Probe Assist for Efficient Atom Probe Tomography Specimen Preparation

Technique Challenge

Atom Probe Tomography (APT) is a high spatial resolution chemical analysis technique that allows elemental identification at the atomic level. Atoms are evaporated from the apex of a sharp needle-shaped sample and both the atomic number and the original position of the atom are determined. Subsequent time-of-flight mass spectrometry allows a three dimensional atomic reconstruction of a material.

The APT technique is ultimately suitable for detailed analysis of low levels of trace elements and their distribution in a material, for example to visualize grain boundary segregation. However, this high resolution also severely limits the volume of material that may be analyzed. Typical specimens are needle-shaped with tip diameter less than 100 nm and a typical analysis depth less than 500 nm.

Specimen preparation is relatively straightforward if the question does not require site-specific analysis in a homogeneous specimen, but targeted sample preparation of a specific feature requires high resolution imaging methods to ensure that the area of interest is present near the tip of the needle. For this type of sample, the Atom Probe Assist provides unique tools to help efficiently prepare and position the feature of interest for analysis.

Comparison with Existing Techniques

Conventional specimen preparation to produce the sharp needles required for APT analysis is typically done via electropolishing. This is often combined with Focused Ion Beam (FIB) milling and Transmission Electron Microscope (TEM) imaging to position a specific feature near the tip of the needle.

Electropolishing

- Conventional electropolishing of small ($10 \times 0.3 \times 0.3 \text{ mm}^3$) rods that are pre-cut mechanically works well for “bulk” analysis.
- This method does not allow for accurate positioning of a specific site near the tip apex.
- Grain boundaries may be lost during processing.

FIB Refinement

- When preparation of a specific feature is required, a coarse tip containing the area of interest may be produced by electropolishing, which is then refined by targeted FIB milling of the feature of interest.
- A special region of interest can be lifted out of a bulk sample and positioned on a prepared post.

- The main difficulty with this method is that the orientation contrast in the FIB image becomes very low when the sample approaches the required dimensions.
- Grain boundaries in the top 200 nm of the sample are invisible.

TEM Position Verification

- When the FIB contrast becomes insufficient to locate the feature accurately, the contrast may be improved by viewing the needle in a TEM.
- Diffraction contrast in TEM images allows easy identification of boundaries, but images need to be compared to identify the location again when the sample is returned to the FIB.
- This requires moving the specimen between instruments several times, which is time consuming and risks damaging the sample during re-mounting.

Optimization of site specific APT specimen preparation using Atom Probe Assist

- Atom Probe Assist utilizes the electron transparency of the sample to collect diffraction patterns of the needle on a standard EBSD detector.
- Analysis can be done in-situ in the FIB-SEM, the sample can remain in vacuum.
- Indexing the patterns allows for accurate orientation determination with up to 5 nm spatial resolution.
- In addition to locating the feature of interest, the orientation measurements may also be used to characterize the crystallographic character of the boundary, e.g. if it is a low-angle, high-angle, or twin boundary.

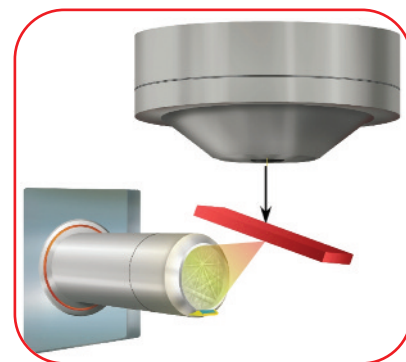


Figure 1. t-EBSD configuration.

APT Specimen Preparation Geometry

The sample is mounted in the FIB-SEM such that the needle is parallel to the FIB beam (Figure 2).

(Continued from Page 4)

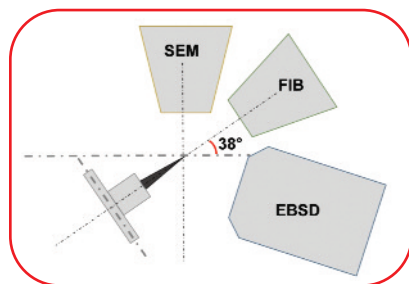


Figure 2. APT specimen preparation geometry.

This configuration allows milling, SEM imaging and Atom Probe Assist mapping without moving the specimen.

The diffraction patterns originate from the side of the specimen that faces the EBSD detector. The thickness variation across the needle tip affects the contrast in the patterns such as these collected at the tip of the specimen (Figure 3 left-thin) and further into the needle (Figure 3 right-thick). Enhanced image processing ensures optimum band detection and indexing over a range of specimen thickness up to a thickness of ~300 nm.

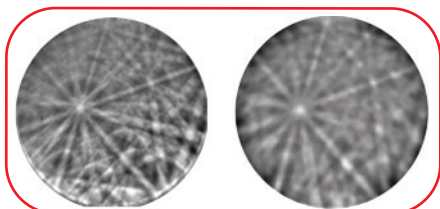


Figure 3. t-EBSD patterns collected at the tip of the specimen (left) and further into the needle (right).

The Atom Probe Assist mapping can be used to collect orientation snapshots between the successive FIB milling steps to bring the grain boundary closer to the tip (Figure 4). The individual maps were collected in three minutes.

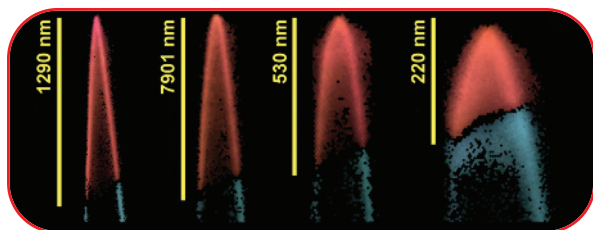


Figure 4. Orientation snapshots between successive FIB milling steps, bringing the grain boundary closer to the tip.

Example: grain boundary analysis

The presence of boron segregation on a prior austenite grain boundary was investigated by APT. The grain boundary was identified in the electropolished APT sample by applying a picric acid etch. The target boundary was then positioned close to the tip apex using the in-situ transmission-EBSD (t-EBSD) and FIB milling technique (Figure 5).

EBSD maps (Figure 6) were collected from three directions by

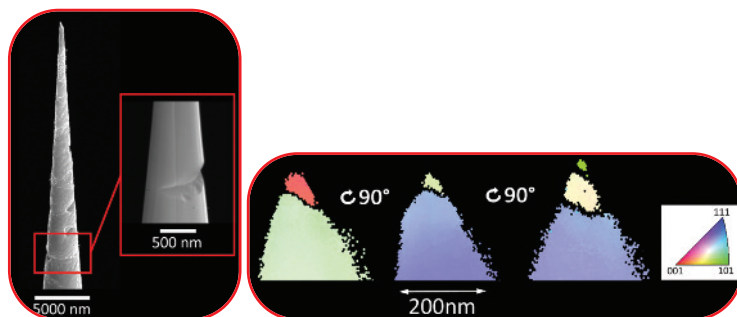


Figure 5 (left). Tip after etching.

Figure 6 (right). EBSD maps collected from three directions by rotating the needle to illustrate the interface of the boundary in the tip of the needle.

rotating the needle to illustrate the interface of the boundary in the tip of the needle.

The EBSD maps show the crystal direction pointing to the EBSD detector for three different sample orientations. The trace of the prior austenite grain boundary in the maps can be matched with the B and C segregation in the atom map (Figure 7).

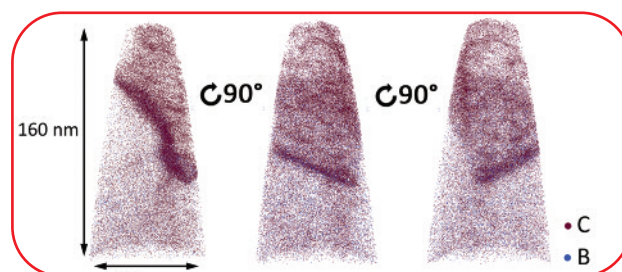


Figure 7. Atom map showing B and C segregation.

Recommended EDAX Solution

The EDAX Atom Probe Assist tool is recommended to help Atom Probe users prepare APT samples more efficiently and provide quantitative information on grain boundary position and character to accelerate research and understanding of grain boundary segregation. This product includes both a Hikari Plus EBSD Camera, which provides fast and accurate collection of EBSD and t-EBSD patterns for crystallographic characterization of APT samples, and TEAM™ EBSD software with a dedicated Atom Probe analysis mode with customized features for the unique analysis of APT samples.

Acknowledgements

- The material for this application note was provided by voestalpine Stahl Donawitz GmbH.
- The APT analysis was performed at the Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben.

2015 Worldwide Events

October 4-7

GeoBerlin 2015

Berlin, Germany

November 12

National Metallurgists Day (NMD) 2015

Pune, India

October 4-8

Materials Science & Technology (MS&T) 2015

Columbus, OH

November 24-27

The 2nd East-Asia Microscopy Conference

Hyogo, Japan

October 13-17

Northeastern Association of Forensic Scientists (NEAFS)

Hyannis, MA

November 29-December 3

2015 MRS Fall Meeting

Boston, MA

October 14-15

Appalachian Regional Microscopy Society (AReMS) 2015

Boone, NC

November 29-December 4

Microscopy Society of Southern Africa 2015

Pretoria, South Africa

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

2015 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	
TEAM™ EDS	
November 16-18	Wiesbaden#
November 30-December 2	Wiesbaden#
December 8-10	Tilburg*
Particle Analysis	
December 3	Wiesbaden#
EBSD	
November 18-20	Wiesbaden#
TEAM™ Pegasus (EDS & EBSD)	
November 16-20	Wiesbaden#
TEAM™ WDS	
November 17-19	Tilburg*
XRF	
October 20-22	Tilburg*
November 5-6	Wiesbaden#

JAPAN

EDS Microanalysis	
Genesis	
October 8-9	Tokyo
November 12-13	Osaka

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
November 17-18	Mahwah, NJ
EBSD OIM™ Academy	
October 27-29	Draper, UT
Micro-XRF	
October 6-8	Mahwah, NJ

CHINA

EBSD OIM™ Academy	
October 20-22	Shanghai (ACES)
Particle Analysis	
December 8-10	Shanghai (ACES)

*Presented in English

#Presented in German

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EMPLOYEE SPOTLIGHT



(left to right): Braden, Brodhi, Roger, Vance, Vonn, and Rebecca Kerstin.

Roger Kerstin

Roger joined EDAX in September 2013. He is the United States southeast regional sales manager. Roger's territory includes 11 states from Maryland to Arkansas. He guides, teaches and informs customers and potential customers about EDAX's product lines. Roger also participates in customer demonstrations, visits, and workshops to provide further insight into the company's products. He also serves as a board member for the Appalachian Regional Microscopy Society (AReMS).

Prior to EDAX, Roger worked at PANalytical for two years. Previously, he was employed at OIMS for 15 years in Chicago, IL and then moved to the Georgia office. Roger has over 20 years of experience in purchasing, marketing, product management, sales, and sales management.

He received a Bachelor of Arts degree in public relations/marketing with a minor in theatre from Illinois State University in Normal, IL. Recently, Roger has taken courses in chemistry and microscopy.

Roger currently lives in Suwanee, GA with his wife, Rebecca. The couple has been married for 19 years and they have four boys, Braden (13), Brodhi (11) and identical twins, Vance and Vonn (six).

In his spare time, Roger enjoys doing various projects around his home, such as woodworking, fixing things, and teaching his boys how to do the same. He also likes golf, boating, swimming, gardening, and helping his neighbors. Roger coaches the twins' soccer team, serves as an assistant coach for his oldest sons' soccer team, and is also a soccer referee. An avid sports fan, he is still a die hard Bulls, Blackhawks, Bears, and White Sox fan, even though he has lived in Georgia for over eight years.



(left to right): Vinitha, Emelda, Ebenezer, and Evangeline Phillip.

Ebenezer Phillip

On June 9, 2014, Ebenezer (more commonly known as Phillip) joined EDAX as the human resources manager in the Mahwah, NJ office. He came to EDAX after serving as the human resources manager for AMETEK Instruments India Pvt. Ltd. from 2010-14. Phillip was instrumental in supporting the growth of AMETEK infrastructure in India, which supports over 30 business units. He was also a major contributor to the substantial growth of human capital and talent needs of AMETEK India.

Before joining AMETEK, Phillip worked in various human resource generalist roles in India for 16 years. His background includes engineering, information technology, leadership and development institutes, and community service organizations. Phillip has always had a passion for training and development.

Phillip earned a bachelor's degree in business from Bangalore University in 2002. He received his masters in business administration from the Indian Institute of Business Management and Studies in 2010. Phillip is also certified in various programs including Customer Relationship Management (CRM) and he is a green belt six-sigma holder. He also mentors within the human resource community.

Phillip is married to Vinitha, who is a professor at Saint Joseph's College of Commerce, Bangalore, India. They have two daughters, Emelda Veronica (nine) and Evangeline Venessa (six).

An active member in his church committee, Phillip also enjoys wildlife sightseeing, trekking and his new hobby, flying remote aircrafts.

Planetary Materials Research Group Lunar and Planetary Laboratory, University of Arizona Tucson, AZ

The Planetary Materials Research Group (PMRG) at the University of Arizona's Lunar and Planetary Laboratory studies planetary materials, such as interplanetary dust particles, grains of stardust, primitive meteorites, and soils from the moon and asteroids. These materials are pieces of condensed matter that were left over from when our solar system formed over 4.5 billion years ago.

The PMRG investigates the constituent materials within samples at scales ranging from micrometers down to the atomic level. The group uses information about the crystal structure and chemistry of these materials to understand the conditions under which the minerals were formed and the processes that led to their formation and alteration, either in the early solar system or the circumstellar environments of ancient stars. The group is trying to gain insight into our origins by learning how the building blocks of our solar system were formed. Many of the samples contain complex organic compounds, including amino acids, so in some samples the PMRG is actually looking at precursor molecules to life as we know it.

"We're still getting to know our TEAM™ system, but it's clear that it has a lot of flexibility with analytical protocols. It is well suited to measuring chemical complexity in a wide range of material types. We have users that analyze rock samples, ceramics, electronic materials, etc. The reputation of the instrumentation and service, as well as ease of use were the key factors in deciding to go with EDAX." said assistant professor Thomas J. Zega.



Figure 1. University of Arizona Lunar and Planetary Laboratory with an FEI Helios dual-beam FIB-SEM and EDAX TEAM™ Pegasus Analysis System.

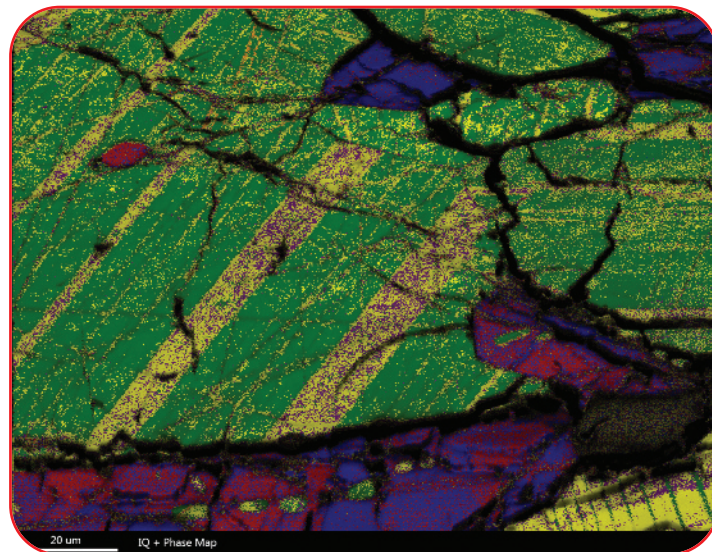


Figure 2. Phase map of a sample from a Howardite meteorite.
Image credit: Dr. Ken Domanik.

The group purchased an EDAX TEAM™ Pegasus Analysis System with an Octane Plus Silicon Drift Detector (SDD) and Hikari XP Electron Backscatter Diffraction (EBSD) Camera for integrated Energy Dispersive Spectroscopy (EDS) and EBSD. It is using the system to explore chemical and structural heterogeneities in its samples. The PMRG is utilizing the combined analytical capability of EDS and EBSD in its Focused Ion Beam Scanning Electron Microscope (FIB-SEM) with slice-and-view imaging to give it new insights into the way crystal growth occurred in two and three dimensions billions of years ago under extreme conditions.

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