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EDAX NEWS

EDAX Increases the Speed of the Hikari XP More Than 50%

Learn how the improved Hikari XP EBSD Camera and TEAM™ software deliver a winning combination to maximize system performance and data quality.

At the recent Microscopy & Microanalysis meeting, EDAX announced a more than 50 percent increase in the data collection speeds of its Hikari XP EBSD camera, while maintaining its industry-best indexing quality standards. The improvements to the Hikari XP provide outstanding performance across the complete range of EBSD applications by offering a greater range of camera operating conditions.

An orientation map from a nickel-based superalloy sample is shown in Figure 1. This map was collected at approximately 1000 indexed patterns per second with an incident beam current of approximately 4 nA at 20 kV, with indexing success rates greater than 99 percent. This data represents a 53 percent improvement over the previous generation of the Hikari camera.

To better illustrate the advances of the Hikari XP, it is worthwhile to explain how the EDAX EBSD cameras are operated, what variables are in play, and how these are optimized for faster data collection. The primary goal of EBSD pattern analysis is to detect the diffraction band positions using a Hough transform in order to correctly determine the crystallographic orientation.

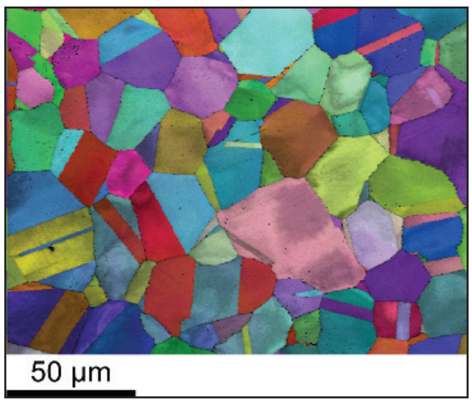

Figure 1. Orientation map of a nickel-based superalloy sample.

AMETEK®
MATERIALS ANALYSIS DIVISION

EDAX
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This pattern analysis rarely requires the use of the full resolution of the sensor, but instead the patterns are "binned" in order to create a lower resolution image by combining blocks of pixels. The more the patterns are binned, the smaller the resolution and the faster they can be obtained. Therefore, the selection of a pattern resolution is one variable in the determination of the data collection speed. One of the important improvements in the new Hikari XP is the readout rate of the patterns, which translates into potential speed increases for each resolution setting. For example, at 5x5 binning, or a 96x96 pixel image, the maximum speed has increased from 258 to 448 frames per second, a 74 percent increase.

The Hikari XP also offers fully adjustable gain settings. Increasing gain value electronically amplifies the signal level of the EBSD pattern and decreases the time required to obtain each pattern. Gain works in conjunction with the camera exposure time to determine the overall signal level of the EBSD patterns. Generally, at any given beam current, the exposure and gain are adjusted to obtain the desired exposure level. However, increasing gain also incurs an increase in signal noise level. Figure 2 shows a small-scale example of the resolution and gain variables. The nicest looking patterns are obtained at high resolution and low gain settings, but take the longest time to collect, while the patterns for the fastest collection are obtained at lower resolutions and higher gains. Increasing beam current lowers the exposure time and gain requirements while decreasing current increases those requirements.

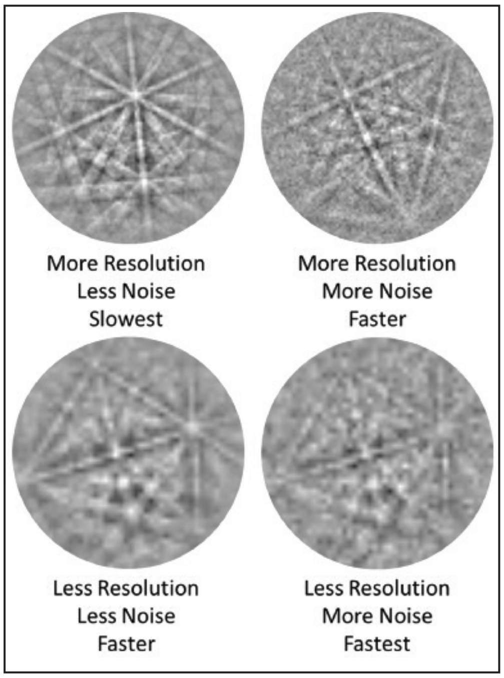


Figure 2. A small-scale example of resolution and gain variables.

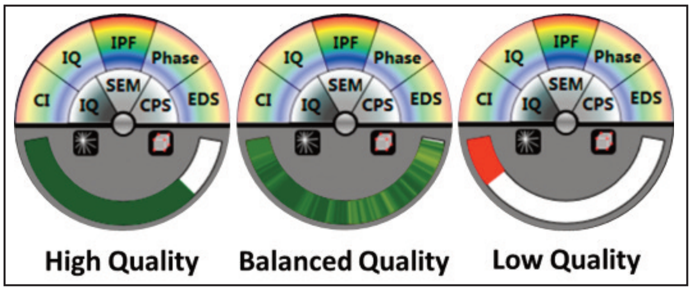


Figure 3. Map quality feedback via the MASCOT display.

For any material, with its given crystal structure, density, level of deformation, and sample preparation quality, there is a threshold combination of resolution and gain that provides high-quality data and beyond which quality suffers. It is often difficult to correctly determine this threshold visually, as the human eye is not as proficient at reliable band detection as a well-programmed Hough transform. It is important to emphasize that high-quality data is obtained by finding the band positions correctly. Quality degradation will occur due to a failure of band detection, which translates into a lower confidence index, a patented measurement of the quality of the orientation solution.

The TEAM™ software platform provides two important functions to maximize the potential of the Hikari XP. The first is the Smart Camera optimization, where the user selects the desired operational mode (fast, low-noise, simultaneous EDS-EBSD, max speed, etc.) and the software selects the appropriate camera resolution and determines a recommended exposure time and gain combination to meet the operation requirements. The second is map quality feedback via the MASCOT display as shown in Figure 3. Within the MASCOT (Map Selection Control) tool, the progress curve is increasingly colored by indexing quality. For the higher quality data, it is usually fully colored green, while for very poor quality data it is colored red, which would indicate that band detection is consistently failing and settings should be adjusted to more conservative values. The middle example, labeled Balanced Quality, indicates regions where indexing quality approaches the lower levels of acceptability by using yellow coloring. When trying to optimize acquisition speed, MASCOT coloring similar to this indicates that high-quality data is being obtained, but that further speed increases will probably require a beam current increase rather than camera parameter adjustments.

By combining the improvements of the Hikari XP and the features and feedback of the TEAM™ software platform, it is possible to easily acquire data faster across all ranges of samples without sacrificing quality.

Merging Orbis Elemental Map Images

The elemental map merging tool available in the Spectral Utilities program, located in C:\edax32\UTL, is useful for merging maps acquired from adjacent regions of the sample. This is typically done by using a camera video field to define the region on the sample to be mapped. Then adjacent video fields, for example, a column or row of video fields or quadrants of a rectangular area, are mapped. This gives a sample video image using the full resolution of the sample video camera while having more flexibility in defining mapping areas.

The map merging utility also has the capability to increase the contrast of the maps while merging them. This process benefits weak intensity maps by improving the contrast of the maps. However, high intensity maps can be saturated, washing out the features of the sample. Therefore, it may be beneficial to merge the maps under a variety of contrast conditions and select the maps with best contrast.

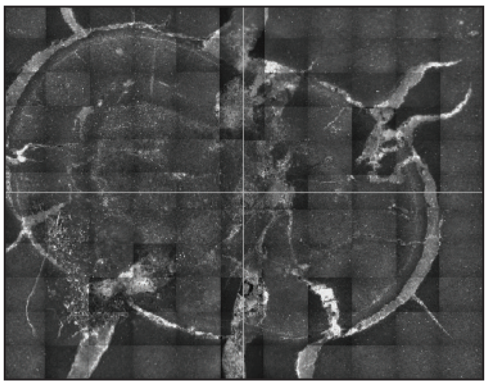


Figure 1. Sample video comprised of 4 high magnification montage images merged together. The individual video montages were collected as quandrants of a rectangle showing an inclusion in a geological sample. The overall, merged field of view is about 25 mm across.

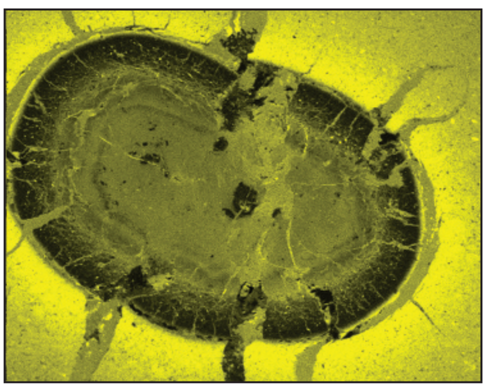


Figure 2. Mn(K) merged elemental map showing the inclusion is Mn poor.

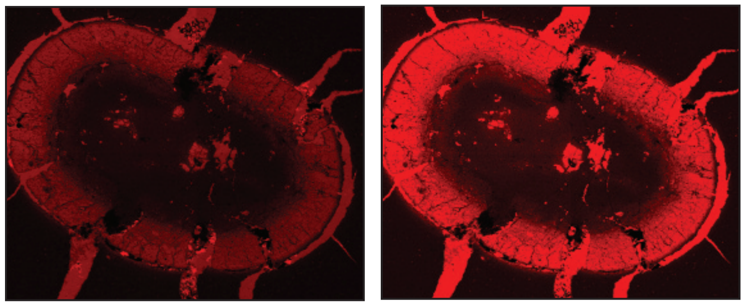


Figure 3a. Figure 3b.
Ca(K) merged elemental maps under low contrast (a) and high contrast (b) conditions. The CaK map merged with higher contrast has improved visibility of various sample features.

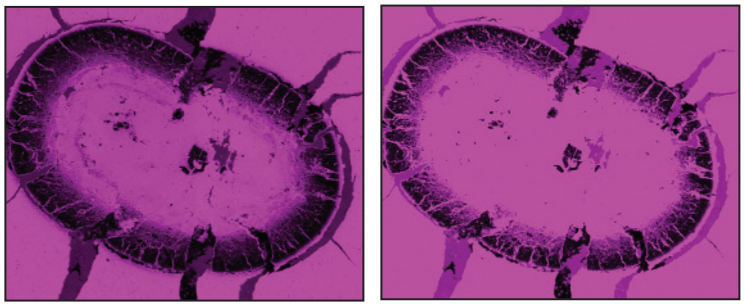
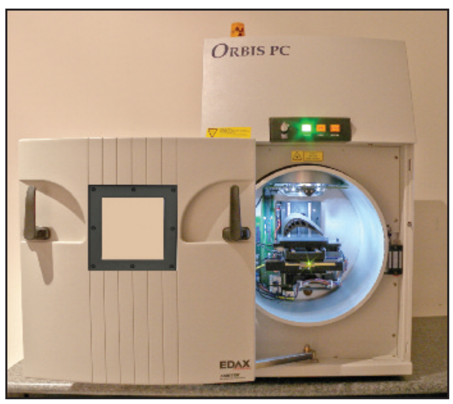


Figure 4a. Figure 4b.
Figure 4: Fe(K) merged elemental maps under low contrast (a) and high contrast (b) conditions. The FeK map merged with low contrast has improved visibility by reducing saturation.

The EDAX Orbis Micro-XRF Elemental Analyzer combines multiple spot sizes and a coaxial video/X-ray design to provide users with a flexible, easy to use tool capable of non-destructive inorganic elemental analysis from powders and fragments to topographical samples.



Benefits of WDS Spectral Resolution for Elemental Confirmation

Abstract

High confidence in the elemental identification of EDS spectra is extremely important for understanding material constituents and quantifying compositions. Because peak overlaps occur in the spectra from many samples, WDS can be used to confirm the existence of the overlapped or trace elements. This article will provide results illustrating how the superior spectral resolution of WDS aids the analyst in proper elemental identification for superior confidence in all subsequent analyses.

Background

The first task of a spectroscopist is the discovery and identification of all of the elemental peaks within the acquired spectrum. Isolated peaks are easy to locate and identify, but overlapped peaks and/or low amplitude peaks may be more difficult to discover. Many software algorithms are available to aid in this task, but the user typically wished for better confirmation techniques.

EDS detectors have a specified resolution which is defined as the FWHM of the spectral peak at the Mn-Ka X-ray energy, with a typical value being 130 eV for SEM microanalysis systems. However, the energy resolution is not constant across the whole spectral energy range but varies as the square-root of the energy from approximately 50 eV for C-K to 160 eV for Cu-Ka. Another complication is that the resolution is only valid for the slowest electronics processing setting of the detection system. All other electronics settings, which are more useful and popular for high-acquisition rate analyses, degrade the resolution and broaden the peaks. In addition, the resolution gives no indication that there is a substantial Bremsstrahlung background energy in the spectrum that adds confusion to peak identification. Small amplitude elemental X-ray peaks become more difficult to discover when the background intensity is larger or these peaks are in the neighborhood of larger amplitude peaks.

WDS has the benefit of superior spectral resolution than EDS at every X-ray energy. This provides the benefit of easily resolving elemental X-rays which are closely spaced to each other. The spectral resolution does not vary with acquisition conditions because there is no variation in the electronics settings for the system. In addition, the intrinsic spectral background is not dictated by the Bremstrallung intensity and is significantly lower than EDS which aids in the discovery of very small amplitude peaks. The disadvantage of WDS compared to EDS is the serial nature of the spectral acquisition by the need to scan the energy range which takes time.

The spatial resolution of electron microanalysis depends primarily on the incoming electron beam energy and to a lesser extent on the elemental X-ray energy. Most historical analyses were performed at 10-30 keV beam energies for micron-scaled analyses. Most modern high spatial resolution analyses are being performed at energies well less than 10 keV, mostly at 3-5 keV, with some specialized experiments being performed below those energies. At high beam energies, the wide available energy range reduced but did not eliminate the opportunity for elemental peak overlaps. At the modern lower beam energies, the increased potential for elemental X-ray peak overlaps at low energies creates an increasingly difficult situation where most of the peaks in the spectrum may be overlapped. High spectral resolution techniques are required by the analyst to gain the needed confidence to arrive at the correct interpretation.

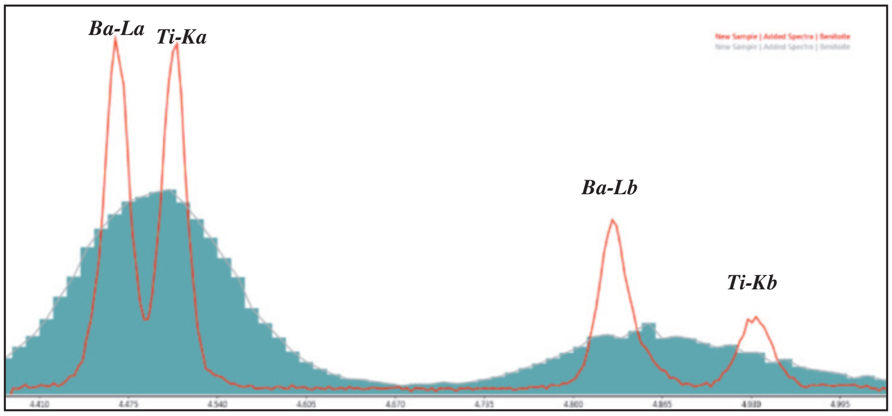
Results

The following examples compare the results of EDS (teal) and WDS (red) to illustrate the benefits of WDS in the discovery and identification of the elemental peaks in a variety of materials.

(Continued from Page 4)

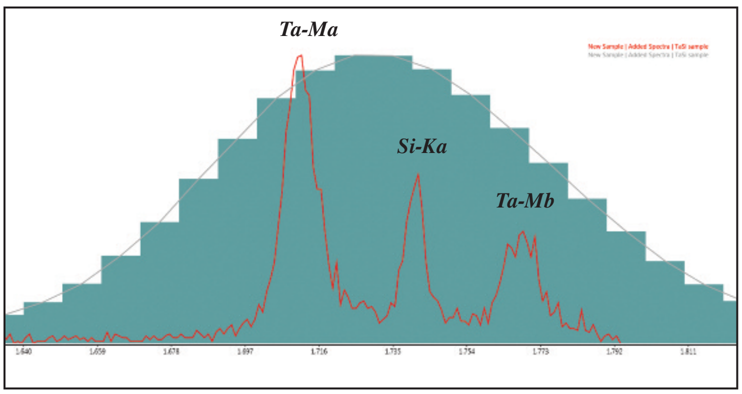
Barium Titanate

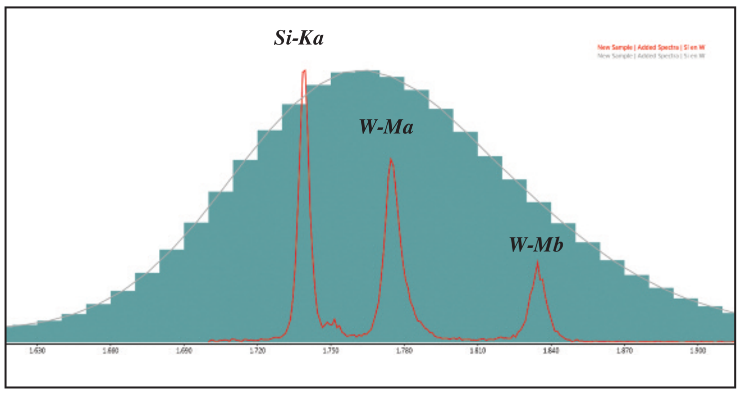
Some elemental peak overlaps cannot be avoided or the elements discovered even at high beam energies. This example of Barium Titanate ($BaTiO_3$) is a perfect illustration. There are no other X-ray lines for Ba that can be easily used for discovery except the L-lines at 4.4 keV. Those lines are very close in energy to the Ti-K lines and appear overlapped in an EDS spectrum. The WDS has sufficient spectral resolution to separate the lines and confirm the existence of both elements in the sample.



Silicon Overlaps in Semiconductor Samples

Many structures and defects in semiconductors contain refractory metals. Identification would be simple if analyses were performed at high beam energies, but spatial resolution preferences restrict analyses to low beam energies. At these energies, only M-line X-rays are generated and many of these overlap with the substrate Si-K X-rays, like Ta-M and W-M. The peaks in the EDS spectra are slightly broader and mask the presence of the impurity. Only by using the WDS can the elements be uniquely discovered and identified.





Conclusions

WDS is a technique that is complementary to EDS and provides the necessary confidence in discovering and correctly identifying all of the elemental x-ray peaks in a spectrum for further processing.

Worldwide Events

September 18-20
Materialographie — Friedrichshafen, Germany

October 27-31
MS&T 2013 — Montreal, Quebec, Canada

November 5-6
ISTFA — San Jose, CA

December 1-6
Materials Research Society (MRS) Fall — Boston, MA

Please visit www.edax.com for a complete list of our tradeshows.

2013 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	
November 7-8, 2013	Tilburg
TEAM™ EDS	
September 24-26, 2013	Tilburg
November 11-13, 2013	Wiesbaden
Genesis	
November 19-21, 2013	Tilburg

EBSD	
October 28-30, 2013	Tilburg
November 13-15, 2013	Wiesbaden

TEAM™ EDS & EBSD (Pegasus)	
November 11-15, 2013	Wiesbaden

WDS LEXS	
October 8-9, 2013	Tilburg

Orbis: Course & Workshop Presented in English	
October 29-30, 2013	Wiesbaden

JAPAN

EDS Microanalysis	
Genesis	
October 10-11, 2013	Tokyo
November 14-15, 2013	Osaka

CHINA

EDS Microanalysis	
TEAM™ EDS	
September 24-26, 2013	Shanghai
October 10, 2013	Xi'an
December 3-5, 2013	Shanghai

EBSD OIM™ Academy	
October 15-17, 2013	Shanghai

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
September 17-19, 2013	Mahwah, NJ
December 10-12, 2013	Mahwah, NJ

EBSD OIM™ Academy	
November 12-14, 2013	Draper, UT

Micro-XRF	
October 1-3, 2013	Mahwah, NJ

Please visit www.edax.com/support/training/index.aspx for a complete list and additional information on our training courses.

EMPLOYEE SPOTLIGHT

Gene Griggs

Gene returned to EDAX as a Senior Sales Manager in the western region of the United States in July 2013. Previously, he was a salesman at EDAX from 2000-06. Gene is responsible for all sales related activities in his territory. He participates in trade shows and works closely with the applications team to set up demonstrations and training.

Gene worked as a sales engineer at Thermo Fisher Scientific from 2012-13. He was responsible for the western United States. From 2007-12, Gene was an account manager in the southwestern territory for FEI. He worked closely with the applications and marketing teams to provide SEM, TEM and dual beam solutions for customers. Gene served as a sales engineer in the southern region for Saint-Gobain (formerly Norton Industrial Ceramics) for six years (1994-2000). He provided sales and applications support for high purity ceramic products used in semiconductor diffusion processes. Primarily, he handled high temperature and chemical vapor deposition (CVD) process applications, such as poly and nitride.

In 1987, Gene received a Bachelor of Business Administration degree in marketing from Tarleton State University in Stephensville, TX. In 1996, he earned a Master of Science degree in science and technology commercialization from the University of Texas in Austin, TX.

Currently, Gene resides in Fort Collins, CO with his wife, Melinda and two daughters, Haleigh (nine) and Caroline (six). In his spare time, he attends his girls' soccer games in the Arsenal U10+ League. Gene is an avid hiker and enjoys camping in the Rocky Mountains at altitudes above 9,000 feet. He also likes prospecting for minerals.

Mikiko Sugimoto

Mikiko joined EDAX in June 2005 as a Sales Assistant in Tokyo, Japan. Over the past eight years, she has worked with customers, operations and purchasing to ensure that EDAX meets the needs of its clients.

Prior to EDAX, Mikiko exported gardening supplies for a Japanese trading company in Yangon, Myanmar (Burma). She was responsible for inspecting potted plants prior to their export. Mikiko was also involved in the product development process. After returning to Japan, she worked for a trading company in Nagoya.

In college, Mikiko studied politics and economics, with a major interest in Southeast Asia. After graduation, she became a Japanese language teacher in Yangon.

Mikiko lives with her husband, Tatsumi by the ocean in Kamakura, Japan. She enjoys running and cycling along the coast. As a hobby, Mikiko and her husband take trips to participate in marathons and short distance races. Currently, she is learning aromatherapy on the weekends and finds it to be very useful in her life. Mikiko likes spending time with her boss and colleagues after work. She is grateful for having the opportunity to work with such wonderful people.

CUSTOMER NEWS

Center for Electron Microscopy and Analysis (CEMAS) Ohio State University, Columbus, Ohio

On September 18, Ohio State University will hold a ribbon cutting ceremony for its state-of-the art Center for Electron Microscopy and Analysis (CEMAS). The $15 million cutting-edge facility seeks to become the new hub for business and academia for materials characterization.

CEMAS' capabilities will allow it to be one of the top ten centers in analytical electron microscopy in the world. It will serve as Ohio State's center for multidisciplinary imaging research in physical and biological sciences. Research students and associates at CEMAS will be at the forefront of developing new techniques and methods for analytical electron microscopy.

Current and future challenges in medicine, healthcare, environment, energy and technology need to be addressed. The solutions required to meet these challenges require a multidisciplinary approach to research, which is the type of expertise CEMAS is seeking to obtain.

"Our goal is to provide Ohio State researchers with a world-class electron microscopy facility with a comprehensive range of imaging and spectroscopy techniques," stated Senior Research Associate Cameron Begg. "To support this, we need to be able to prepare and analyze a wide range of specimens. The ability to provide rapid chemical and structural information on a sample is fundamental to the understanding of materials. The speed of data collection, compatibility with our other EDAX systems and ease of use for our researchers with their many and varied backgrounds led us to choose EDAX."

The researchers at CEMAS use the EDAX Octane Silicon Drift Detector for many different applications. They cover every aspect of materials analysis from imaging the positions of atoms in new alloys to providing information on the surfaces of old photographs for an art professor. The Octane is primarily used to obtain information about surface chemistry of samples either from individual points or from an area.

"The new Octane detector is part of a suite with capabilities ranging from sample preparation using focused ion beams and ultra high resolution imaging in state-of-the-art TEMs to imaging and analysis of surfaces using SEMs," continued Begg. "The extremely high throughput of the new detector allows us to make this possible in minutes instead of the hours it took previously. Being able to acquire high resolution maps of the chemistry of samples permits us to identify and home in on the areas of interest in a much more timely and helpful manner."

Lobby of the new Center for Electron Microscopy & Analysis at Ohio State University. Photo courtesy of Lee Casalena

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