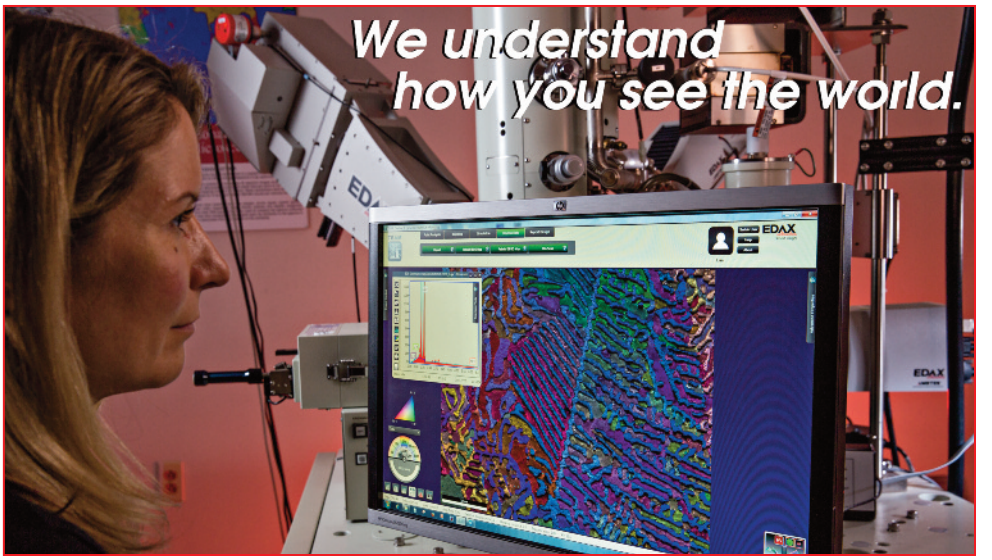
EDAXinsight

December 2013

Volume 11 Issue 4

INSIDE



EDAX NEWS

Transmission-EBSD: Improving the Spatial Resolution of EBSD

Electron Backscatter Diffraction (EBSD) has become a well-established microstructural characterization tool in the materials and earth sciences. When coupled with a Field Emission Gun Scanning Electron Microscope (FEG SEM), spatial resolutions less than 50 nm have been reported. However, the ability to achieve this performance depends on a number of factors including the average atomic number of the sample, the deformation state of the material, the acceleration voltage and beam current of the SEM, and the average grain size and grain size distribution. The size of the electron beam interaction volume increases with decreasing atomic number and with increasing acceleration voltage. As the interaction volume size approaches the grain size, EBSD patterns will become more diffuse, begin to overlap, become more difficult to index, and eventually disappear as multiple grain orientations are sampled at a given location. Therefore, EBSD spatial resolution will be better with higher density, deformation free samples.

In an effort to improve upon the spatial resolution of EBSD, Keller and Geiss demonstrated that EBSD-like patterns could be obtained using electrons that transmit through a sample thin enough to be electron-transparent using a standard EBSD detector configuration. Spatial resolutions less than 5 nm have been reported using this approach. Although the electrons forming the patterns are not backscattered, this technique has been termed transmission-EBSD or t-EBSD. While the more technically correct name transmission Kikuchi diffraction (TKD) has been proposed, this operational mode will be referred to here as t-EBSD out of respect for the initial work of Keller and Geiss.

(Continued from Page 1)

One of the appealing benefits of t-EBSD is that it can be done using existing EBSD hardware and software. For more details on sample positioning, read the 'Tips and Tricks' on the following page. As with regular EBSD, it is important to find the bands correctly to enable accurate and consistent pattern indexing. The flexibility of the Hough transform within the TEAM™ software allows optimization of band detection for t-EBSD samples. This is important as the band projections onto the phosphor screen are different in the t-EBSD geometry and varying the acceleration voltage with sample thickness will change the widths of the diffraction bands within the patterns.

Sample preparation for t-EBSD can be challenging in comparison to standard EBSD preparation requirements. For t-EBSD, the sample needs to be electron-transparent at acceleration voltages between 15-30 keV. This translates to a recommended thickness range of approximately 75-300 nms. When thinner than this, the detected patterns are generally weak and noisy, and when thicker than this the patterns lose sharpness and detail. The sample preparation approaches are generally the same as with Transmission Electron Microscopy. One approach is grinding and dimpling a sample followed by low-energy ion milling to produce a hole near the center of the analysis region. Adjacent to this hole, the sample is very thin. This region generally gives very good t-EBSD patterns. Another approach is to use the Focused Ion Beam (FIB) lift out technique. This allows for a site-specific analysis on a particular region of interest.

In addition to t-EBSD working with standard EBSD hardware, the Forward Scatter Detector (FSD) also provides useful and interesting information in the t-EBSD geometry. Figure 1 shows an FSD image collected from a copper film sample prepared by FIB liftout. The FSD image shows strong crystallographic contrast within the thinned center region. Figure 2 shows a combined image quality and inverse pole figure orientation map from within the thinned region. High quality and indexable EBSD patterns were obtained from the majority of the thinned region. The sharpness of the boundaries observed within the image quality map decreases with increasing sample thickness. Figure 3 shows an FSD image at higher magnification from the center area of the thinned region with Figure 4 showing a corresponding image quality and inverse pole figure orientation map from approximately the same region collected with a 5 nm step size. These images show subtle differences in the observed microstructure. Within the black box in Figure 3, a twinned grain is observed. However, within the same region in Figure 4, no such grain is seen. This difference is most likely due to thickness effects and how the t-EBSD patterns are formed. The FSD image is produced by measuring the intensity of the signal landing on the FSD detector and is influenced by all the grains within the thickness of the sample, while the t-EBSD pattern is generated from the bottom-most grain within the sample thickness. The observation that t-EBSD patterns are formed from these grains helps explain the improvement in spatial resolution.

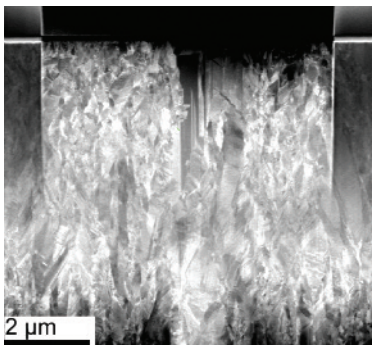


Figure 1. FSD image collected from a copper film sample prepared by FIB liftout, showing strong crystallographic contrast within the thinned center region.

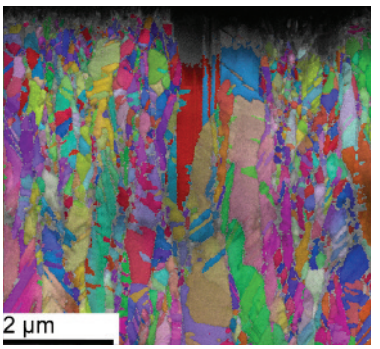


Figure 2. A combined image quality and inverse pole figure orientation map from within the thinned region.

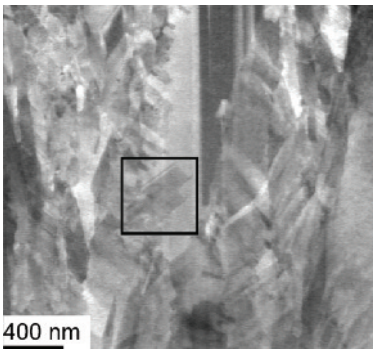


Figure 3. An FSD image at higher magnification from the center area of the thinned region.

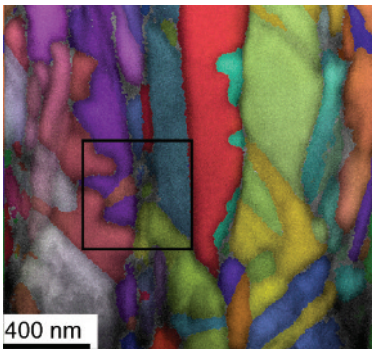


Figure 4. A corresponding image quality and inverse pole figure orientation map from approximately the same region collected with a 5nm step size.

Transmission-EBSD is an exciting new development that helps extend the spatial resolution of the EBSD technique using the same hardware and software used for traditional EBSD analysis. Spatial resolutions better than 10 nm have been reported, and this performance will help the fine-scale characterization of lower density materials such as aluminum and magnesium, as well as heavily deformed materials where reducing grain size is key to improving material properties.

Transmission-EBSD

With the decreasing size of grains and other microstructural features in crystalline materials the introduction of transmission-EBSD (t-EBSD) in the Scanning Electron Microscope (SEM) offers a characterization technique to close the gap between analysis in a SEM and a Transmission Electron Microscope (TEM). The benefit of t-EBSD is that it offers near TEM resolution, while still enabling the analysis of larger areas than is possible in the TEM. While the EBSD system used with standard EBSD (also called reflective EBSD) is the same for t-EBSD, the user selected settings used to scan a sample vary from standard EBSD.

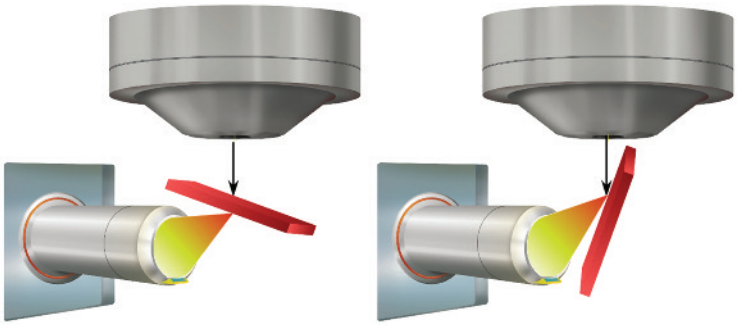


Figure 1. Schematics of t-EBSD (left) and standard EBSD (right).

Sample preparation – TEM/thin sample, resolution

An important part of sample preparation is first deciding if a sample is right for t-EBSD. While any crystalline sample that can be analyzed with standard EBSD can also be analyzed with t-EBSD, this technique was developed to analyze materials with features smaller than could be accurately studied with standard EBSD. Among others these features can include nanometer scale grains, small inclusions, and thin twins found in many developing materials. Additionally, preparation of specimens for t-EBSD requires the same processes as those for TEM, which produce samples thinner than ~200 nm.

SEM/EBSD/specimen geometry – tilt, working distance, specimen mount

With standard EBSD the SEM/EBSD/specimen geometry has been established for many years, but still operates within flexible tolerances of these parameters.

Recent studies with EDAX equipment have shown similar optimal geometry conditions for t-EBSD (see table).

	Standard EBSD	Transmission EBSD
Tilt Angle	70°	-45°
Working Distance	14 mm	5 mm
Accelerating Voltage	5 to 30 kV	15 to 30 kV

Figure 2. Typical conditions for standard EBSD vs. t-EBSD.

SEM e-beam parameters – kV, spot/current

In addition to geometry considerations, SEM e-beam parameters such as accelerating voltage (kV) and beam current significantly affect the quality of data collected with t-EBSD. Because the electron beam must pass through the sample in order to generate a signal on the EBSD detector, higher kV values are recommended. As samples become thinner, a lower kV is advised to create sufficient signal from the sample. A large enough current will also be required to create enough signal for the EBSD detector, but it should be kept low enough to avoid detrimental charging.

Image processing – background removal

Once the sample is prepared and in place, the EBSD images will require some image processing to optimize the pattern quality for data collection. One of the most important parts in this process is to collect and subtract a good background image due to the high intensity of the background image created with t-EBSD. With TEAM™ software this process is handled automatically by the smart camera features.

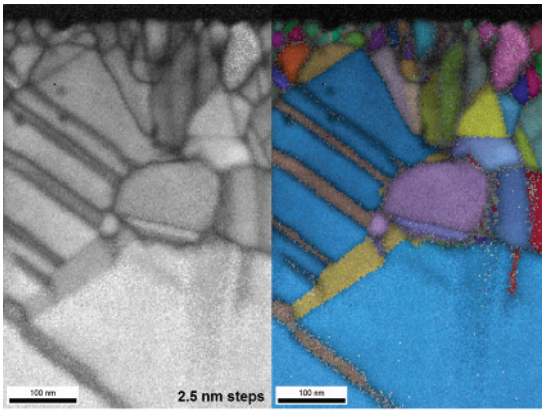


Figure 3. Image quality (left) and inverse pole figure (right) of a t-EBSD scan of Cu using a 2.5 nm step size.

APPLICATION NOTE

Elemental Analysis Through a Plastic Barrier

Most elemental analysis techniques require a sample to be exposed to the excitation source and/or placed under vacuum. For example, ICP-MS and atomic absorption (AA) require sample digestion or ablation and then exposure to the excitation source, while SEM-EDS requires exposure of the sample to the electron beam while under, at least, low vacuum. There are certain situations where it is highly desirable to leave the sample in a plastic barrier bag or analyze the sample at atmospheric pressure. For example, the analyst could be concerned about contamination from toxic, corrosive or radioactive samples or may need to prevent environmental contamination of the sample. For micro-spot elemental analysis, the Orbis micro-XRF spectrometer is unique as an elemental analysis tool in that the analysis can be done through a plastic bag and, if necessary, at atmospheric pressure.

Using SEM/EDS for analysis

- Sample must be removed from barrier bag for analysis via electron beam
- Analysis is done under vacuum conditions
- Wet sample outgassing and particle debris will put the SEM column at risk for contamination

Using ICP MS for analysis

- Sample must be removed from barrier bag for analysis
- Radioactively decaying materials can contaminate the equipment

In comparison, analysis with the Orbis Micro-XRF system offers an attractive alternative:

- Analysis can be performed through a plastic barrier bag and at atmospheric pressure, facilitating the analysis of powders and slurries
- Sample targeting through the barrier bag is simple and accurate with the perpendicular analysis geometry provided by the Orbis system
- Analysis can be made on small spots to measure inclusions and defects
- Both qualitative and quantitative analysis are possible and additional features such as primary beam filters extend the analytical detection limits

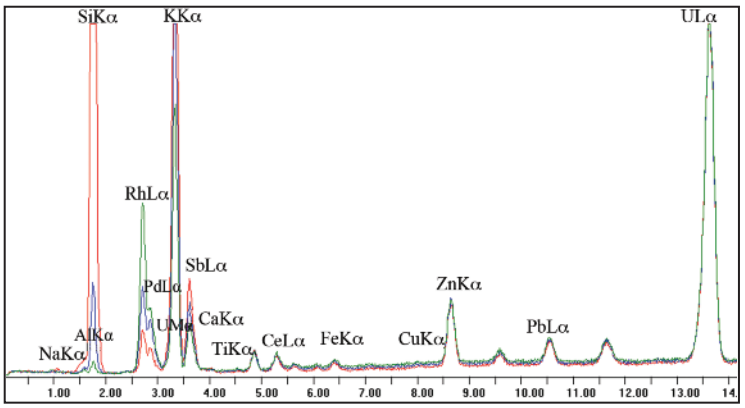


Figure 1. Analysis results for a uranium glass sample. Red - with no barrier; blue - enclosed in a single 50 µm bag; green - enclosed in two layers of 50 µm bag.

Experimental Conditions

Micro-XRF is performed using X-rays as the exciting source with fluoresced X-rays characteristic of the elements present as the signal. Lighter elements (e.g. Na - Ar) fluoresce lower energy X-rays, which are more readily absorbed or scattered while traveling through a plastic barrier or through air at atmospheric pressure. Heavier elements fluoresce higher energy X-rays which experience minimal attenuation while traversing typical plastic barrier materials or the atmospheric path to the detector. Hence, where it is desirable to keep a sample in a plastic containment bag and/or analyze the sample at atmospheric pressure, micro-XRF can provide valuable elemental analysis data even if there is a trade-off on the detection limits of lighter elements.

Results

The following analytical results include: a uranium rich glass, a mining standard powder, and moon rock, all performed under varying chamber pressure and all contained using plastic bags. The conditions for each sample are briefly described. In all the samples there is signal attenuation at the lighter emissions (<4 keV) while very little at the higher emissions.

Uranium Glass Overlay

Figure 1 shows three spectra with variation in the plastic bagging. The measured concentrations of uranium were approximately 1.68 wt% using the exposed sample in air. Red is the uranium glass exposed with no barrier, blue enclosed in a single 50 µm bag and green enclosed in 2 layers of 50 µm bag, for a total barrier of 100 µm. Above 4 keV there is little signal attenuation; however, below 4 keV there is distinguishable attenuation.

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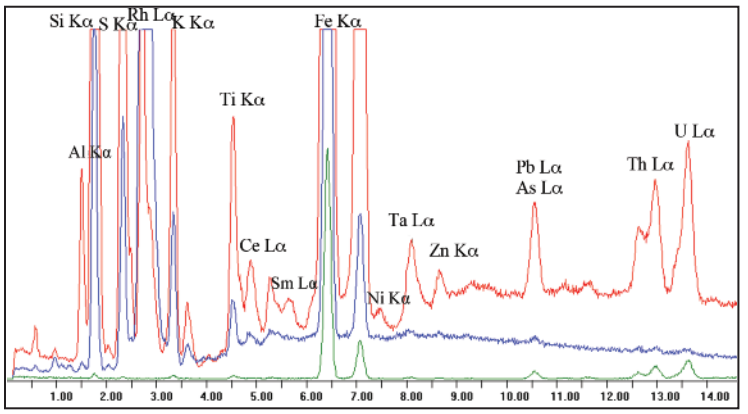

Figure 2. Analysis results for a Canmet DH-1 sample. Red - enclosed in thin Mylar envelope; blue - enclosed in 50 µm plastic bag; green - enclosed in 50 µm plastic bag using rhodium primary beam filter.

Mining Standard Powder

Figure 2 shows a mining sample powder of Canmet DH-1, which has a known concentration of uranium at 0.26 wt% compared with a measured concentration in the previous glass sample of 1.68 wt%. Here the red line represents the powder contained within a thinner Mylar envelope; blue within a thicker 50 µm plastic bag and green the same 50 µm sample bag using a rhodium primary beam filter which improves lower limits of detection for the uranium X-ray lines. Both the Mylar and blue 50 µm plastic bag samples were analyzed without a filter in place. By comparing the blue and green spectra it is evident that filtering improves the peak to background for the U(Lα) line allowing for improved detection limits while using the thicker barrier plastic.

Lunar Rock

Figure 3 shows a lunar rock composed of transition elements rather than higher energy actinides. The data is represented by elemental maps instead of spectral plots. Starting from the left, these are the results from an uncontained sample, followed by samples contained with 50.8 µm nylon, 50.8 µm Teflon, and 127 µm Teflon containment bagging with analysis done in vacuum. The higher intensity color indicates stronger signal intensity with less signal absorption by the barrier material. Even using the higher density Teflon barrier material in comparison to the nylon, it is possible to efficiently analyze lower energy transition elements with the barrier in place. The goal in this case was to evaluate the impact of using increasingly stronger gas tight containment bags on the elemental analysis via micro-XRF.

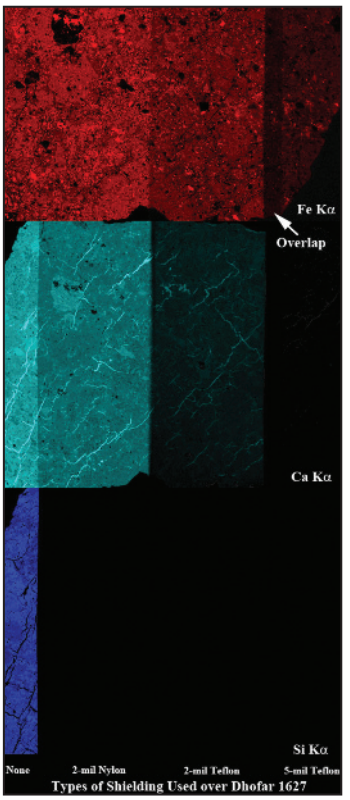

Figure 3. Analysis results for lunar rock with data represented by elemental maps.

Conclusion

The Orbis micro-XRF spectrometer has been used to provide valuable elemental analyses on samples contained within a barrier bag under atmospheric pressure and vacuum. Two capabilities have been shown here, single point analysis and elemental mapping. Where analysis of inclusions and defects or higher resolution mapping is of interest, the ultra-high intensity 30 µm polycapillary optic is particularly useful. Therefore, the Orbis PC configuration with this type of optic is recommended for this type of work. The standard hardware suite includes the primary beam energy filters used here for improved detection limits over limited spectral ranges as well as the automated stage for sample positioning and mapping. The Orbis can also be configured with two larger spot collimators along with the polycapillary optic for sample measurements more suited to a larger spot analysis.

EDAX Smart Insight

EVENTS AND TRAINING

Worldwide Events

February 2-6		**March 21**	
ACMM23 & ICONN2014	Adelaide, South Australia	Arizona Imaging & Microanalysis Society (AIMS)	Tucson, AZ
February 17-20		**May 11-13**	
Minerals, Metals & Materials Society Meeting (TMS)	San Diego, CA	Microscope Society Meeting	Chiba, Japan
February 27		**May 22-24**	
FIB SEM User Group	Laurel, MD	The Southeastern Microscopy Society (SEMS)	Greensville, SC
March 3-6		**June 8-13**	
Pittcon	Chicago, IL	Lehigh Microscopy School	Bethlehem, PA

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2014 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

March 13-14, 2014	Tilburg
June 19-20, 2014	Tilburg
TEAM™ EDS	
February 25-27, 2014	Tilburg
May 13-15, 2014	Wiesbaden
May 20-22, 2014	Tilburg
Genesis	
March 18-20, 2014	Wiesbaden
April 14-16, 2014	Tilburg

EBSD

March 10-12, 2014	Tilburg
June 16-18, 2014	Tilburg

Pegasus (TEAM™ EDS & EBSD)

February 17-21, 2014	Wiesbaden

TEAM™ WDS

April 1-3, 2014	Tilburg

Orbis: Course & Workshop Presented in English

May 6-8, 2014	Wiesbaden

JAPAN

EDS Microanalysis

Genesis	
February 13-14, 2014	Tokyo
April 10-11, 2014	Osaka
October 9-10, 2014	Tokyo
November 13-14, 2014	Osaka
TEAM™ EDS	
June 12-13, 2014	Tokyo
July 10-11, 2014	Osaka

CHINA

EDS Microanalysis

TEAM™ EDS	
March 17-20, 2014	Shanghai (ACES)
May 8, 2014	Beijing
June 9-12, 2014	Shanghai (ACES)
Genesis	
September 15-18, 2014	Shanghai (ACES)

EBSD OIM™ Academy

April 14-17, 2014	Shanghai (ACES)
October 20-23, 2014	Shanghai (ACES)

NORTH AMERICA

EDS Microanalysis

TEAM™ EDS	
February 4-6, 2014	Draper, UT
March 25-27, 2014	Mahwah, NJ
May 20-22, 2014	Mahwah, NJ

EBSD OIM™ Academy

March 11-13, 2014	Mahwah, NJ
June 10-12, 2014	Mahwah, NJ

Micro-XRF

April 8-10, 2014	Mahwah, NJ
October 7-9, 2014	Mahwah, NJ

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AMETEK®
MATERIALS ANALYSIS DIVISION

EMPLOYEE SPOTLIGHT

Udo Böttcher

In January 2006, Udo joined EDAX as a Sales Engineer in the eastern part of Germany, near Dresden. His sales area also includes parts of northern Germany, Bavaria and Austria.

Prior to EDAX, Udo worked in the department for analytical TEM research at the Institute for Solid State and Material Research, Dresden. In addition to performing TEM analysis, he learned Energy Dispersive Spectroscopy (EDS), Electron Energy Loss Spectroscopy (EELS), and other analytical techniques. In 1994, Udo joined Philips Electron Optics as a Sales Engineer for the eastern part of Germany.

Udo received a professional education as a steel worker, while he was completing his final secondary-school studies. He went on to earn a diploma in physics from the Technical University of Dresden.

Udo lives with his wife, Sylke in Freital. They have two children, David (30) and Julia (23). In his spare time, Udo enjoys reading detective stories, photographing Dresden and the surrounding areas, and playing goalkeeper on the senior football team. He has held a motorbike license since he was 17 years old.

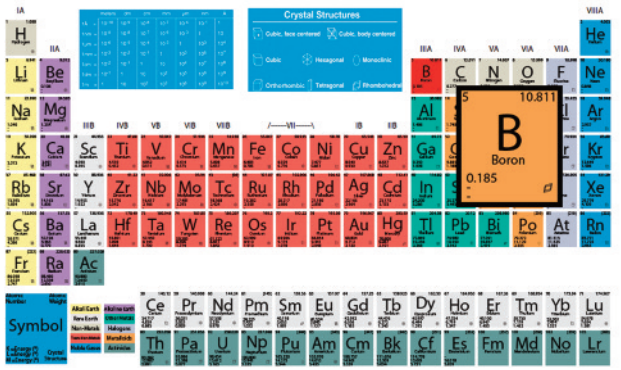
Narayan Vishwanathan

Narayan took over as Materials Analysis Division Vice President and EDAX Business Unit Manager in June 2013. Located in Mahwah, his responsibilities include product development, global expansion, strategy alliance, growth, and profit and loss management.

Narayan joins EDAX after serving as the Director of AMETEK's HDR Power Systems in Columbus, OH from 2008-13. Previously, he was the Director of Marketing at Solidstate Controls, an AMETEK subsidiary in Columbus, OH from 2005-08. For two years (2003-05), Narayan was a Product Manager at Ingersoll Rand in Charlotte, NC.

Narayan earned bachelor's and master's degrees in chemical engineering from the University of Mumbai in 1994 and 1996, respectively. In 2004, he received a master's in business administration from the University of South Carolina.

He lives with his wife, Isvarya Ramamurthy and one-year old son, Abhyuth "Abe". In his free time, Narayan enjoys reading, hiking and spending time with Abe.



New From EDAX

Explore new the interactive Periodic Table of Elements to see how Electron Backscatter Diffraction (EBSD) can be used to solve your materials characterization problems. By clicking on select elements you can view examples of how EBSD can be used to provide new insight into materials containing that element. The interactive periodic table can be accessed at:
http://www.edax.com/periodic-table/index.aspx

EDAX Smart Insight

CUSTOMER NEWS

University of California, Santa Barbara
Santa Barbara, California

Historically, acquisition of 3-D datasets for materials characterization, property prediction, and subsequent component/system design has been an extremely time-consuming and cumbersome process. This has resulted in system generational delays to the introduction of new materials into the engineering design process. A new approach to rapid acquisition of mesoscale-sized materials datasets has been developed by researchers at the University of California, Santa Barbara, with the support of the Office of Naval Research. The new TriBeam system (Figure 1), incorporates an ultrafast femtosecond laser beam with a Focused Ion Beam (FIB) in a Scanning Electron Microscope (SEM) platform that contains EDAX OIM™ Hikari XM4 Electron Backscatter Diffraction (EBSD) and Apollo X Energy Dispersive Spectroscopy (EDS) detectors.

Integration of the femtosecond laser provides unique capabilities due to the fact that the laser pulses interact with material on timescales that are substantially shorter than the timescale for physical damage processes (e.g. local melting or phase changes) normally encountered with nanosecond and picosecond pulsed lasers. Layer-by-layer femtosecond laser machining in the vacuum chamber, where the FIB can also be for precision milling or surface polishing, greatly expands the options for acquiring quantitative multimodal data at unprecedented speeds. In a typical sectioning experiment, a 1 cm^2 material slice 50 nm in thickness is removed in approximately 20s and quantitative chemical (EDS), crystallographic information (EBSD), structural information (SE), and/or qualitative chemical (BSE) is collected for the slice. Repeated slicing permits assembly of large, multimodal tomographic datasets. An example of a dataset of an interpenetrating Cu-W composite for high heat flux hypersonics applications is shown in Figure 2.

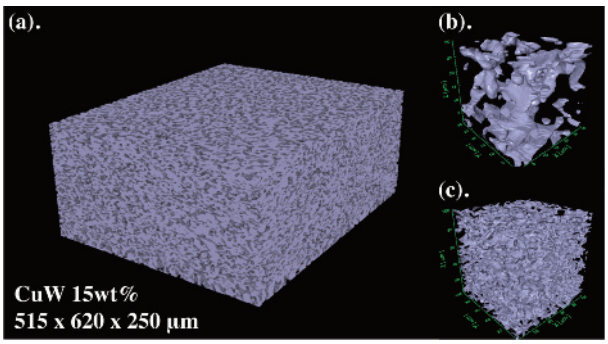


Figure 2. A TriBeam tomography dataset for a W-Cu composite showing the morphology of the Cu phase at full dataset size (left) and two smaller subsets (right).

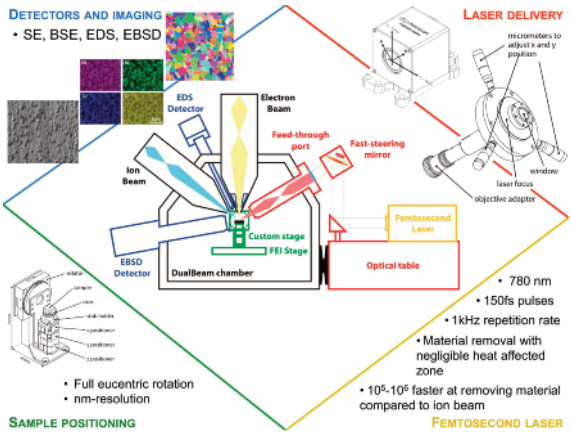


Figure 1. Schematic of the TriBeam System with major engineered components.

This dataset would require time periods of six months or longer to gather using conventional techniques, whereas this dataset has been collected in a matter of days in a fully automated fashion in the TriBeam system.

Published online 3 February 2012 in Review of Scientific Instruments (83 023701 (2012)), this work significantly advances the capabilities of material scientists to digitally catalogue material properties, supporting one of the goals of the National Material Genome Initiative. These types of advances in characterization tools will enable voluminous 3-D datasets that will be accessible via networked collaborative archives needed to support the engineering design of systems and components based on the desired fundamental material attributes required for a specific application.

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