EDAXinsight

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INSIDE

EDAX NEWS

Octane Elite - Sharpening Edges in Energy Dispersive Spectroscopy (EDS) Detection

With new electronics and its Si_3N_4 window, Octane Elite (cover image) adds a new dimension to EDS, particularly in the low energy range. In order to evaluate and measure Silicon Drift Detector (SDD) performance for sensitive and low energy applications, EDAX uses collection efficiency as an alternative metric based on X-ray transmissivity of the window and the solid angle. The collection efficiency of the SDD sensor is defined by an effective solid angle representing the total amount of X-ray signals saved in the spectra. This quantity is determined by several factors:

- The design of the preamplifier to control signal sensitivity and efficient conversion of signal output.
- The transmission properties of the window.
- The design of the window support grid and the whole mechanical design of the sensor module, including the internal and external collimators and module vacuum.
- The size and geometry of the SDD detector.

To highlight the difference between the traditional solid angle metrics, which define the input signal to the detector, and the collection efficiency of the sensor, our applications team conducted a simple experiment.

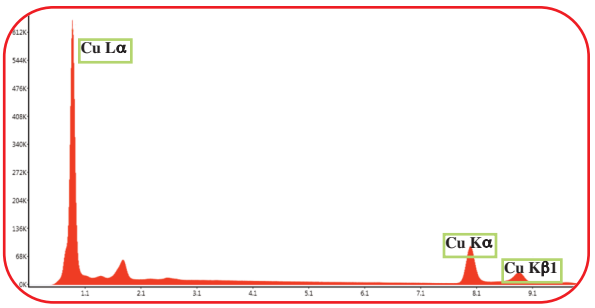

Figure 1. Cu grid spectrum acquired with an Octane Elite 30 mm² SDD at 650 kcps OCR and 40% dead time.

In this experiment, the team mounted two different EDS spectrometers on the same Scanning Electron Microscope (SEM) column at exactly the same working distance from the sample. A standard Transmission Electron Microscope (TEM) copper grid was chosen as the sample. The first EDS system included the new Octane Elite 30 mm² SDD with vacuum encapsulated sensor module and a Si_3N_4 window, while the second system had an Octane Plus with a 60 mm² sensor module and a traditional polymer Super Ultra Thin Window (SUTW).

(Continued from Page 1)

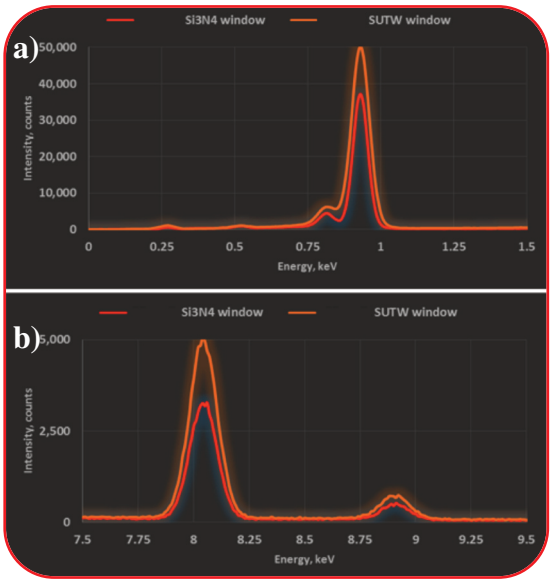

Figure 2. (a) Cu L spectra and (b) Cu Kα and Cu Kβ acquired by both detectors.

The EDS spectra for both spectrometers were collected in parallel from the same location in the sample. The data was acquired at 15 kV with 7,680 μs amp time and an acquisition time of 50 seconds (Figure 1).

The spectra for Cu L (Figure 2a), and Cu Kα and Cu Kβ (Figure 2b), acquired at these conditions, were used to measure and compare the intensity (counts) of the Cu peaks. The intensity was measured both at the maximum height of the peaks at the characteristic X-line energies: 0.93 keV (Cu L), 8.04 keV (Cu Kα), and 8.89 keV (Cu Kβ) and using the integrated area under the peaks (Table 1).

The collected X-ray counts for Cu L and Cu K, measured using peak area and peak height methods, were calculated in cps, cps/mm^2 and cps/nA/mm^2 using a collection time of 50 seconds, beam current of 0.5 nA, and 30 mm^2 and 60 mm^2 sensor areas (Tables 1 and 2).

			Si_3N_4 30 mm^2	SUTW 60 mm^2
Method	**Line**	**Integration Range (keV)**	**Intensity, Counts**	**Intensity, Counts**
Peak Area	Cu L	0.69-1.01	281357	453348
Peak Area	Cu Kα	7.84-8.24	52742	81817
Peak Area	Cu Kβ	8.75-9.05	8931	13428
		Intensity at (keV)		
Peak Height	Cu L	0.93	37239	50335
Peak Height	Cu Kα	8.04	3260	5067
Peak Height	Cu Kβ	8.89	504	720

Table 1. Intensity data for the Cu L and Cu K lines.

For the high energy range (Cu K), the count rate normalized per sensor area for the Si_3N_4 30 mm^2 detector was 29% higher than the collection efficiency of the SUTW 60 mm^2 detector. This can be interpreted as the effective solid angle for the new detector being equivalent to the solid angle of a 39 mm^2 sensor based on the SUTW design (30 mm^2 x 1.29 ≈ 39 mm^2). The same analysis for the low energy range (Cu L), increases the difference to 42% (equivalent to ≈ 42 mm^2 SUTW sensor area), which emphasizes the increased sensitivity of the new sensor for light element detection.

The output of this experiment, which demonstrates the evaluation of the SDD performance based solely on the sensor size, proves that geometrical solid angle alone is not adequate for determining signal detection capability. Alternative metrics using the effective solid angle or collection efficiency are more accurate for comparing SDD performance.

In conclusion, a comparison of the SDD performance with different geometries indicates that sensor size alone is not an adequate metric. Instead, the collection efficiency of the sensor is a more accurate reference in regards to the signal detection capability. Conceptually, employing a smaller sensor with optimized signal collection efficiency at higher count rates does offer an attractive alternative to reach high throughput data acquisition with improved energy resolution.

			Si_3N_4 30 mm^2	SUTW 60 mm^2	Ratio for cps/mm^2	Si_3N_4 30 mm^2
Method	**Line**	**Integration Range (keV)**	**cps/mm^2**	**cps/mm^2**		**cps/nA**
Peak Area	Cu L	0.69-1.01	187.6	151.1	1.24	11254
Peak Area	Cu Kα	7.84-8.24	35.2	27.3	1.29	2110
Peak Area	Cu Kβ	8.75-9.05	6.0	4.5	1.33	357
					1.29	
		Intensity at (keV)				
Peak Height	Cu L	0.93	24.8	16.8	1.48	1490
Peak Height	Cu Kα	8.04	2.2	1.7	1.27	130
Peak Height	Cu Kβ	8.89	0.3	0.2	1.50	20
					1.42	

Table 2. Intensity data renormalized with respect to sensor area and probe current.

TIPS AND TRICKS

Using Manual Phase Mapping to Find Gold

EDAX TEAM™ Phase Mapping is an established and powerful automated software routine with the capability of supporting a large number of Energy Dispersive Spectroscopy (EDS) mapping applications. The automated routine is known for investigating the spectrum from each pixel, looking at the spectral peaks and intensities, and grouping each spectrum with all chemically similar spectra. The result is a spectrum based image that is colored according to its chemical makeup.

The TEAM™ Phase Mapping software also includes several manual options, which put even greater capability into the hands of the microanalyst. The first manual function creates a user generated spectrum as a representative phase, rather than the software generated phase. Any sample can be used to create a manual spectrum phase, including standards. For example, a mineralogist may want to load a simple SiO_2 standard, collect a spectrum, and then use this as his quartz phase for mapping. Likewise, any spectrum can be extracted from a map and then sent to the manual phase library (Figure 1). Manual phases can be added to an auto phase library or a library can be purely a manually generated library.

Figure 1. Use the "Create Phase from Spectra" icon to send an active spectrum to the phase mapping library.

Once phases are generated, this library can be built up, stored and reloaded at any time (Figure 2). It can even be shared between different TEAM™ systems and sites. A great advantage of this mode is that the analyst can change a phase color according to his preference or some other convention. Whenever this phase map library is applied to future maps, the phases will all have the same colors as previous maps. This makes phase map interpretation much easier over time, as the analyst will get used to seeing certain phases as certain colors and can recognize their presence immediately.

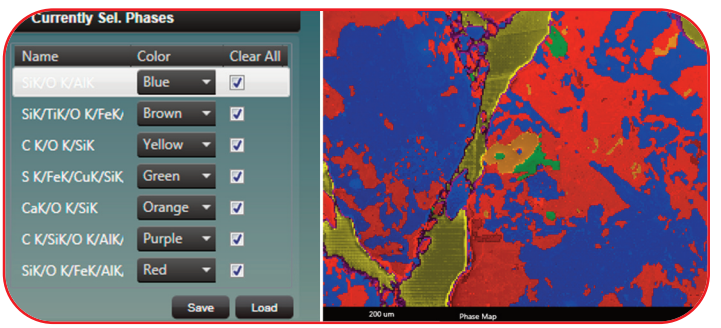

Figure 2. Auto or manually generated phase libraries provide the option to change the colors and to save or reload the library on the same EDAX system or on different EDAX TEAM™ systems.

In the example used here, a phase map was generated automatically, as seen in Figure 2. The goal of this analysis, however, was not to look for all phases, but for only one particular phase of interest, chalcopyrite, of which pyrite, or fool's gold is one form. In the automatically generated map, the chalcopyrite compound was colored green and labeled as S K/Fe K/Cu K. In order to search for and display only this compound, all other phases were excluded and the S/Fe/Cu phase alone was saved into a new library, and with a gold color designation. Additionally, the manual control to "lock" phases was selected for the rebuild (Figure 3). Therefore, when the phase map was rebuilt, all pixels matching the S/Fe/Cu spectrum based phase were colored gold and all other non-matching pixels were left gray.

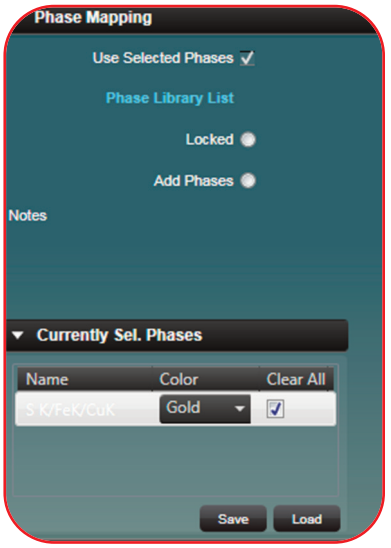

Figure 3. Shows the manually adjusted map for only the chalcopyrite, colored gold and in locked mode.

The resulting rebuilt map in Figure 4 shows only those pixels whose spectra match the chalcopyrite spectrum within the fit tolerance. We found "gold!"

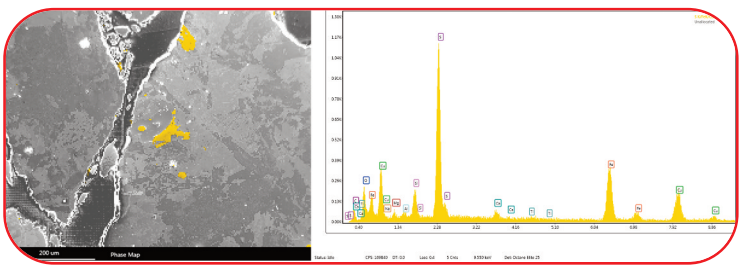

Figure 4. Shows the rebuilt map highlighting all pixels that match the chalcopyrite phase spectrum.

EDAX Smart Insight

APPLICATION NOTE

OIM™ Analysis v8 - The Best Just Got Better

OIM™ Analysis has been accepted by many analysts as the premier microstructural visualization and analysis tool for interrogating and understanding Electron Backscatter Diffraction (EBSD) mapping data. With greyscale and color mapping options for displaying orientations, grain boundaries, phases, Energy Dispersive Spectroscopy (EDS) information, and local deformation, OIM™ Analysis presents data visually in colorful and meaningful maps. Data can also be quantified and compared using the charting options for grain size, grain shape, and misorientations. Specific microstructural features can be investigated with interactive highlighting. Individual microstructural components, such as high confidence points or deformed regions, can be easily extracted, analyzed, and compared both to other components and to all the collected data, with partitioning. This rich toolbox of features allows users to answer common characterization questions and develop new insights in cutting-edge research.

The capability and performance of this tool has been significantly enhanced and improved with the release of OIM™ Analysis v8. New features have been added to provide new approaches to characterization and the underlying routines have been optimized to make analysis faster.

Multithreading

With the introduction of OIM™ Analysis v8 many of the algorithms used in map rendering and microstructural characterization calculations have been optimized for multithreaded operations to take advantage of modern multi-core CPUs. For many functions, the performance improvements scale with the number of available cores. For example, rendering a large mapping image with an eight core CPU will be approximately eight times faster than previous versions of OIM™ Analysis on the same computer.

Pattern Indexing

One of the most significant features added in OIM™ Analysis v8 is the ability to index EBSD diffraction patterns. Traditionally, this functionality has been contained within the EBSD collection software. Its introduction into the OIM™ Analysis framework, now allows users to take advantage of the combination of the analytical power of OIM™ Analysis and the indexing performance of the EDAX triplet indexing routine.

One application would be the identification of the low-confidence points in OIM™ Analysis using the partitioning function. The patterns associated with these points could then be evaluated to determine why the indexing performance was poor. If it was determined that these points belonged to a previously unspecified phase, these points, and these points only, could then be reindexed using the appropriate phase file. If the poor indexing performance was due to EBSD pattern quality, these points could then be reindexed after applying a new background processing routine or the patented Neighbor Pattern Averaging and Reindexing (NPAR) approach to improve indexing performance.

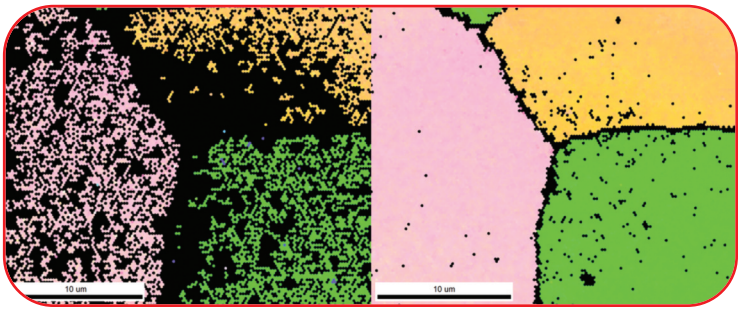

Figure 1. EBSD orientation map from a nickel superalloy collected at high speed, low beam current conditions before NPAR (left) and after NPAR processing (right). NPAR provides significant improvement in indexing success.

Another application, Chi-Scan, is the use of simultaneously collected EDS-EBSD data to differentiate phases. Traditionally, the EDS and EBSD data was collected simultaneously and then chemical phases were determined via the EDS data and the appropriate EBSD structural phases were associated with the chemical phase. Then, all the data was reindexed using the structure appropriate for each point, based on chemistry. With OIM™ Analysis v8, specific phases can be targeted for reindexing, making the final characterization much more efficient.

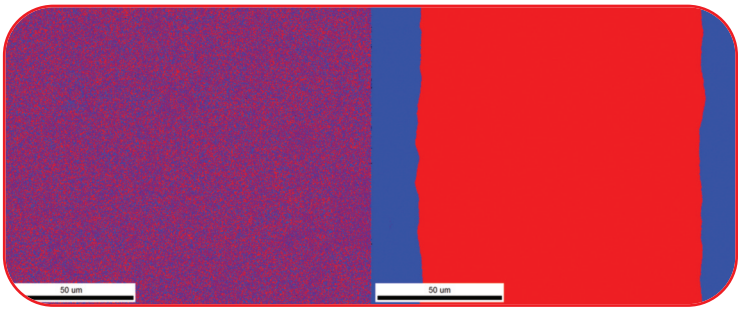

Figure 2. EBSD phase map from a two phase microelectronic component with similar crystallographic phases, Fe-Ni (FCC - Red) and Copper (FCC - Blue). It is difficult to resolve phase structure correctly by EBSD alone (left) but when combined with EDS the phase structure is correctly identified using Chi-Scan (right).

(Continued from Page 4)

Batch processing has also been implemented so that multiple scans can now easily be reindexed, with Chi-Scan and NPAR as batch options. This functionality is extremely useful for 3D or in-situ EBSD experiments, where multiple scans are collected serially, and batch reprocessing is an efficient method of preparing this data for analysis.

Anti-Grains

Traditionally, measurement points with similar crystallographic orientations (typically defined with a grain tolerance angle of 5°) are grouped together as grains and then metrics, such as grain size and shape, are calculated. Often as part of this analysis, points with a low confidence index (generally below 0.1) are excluded. These low confidence points can be caused by a number of factors, including porosity in the sample and regions where diffraction patterns are not obtained (amorphous or ultra-fine grained materials). With OIM™ Analysis v8, new functionality has been implemented to allow these low confidence points to be grouped together in a similar fashion as grains. In this case, spatially-adjacent low confidence points are grouped together as anti-grains. Once anti-grains are determined, the size and shape distributions are calculated, and provide another microstructural characterization metric for materials.

Correlation Plots

OIM™ Analysis has always provided distribution charts and statistics of different measurement values, such as image quality, confidence index, grain size, and local misorientations, including kernel average misorientation (KAM) and local orientation spread (LOS). With OIM™ Analysis v8, users can now plot correlated values of these measurement metrics, which can lend new insight into the measured microstructure.

Correlative Microscopy

The microscopy community has experienced increased interest in correlating one characterization technique with another to better describe a sample of interest. New correlative microscopy features have been introduced in OIM™ Analysis v8 to better facilitate this type of analysis. With these features, users can import spatially-specific mapping measurements from other techniques (CL, Nanoindentation, AFM, etc) and register and align these measurements with the EBSD mapping data (and any simultaneously collected EDS data). The structural EBSD data can then be analyzed in conjunction with this complementary information to gain new insights and understandings.

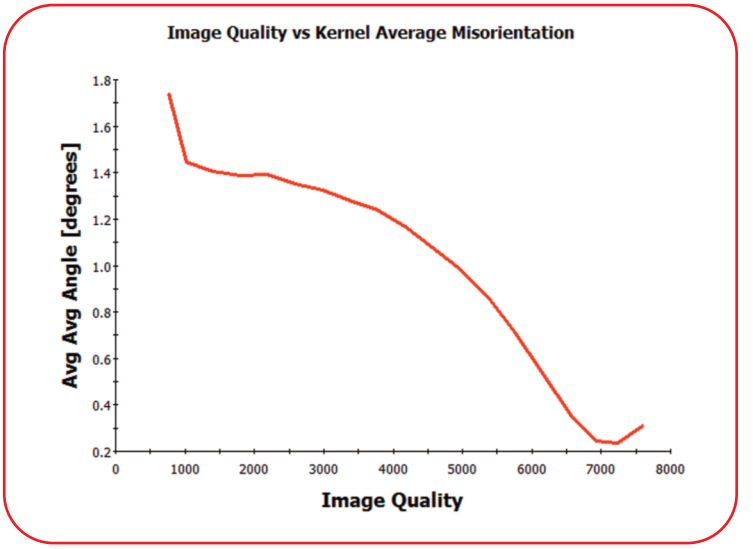

Figure 3. Correlation plot of Image Quality vs. Kernel Average Misorientation (KAM) for a dual phase steel microstructure. This plot indicates that in general, as pattern quality improves the local misorientation decreases, as expected for a ferrite-martensite structure.

HDF5 Support

With the development of new EBSD applications including Pattern Region of Interest Analysis System (PRIAS), NPAR, and High-Resolution (HR)-EBSD analysis, there is more driving force than ever to save EBSD patterns collected during map acquisition. However, as the number and/or size of EBSD patterns saved increases, it can become difficult to save these patterns individually. With the TEAM™ EBSD software, EBSD patterns can be saved to a *.pat file format. Now with OIM™ Analysis v8, these saved patterns can also be saved into the HDF5 file format for easier storage and access. This file format is compatible with Cross Court for HR-EBSD analysis and Dream3D for 3D analysis.

Summary

OIM™ Analysis v8 provides users with new functionality and performance that will enable them to further investigate the crystallographic microstructure of materials with greater efficiency.

EVENTS AND TRAINING

2016 Worldwide Events

July 24-28		**September 7-9**	
Microscopy & Microanalysis (M&M) 2016	Columbus, OH	Japan Analytical & Scientific Instruments Show	Chiba, Japan
August 1-5		**September 7-9**	
Denver X-ray Conference	Rosemont, IL	Semicon Taiwan 2016	Taipei, Taiwan
August 14-19		**September 21-23**	
International Materials Research Congress	Cancun, Mexico	Metallographie	Berlin, Germany
August 28-September 2		**September 25-29**	
European Microscopy Congress (EMC) 2016	Lyon, France	GeoTirol 2016	Innsbruck, Austria

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

2016 Worldwide Training

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

EUROPE

EDS Microanalysis	
TEAM™ EDS	
September 26-28	Weiterstadt#
November 1-3	Tilburg*
November 14-16	Weiterstadt#
November 29-December1	Weiterstadt#
December 6-8	Tilburg*
Microanalysis	
September 15-16	Tilburg*
November 10-11	Tilburg*
TEAM™ EBSD	
September 12-14	Tilburg*
November 16-18	Weiterstadt#
November 28-30	Tilburg*
TEAM™ Neptune (EDS & WDS)	
September 26-30	Tilburg*
TEAM™ Pegasus (EDS & EBSD)	
September 12-16	Tilburg*
November 14-18	Weiterstadt#
TEAM™ WDS	
November 22-24	Tilburg*
XRF	
October 11-13	Tilburg*

JAPAN

EDS Microanalysis	
Genesis	
October 6-7	Tokyo
November 10-11	Osaka
OIM™ School	
Advanced	
September 12-13	Tokyo
Entry	
November 8-9	Tokyo

CHINA

EDS Microanalysis	
TEAM™ EDS	
September 6-8	Shanghai
December 6-8	Shanghai
TEAM™ EBSD	
November 8-10	Shanghai

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
September 20-22	Mahwah, NJ
TEAM™ EBSD	
October 25-27	Draper, UT
XRF	
October 4-6	Mahwah, NJ

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AMETEK®
MATERIALS ANALYSIS DIVISION

(left to right): Jim, Luke, Allison, and Carly Tebolt.

Jim Tebolt

Jim joined EDAX in November 2013 as the Manufacturing Supervisor of the Mahwah, NJ facility. He is responsible for scheduling the work force to meet the delivery requirements of production, as well as service orders.

Prior to EDAX, Jim was the Quality Manager for a manufacturer of Fluid Control Systems for 12 years. He has also held positions of Project Manager, Cost Estimator, and Buyer for a contract manufacturer.

Jim holds a Bachelor of Science Degree in Business Administration from Syracuse University/Utica College.

Jim and his wife, Allison, have been married for 22 years and reside in Dutchess County, New York with their two children. They enjoy watching their kids participate in sporting activities. Their daughter, Carly, played high school field hockey, lacrosse, and track. She is a member of the field hockey and track teams at Utica College, where she just completed her freshman year. Their son, Luke, played high school baseball and basketball. He will continue to play baseball at Manhattanville College, where he will be a freshman this fall. Jim and Allison also like hiking, mountain bike riding, and gardening.

Visit EDAX at Booth #1114
at M&M 2016 in Columbus, OH
July 24-28, 2016

For more information visit: http://www.edax.com/MM2016

Sophie Yan

Sophie Yan

Sophie joined EDAX in July 2015. She is the Application Engineer/Scientist in Shanghai, China. Sophie's duties include sales support in China, visits to potential customers, performing product demonstrations, and attending and presenting at conferences and workshops. The Shanghai office provides her with the perfect combination of a flexible work style with the comfort of a humid climate.

Prior to EDAX, Sophie was an Application Specialist for Carl Zeiss Microscopy in Beijing, China from 2012-15. She handled focused ion beam (FIB) applications, including FIB milling, transmission electron microscope (TEM) sample preparation, 3D reconstruction, and Energy Dispersive Spectroscopy (EDS)/Electron Backscatter Diffraction (EBSD) support. Although she enjoyed the position, Sophie was forced to leave Beijing due to the air pollution. From 2008-12, Sophie worked as a Failure Analysis Engineer at Intel in Dalian, China. She was in charge of daily operation of the semiconductor fabrication laboratory and specialized in failure location and scanning electron microscope (SEM)/FIB operation.

Sophie earned her bachelor's degree from Wuhan University of Technology. Later she received a Ph. D. from the Chinese Academy of Sciences, Shanghai Institute of Ceramics.

In her spare time, Sophie enjoys reading novels. She finished all of the famous martial arts novels during her time in high school and college. Sophie attempted to write a novel while in college, but never finished it. She also likes poetry, different handwriting, and antithetical couplet at exhibitions. One of Sophie's favorite Chinese sayings is, "Beyond the daily grind of mundane existence, there are still poetry and those far off places".

EDAX® Smart Insight

Materials Research Institute Aalen Germany

Last November, many regional business partners and company representatives were excited to visit the new laboratories and instruments at the Materials Research Institute (Institut für Materialforschung (IMFAA)) in Aalen, Germany. The new facilities and equipment allow Aalen University to perform research activities at a very high level. The total purchase of upgrades to the facility, subsidized by the Federal Ministry of Education and Research (Bundesministerium für Bildung und Forschung), the Federal Ministry of Economy and Energy (Bundesministerium für Wirtschaft und Energie), German Research Foundation (Deutsche Forschungsgemeinschaft - DMG), and the state of Baden-Wurttemberg, amounted to about €2.1 million. The highlight of the event was a guided tour of the IMFAA's laboratories and workshops.

The funding enabled IMFAA to invest in two new Carl Zeiss Microscopy high-resolution electron microscopes to further enhance its tools used for elementary and structural analysis. The Carl Zeiss Crossbeam 540/Laser microscope is equipped with an EDAX TEAM™ Pegasus Analysis System with an Octane Plus Silicon Drift Detector (SDD) and DigiView Electron Backscatter Diffraction (EBSD) camera, as well as laser and ion-beam nano-processing for analyzing sample volumes in three dimensions. The second microscope is a Sigma 300 VP with an EDAX TEAM™ Trident Analysis System for Energy Dispersive Spectroscopy (EDS), EBSD and Wavelength Dispersive Spectrometry (WDS) analysis. The systems provide IMFAA with the ability to perform in-depth analysis on various new materials for applications such as lithium-ion batteries used for power storage, magnetic materials for electric drives, high performance tools for cutting and machining, and more common analysis of steel materials.

Figure 1. Scientists working in the new IMFAA laboratory equipped with two Carl Zeiss Microscopy high resolution scanning electron microscopes and EDAX TEAM™ Pegasus and TEAM™ Trident Analysis Systems.

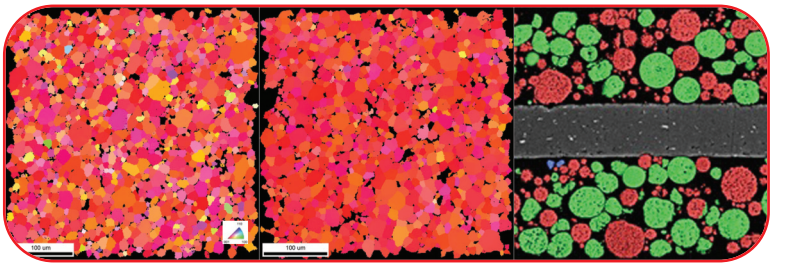

Figure 2. EBSD maps created using the EDAX Hikari Super EBSD camera on two sintered magnets with different grades of degree of grain alignment: strong (left) and smooth misalignment (middle). The right image shows a phase map of a cathode collector foil of a Lithium ion battery based on an EDS elemental map using the EDAX Octane Plus SDD. The green areas are lithium nickel cobalt aluminum oxide and the red regions are lithium manganese oxide.

The institute has made use of texture analysis using the Sigma 300 VP and TEAM™ Trident System to obtain excellent representation and quantification of the degree of preferred orientation of magnetic grains in sinter magnets for electrical devices. The EBSD mapping data can be used to detect local inhomogeneity and to calculate a texture parameter, quantifying the grain alignment. IMFAA has also analyzed blend cathodes that consist of mixtures of various lithium metal oxides. The scientists use the data to determine the volume foil phase fraction and distribution of the active materials on the collector and thus correlate performance and processing issues.

There are very few laboratories across Germany as equipped to handle microscopy and materials analysis as Aalen University. This is emphasized by the fact that the University has been honored as the strongest research university for applied sciences in the state of Baden-Wurttemberg for each of the past nine years.

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