INSIDE

EDAXinsight

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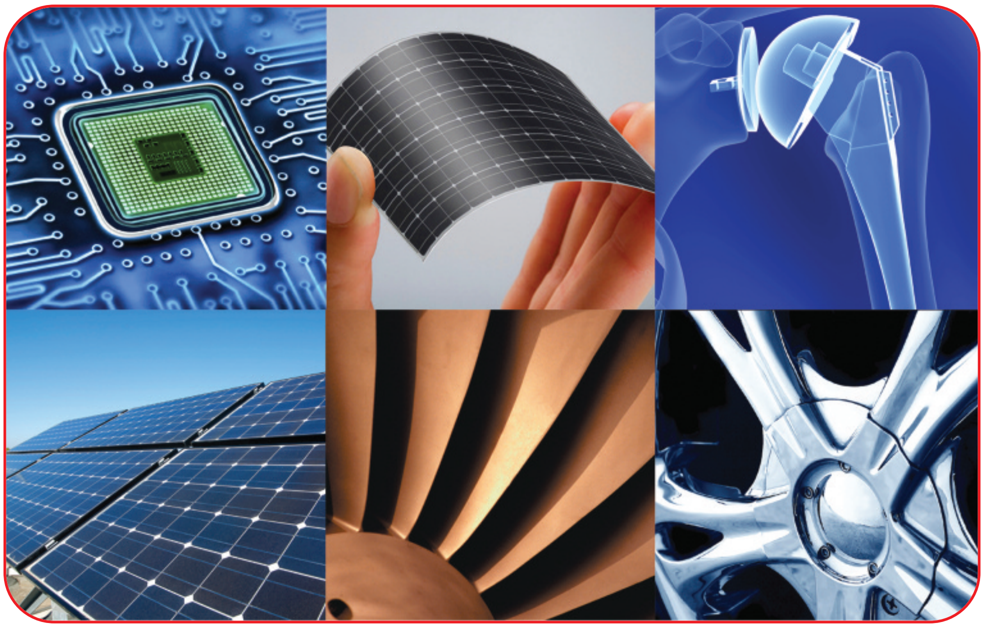


EDAX NEWS

EDAX Launches the XLNCE X-ray Metrology Series

Now that the launch of the XLNCE product line at Pittcon 2015 is behind us, we are looking forward to communicating and explaining the analysis opportunities that the instruments offer to potential customers in the upcoming months. Moving from strong laboratory based applications historically, the addition of the XLNCE line of Energy Dispersive X-ray Fluorescence (EDXRF) analyzers expands EDAX's X-ray Fluorescence (XRF) portfolio into the field of X-ray Metrology for process control applications.

The SMX-BEN is the first model of the XLNCE X-ray Metrology product line being offered for non-destructive analysis of thin film layer thickness and composition measurement applications. SMX-BEN is a versatile EDXRF analyzer designed for R&D, process development, recipe formulation, process control, and failure analysis.

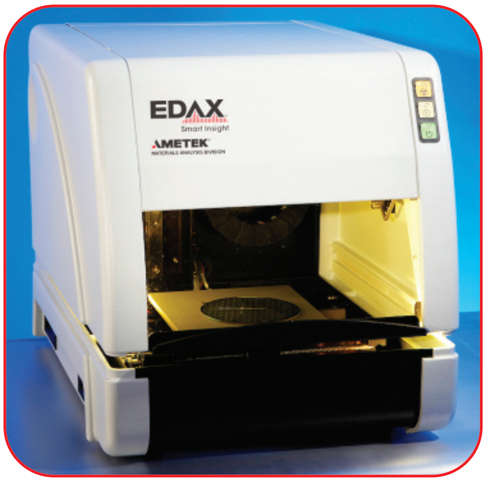


Figure 1. XLNCE SMX-BEN XRF Analyzer.

The XLNCE SMX products are specifically designed for coating applications and offer large analysis chambers, with motorized and programmable X-Y-Z stage positioning. They are all equipped with the latest Silicon Drift Detectors (SDD) with an array of choices for primary filters and X-ray optics.

AMETEK®
MATERIALS ANALYSIS DIVISION

EDAX®
Smart Insight

(Continued from Page 1)

The SMX systems already have a strong foothold in the energy market with photovoltaic (PV) solar applications, where the measurement of multi-layered compositions such as copper indium gallium selenide (CIGS) over molybdenum (Mo) is essential for the production of the solar panels.

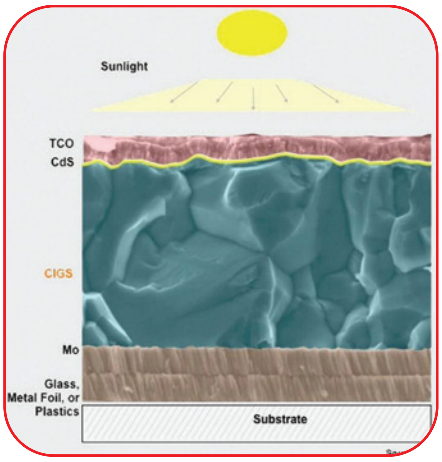


Figure 2. The CIGS layer of a photovoltaic solar cell.

The SMX analyzers are capable of performing coating layer thickness and composition analysis of up to eight layers and 30 elements, with thickness ranging from less than a nanometer to microns. They offer a high degree of accuracy and precision on rigid or flexible substrates, using an advanced software algorithm as the base of operation.

Even though the off-grid solar market has been on a steady rise and is being viewed as a legitimate industry with growth potential for the future, the possibilities for the XLNCE product line go beyond photovoltaic solar applications. The XLNCE products provide excellent utility for wafer metallization, rigid and flexible printed circuit board (PCB) bump analysis, corrosion protection, medical implants, thermal barriers, automotive, and many more applications where coating analysis and control are essential for performance and durability.

The desire to control manufacturing parameters in large production facilities creates a demand for in-line applications, where yield management is essential. The cost associated with production of out of specification materials justifies investment in process control instrumentation, hence an opportunity for in-line X-ray metrology tools.

The early response from the market for the XLNCE product line clearly validates a trend towards the need for an in-process instrument that complements the SMX-BEN model. This investment is based on the economics of fabricating scrap material due to process failure.

The modularity of the SMX-BEN design and its strong analysis software platform provide a base for in-line applications where the SMX systems can be utilized in production for process control, quality assurance, yield management, and failure analysis.

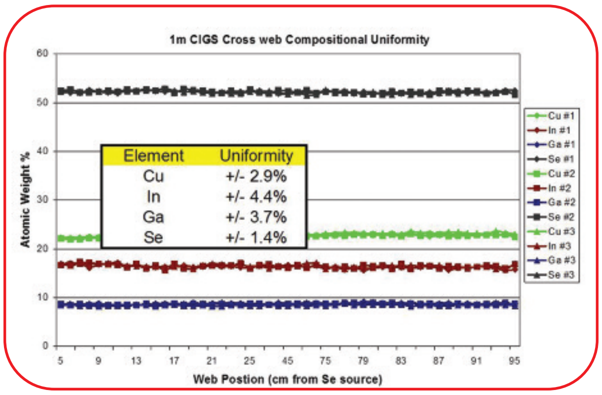


Figure 3. Deposition results. Cross web compositional uniformity (100 cm web width data taken at three locations down web, 1 m spacing).

In the very near future, EDAX is planning to introduce the XLNCE SMX-ILH XRF system to address the market's demand for a viable XRF tool for production, where measurement, analysis, and control of the coating composition and thickness is paramount to the quality and material specifications.

The SMX-ILH, with many practical design features, is equipped with exactly the same measurement module as the SMX-BEN. It is a platform for XRF analysis in an atmospheric environment, either near-line or in-line and provides process control and yield management for flexible web substrate materials, such as stainless steel or polyimides, as well as rigid substrates such as float glass.

The introduction of the SMX-BEN and subsequent offering of the SMX-ILH model will create many opportunities and challenges in the future. As EDAX expands its XRF product line, the company looks forward to growing and establishing a stronger presence in the industrial market place.

Figure 4. XLNCE SMX-ILH.

Spectrum Auto Processing

Have you ever wanted to make changes to a large series of spectral data? For example, have you ever realized only after collection that you wanted to remove the coating element label from coated samples and not quantify them with the current element list? Or has someone reviewing your data ever wanted to add an element to be searched for in all the data you have already collected? If so, then the TEAM™ EDS Spectrum Auto Processing routine is just the software feature for you.

The auto processing function is a standard part of any TEAM™ Enhanced system. With it, you can add an element or elements to all of your spectra, remove an element from all of your spectra, or even make edits to the master element list, regardless of what elements have already been identified.

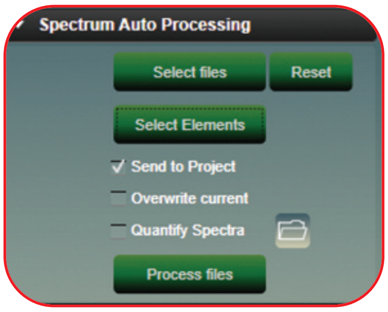


Figure 1. Advanced Properties tab.

First, start by activating the feature in the Advanced Properties tab (Figure 1).

Next, select the project, samples and spectra that you'd like to process (Figure 2).

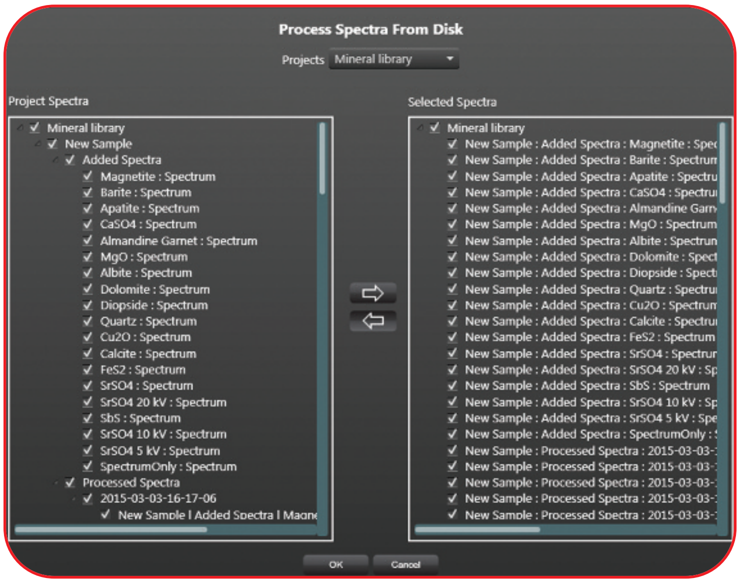
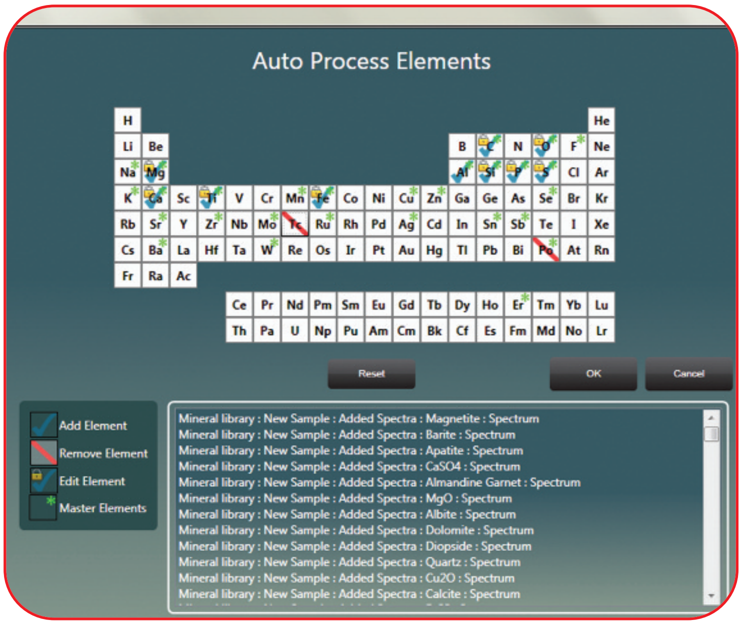


Figure 2. Project, samples and spectra selection.

Then, open up the interactive periodic chart to add, remove or modify your elements (Figure 3).



Mineral library : New Sample : Added Spectra : Magnetite : Spectrum
Mineral library : New Sample : Added Spectra : Barite : Spectrum
Mineral library : New Sample : Added Spectra : Apatite : Spectrum
Mineral library : New Sample : Added Spectra : CaSO4 : Spectrum
Mineral library : New Sample : Added Spectra : Almandine Garnet : Spectrum
Mineral library : New Sample : Added Spectra : MgO : Spectrum
Mineral library : New Sample : Added Spectra : Albite : Spectrum
Mineral library : New Sample : Added Spectra : Dolomite : Spectrum
Mineral library : New Sample : Added Spectra : Diopside : Spectrum
Mineral library : New Sample : Added Spectra : Quartz : Spectrum
Mineral library : New Sample : Added Spectra : Cu2O : Spectrum
Mineral library : New Sample : Added Spectra : Calcite : Spectrum

Figure 3. Add, remove or modify elements from the interactive periodic chart.

- Adding an element will add it to all spectra, whether or not it was there already.
- Removing an element will remove it from all spectra.
- Editing the master list will change all of the spectra element lists to that list at once.

Once you have created all of the element changes that you would like to apply to any series of selected spectra, you can then choose to send these modified spectra back to the project, with the option to overwrite the current ones. If not, the project tree will now include an added area for the processed spectra.

And finally, for any users who want to process and quantify large batches of spectra, you no longer have to drag and drop multiple spectra. Simply click on the Quantify Spectra checkbox and assign a file name and location. This will prepare the spectra to be saved into one Microsoft Excel spreadsheet. When you are finally ready to activate all of these processes, click the Process files button.

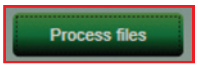


Figure 4. Process files button.

And the Auto Processing feature will do all this work for you!

Analysis of Pb-Free Solder and Solder Films with the XLNCE SMX-BEN XRF Analyzer

Materials Challenge

Pb-free solders became prominent in electronics manufacturing with the advent of the European RoHS/ELV regulations. X-ray Fluorescence (XRF) is a non-destructive, non-contact atomic spectrometry, which is ideal for measuring the composition of major, minor and trace components in Pb-free solders as well as the thickness of solder films. The RoHS directive limits the concentration of Pb in solder to less than 1000 ppm. The formulation of Sn-based Pb-free solders use several other alloying elements, including Ni, Cu, Bi, Ag, In and Sb, at minor and trace levels often in the range of 1000 to 5000 ppm to optimize solder characteristics. XRF spectrometry is easily capable of measuring Pb to the RoHS directive and these other solder alloying elements for quality control and failure analysis.

SMX-BEN Instrument

The SMX-BEN XRF analyzer is a benchtop instrument equipped with a 50 kV, 50W X-ray tube; six programmable collimator sizes and five programmable primary beam filters. Analysis is done in an air atmosphere.

SMX-BEN Measurements

Three standards were measured with composition and thickness described in Table 1. Each standard sample was about 5 mm in diameter, mounted to a 2-pinhole metal frame.

Standard #	Sn (wt%)	Ag (wt%)	Cu (wt%)	Pb (PPM)*	Thickness (µm) **
1	96.17	3.1	0.61	1220	"infinite"
2	97.1	2.4	0.5	NR	10.97
3	97.2	2.4	0.4	NR	18.99

* NR indicates Not Reported

** Infinite indicates the samples were bulk samples with respect to XRF measurement depth

Table 1: composition and thickness of PB-free solder samples measured.

Samples measurements were made under the following conditions:

- 47 kV, 0.960 mA
- 30 seconds measuring time
- 2 mm collimator

Quantification was done using physical computer modeling known as "Fundamental Parameters"(FP). The FP quantitative method, which is commonly known as the "No Standards" method, can be calibrated with a variety of "standards" including a single, pure element, e.g. Cu, up to matching type standards for best accuracy. The method's name "No Standards" derives from the fact that type standards are not a requirement to get quantitative information albeit with lower accuracy. In the measurements in this application note, the accuracy of measurements will be compared between a calibration using matching pure, bulk elements of Sn, Cu, Ag and Pb, which are inexpensive and readily available, and a calibration using a type standard, i.e. standard #2.

Results

Samples #1 and #3 were measured 30 times to obtain measurement statistics. In Table 2, results are shown where the FP calibration was done with pure, bulk elements, i.e. Sn, Ag, Cu and Pb. This is commonly done when type standards are not readily available and the limits on accuracy can be loosened.

Standard#		Sn (wt%)	Ag (wt%)	Cu (wt%)	Pb (PPM)	Thickness (µm)
1	Mean	96.52	3.04	0.35	899.62	
	Max	96.59	3.12	0.38	1077.37	
	Min	96.44	2.97	0.33	767.11	
	Std. Dev.	0.033	0.031	0.011	57.129	
	RSD %	0.03%	1.02%	3.20%	6.35%	
3	Mean	97.06	2.91	0.03		14.67
	Max	97.14	3.03	0.03		14.86
	Min	96.94	2.83	0.03		14.46
	Std. Dev.	0.042	0.042	0.001		0.099
	RSD %	0.04%	1.44%	3.24%		0.67%

Table 2: Statistics on 30 repeat measurements using pure, infinite elements for calibration of the FP quantification method.

(Continued from Page 4)

In these types of calculations, the composition is typically normalized to 100 wt%. Errors tend to affect the accuracy on trace elements, i.e. elements with less than 1 wt%, more heavily due to the low concentrations of the traces. Interestingly, this quantification on sample #1 would indicate that the sample would be flagged for further testing as typical RoHS XRF testing protocols flag samples with XRF results exceeding 700 PPM for Pb for further testing. To improve the repeatability, the measuring time can be increased. A factor of 4 increase in measurement time, i.e. measuring for only two minutes, would improve precision by a factor of two.

Table 3 shows the results for these same 30 measurements using type standards for calibration of the FP method. Type standards can either be purchased from commercial vendors for typical quality control problems and regulatory issues or can be made in-house by validating standards through destructive testing.

Standard#		Sn (wt%)	Ag (wt%)	Cu (wt%)	Pb (PPM)	Thickness (µm)
1	Mean	96.11	3.16	0.61	1278.82	
	Max	96.18	3.22	0.64	1445.06	
	Min	96.05	3.11	0.58	1125.82	
	Std. Dev.	0.036	0.030	0.016	83.037	
	RSD %	0.04%	0.94%	2.66%	6.49%	
3	Mean	97.20	2.40	0.40		18.94
	Max	97.27	2.47	0.42		19.34
	Min	97.11	2.33	0.37		18.67
	Std. Dev.	0.038	0.035	0.012		0.123
	RSD %	0.04%	1.44%	2.96%		0.65%

Table 3: Statistics on 30 repeat measurements using type standards for calibration of the FP quantification method.

Accuracy on the trace elements is now much improved and the thickness measurement accuracy is also greatly enhanced by using a type standard in the range of 10 µm thick. Use of a single type thickness standard will calibrate the FP quantification with high accuracy for a limited range of thickness. Generally, thicker layers are harder to accurately calculate using FP methods because errors in the physical modeling tend to accrue over the thicker layer. A thick, single type standard can correct these errors, but then use of this single, thick type standard calibration model will adversely affect the results for thinner layer measurements. In cases where thickness measurements are required over a "thin" to "thick" range, where thin and thick are defined by the absorptive properties of the materials to be measured and the energy of the available XRF signal peaks, it is best to have a few type thickness standards available which cover the range of thickness to be measured. The FP quantification routine used by the XLNCE SMX family of XRF instruments has several linear and non-linear correction functions available to calculate the internal calibration coefficients as a function of coating thickness. So, if a thickness range of coating standards is available, the FP quantification can be calibrated such that calibration coefficients are also a function of the coating thickness being measured. This provides superior accuracy in thickness/composition measurements over a much broader range of thickness and composition from a single calibration model.

Recommended EDAX Solution

The XLNCE SMX-BEN XRF Analyzer is capable of non-destructive, non-contact measurements to determine layer thickness and composition as well as bulk sample compositional analysis. The SMX-BEN can be used to measure Pb traces in Pb-free solder for RoHS compliance as well Pb content in Pb containing solders for high reliability uses, such as military and aviation. Furthermore, this unit is well-suited for measuring the composition of other minor and trace alloying elements commonly used in Pb-free solders, such as Ni, Cu, Bi, Ag, In and Sb. The SMX-BEN unit can be used for quality control in the laboratory as well as process control measurements with software having operator, supervisor and maintenance access levels to ensure that the right job is done at the right time.

2015 Worldwide Events

April 14-17		
Analitika 2015	Moscow, Russia
April 22-24		
SEMICON Southeast Asia 2015	Penang, Malaysia
May 1		
New England Society for Microscopy (NESM)	Woods Hole, MA
May 3-7		
European Microbeam Analysis Society (EMAS) 2015	Portoroz, Slovenia
May 5-8		
Control	Stuttgart, Germany
May 11-14		
AeroMat 2015	Long Beach, CA
May 13-15		
Japanese Society of Microscopy	Kyoto, Japan
May 20-22		
Southeastern Microscopy Society (SEMS)	Decatur, GA

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

May 18-20	Wiesbaden#
June 2-4	Tilburg*
June 16-18	Wiesbaden#
September 21-23	Tilburg*
November 16-18	Wiesbaden#

Genesis

April 28-30	Tilburg*

EBSD

June 8-10	Tilburg*
September 14-16	Tilburg*
November 18-20	Wiesbaden#

TEAM™ Pegasus (EDS & EBSD)

November 16-20	Wiesbaden#

TEAM™ WDS

May 20-22	Wiesbaden#
September 23-25	Tilburg*
November 17-19	Tilburg*

TEAM™ Neptune (EDS & WDS)

May 18-22	Wiesbaden#
September 21-25	Tilburg*

XRF

October 20-22	Tilburg*

JAPAN

EDS Microanalysis

TEAM™ EDS

June 11-12	Tokyo
July 2-3	Osaka

Genesis

April 16-17	Osaka
October 8-9	Tokyo
November 12-13	Osaka

CHINA

EDS Microanalysis

TEAM™ EDS

September 8-10	Shanghai (ACES)

Genesis

June 9-11	Shanghai (ACES)

EBSD OIM™ Academy

April 14-16	Shanghai (ACES)
October 20-22	Shanghai (ACES)

Particle Analysis

December 8-10	Shanghai (ACES)

NORTH AMERICA

EDS Microanalysis

TEAM™ EDS

May 18-22	Mahwah, NJ
July 21-23	Draper, UT
September 22-24	Mahwah, NJ

EBSD OIM™ Academy

June 16-18	Mahwah, NJ
October 27-29	Draper, UT

Micro-XRF

October 6-8	Mahwah, NJ

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(left to right): Shi Yuanna and Lin Nan.

Lin Nan

Lin joined EDAX as a sales engineer in December 2014. He is responsible for sales in Southeast Asia and works with customers and key accounts, especially electron microscope manufacturers in the region.

Prior to EDAX, Lin worked for Gatan as a sales engineer. He covered sales and applications for all of the company's Scanning Electron and Transmission Electron Microscope products in Southeast Asia. In addition, Lin also worked with existing customers in the region.

In 2009, he graduated from the National University of Singapore with a degree in Physics. He earned a master's degree in Management of Technology in 2014.

Lin and his wife, Shi Yuanna live in Singapore. They married in 2011 after seven years of dating. They are looking forward to starting a family together.

Jens Rafaelsen.

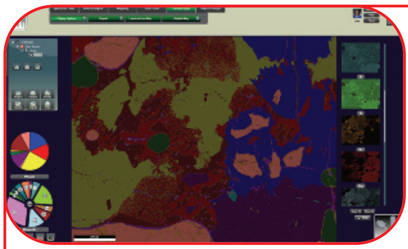
Jens Rafaelsen

Jens joined EDAX in August 2014 as an Applications Engineer specializing in Energy Dispersive Spectroscopy (EDS). Working out of the Mahwah office, he is responsible for conducting demos and running samples for potential and existing customers, teaching training courses, and working with customers to make sure they get the highest quality data and optimized workflow for their tasks. Jens is also involved with testing and qualifying new products and software and developing new methods and techniques using EDS.

Prior to EDAX, he was a technical manager of the clean room facilities at Aalborg University in Denmark. Jens worked with both Scanning Electron Microscope (SEM) and EDS systems for both research and consulting for external partners.

Jens received his Ph.D. in Physics from Aalborg University. His dissertation was entitled "Surface Studies of Germanium Interfaces and Nanostructures". Jens' research focused primarily on surfaces and interfaces using optical techniques and photoemission spectroscopy.

Jens and his wife, Dhevi Natarajan, got married in 2011. They enjoy cooking, traveling and visiting family in Denmark and India.



Back by Popular Demand!
TEAM™ EDS Training Course
May 18-22, 2015 - Mahwah, NJ

During this course, we will cover more in-depth detail of advanced theories of X-ray generation with more practical and software lab sessions, including time for individual sample analysis.

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http://www.edax.com/Support/Training/Americas-EDS-Course-Information.aspx.

Smart Insight

Indian Institute of Technology, Bombay
National Facility of Texture and OIM
Mumbai, India

The National Facility of Texture and OIM is part of the Department of Metallurgical Engineering and Materials at the Indian Institute of Technology, Bombay in Mumbai, India. It is headed by Professor Indradev Samajdar and employs a large number of students and project assistants. The laboratory works with users from academic institutes, national laboratories and industries, without saying no to anyone.

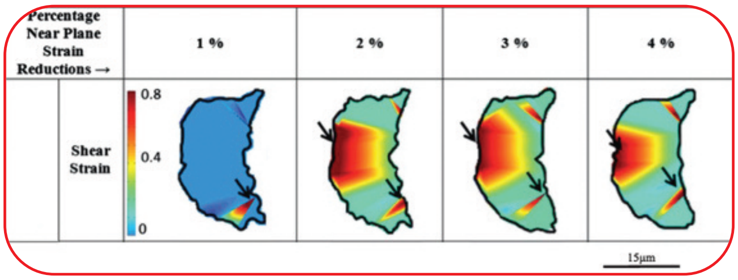
"We believe our role is to help you push the limits of science by giving you easy and fast access to the data necessary to help you uncover new materials insights," said Professor Samajdar.

The National Facility utilizes Electron Backscatter Diffraction (EBSD) for microtexture measurements, which are used for characterizing different features in the microstructure of materials, phase identification, grain boundary analysis and finding out information on local residual stresses and stored energies. Measurements are made on all kinds of materials, including metallic samples, thin films, ceramics and geological samples.

For EBSD analysis, the scientists at the National Facility of Texture and OIM use two Hikari XP cameras and one DigiView V camera on FEI Scanning Electron Microscopes (SEM), which run 24 hours per day, seven days a week. The laboratory also utilizes the TEAM™ Analysis and OIM™ Analysis software. In addition to EBSD, the National Facility of Texture and OIM performs Energy Dispersive Spectroscopy (EDS) analysis using EDAX SiLi, Apollo and Octane silicon drift detectors.

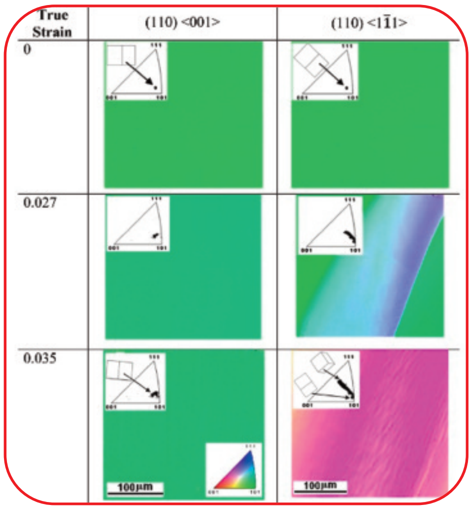
"At this moment, EDAX's EBSD analysis is superior to its competitors. That has been the clinching point," continued Samajdar. "Keeping your edge with the analysis software is crucial to your survival as an EBSD market leader."

Some results from the laboratory's research:



Measurements of shear strain (by an EBSD based digital image correlation) and misorientation during plane strain compression.

Reference: N. Keskar, S. Mikherjee, K. V. Mani Krishna, D. Srivastava, G. K. Dey, P. Pant, R. D. Doherty and I. Samajdar (2014): Quantifying The Redundant Mesoscopic Shear Strains in Plane Strain Compressed Poly-Crystalline Zirconium, **Acta Mater.**, 69, 265-274.



Direct EBSD observations on formation of strain localizations in a transformer steel.

Reference: S. K. Shekhawat, R. Chakrabarty, V. Basavaraj, V. D. Hiwarkar, K. V. Mani, P. J. Guruprasad, A. A. Benzerga, K. G. Suresh and I. Samajdar (2015): Orientation Dependent Plastic Deformation in Transformer Steel: Experiments and Dislocation Dynamics Simulations, **Acta Mater.**, 84, 256-264.

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