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EDAX NEWS

Investigating Localized Deformation Behavior Through Combined Nanoindentation and Orientation Imaging Microscopy

To improve the macroscopic properties of materials in today's world, it is imperative to understand the behavior of a material at the microscopic level. For example, aluminum and magnesium alloys are considered to be ideal components for lightweight materials applications due to their high strength-to-weight ratios. However, these alloys often lack the formability required to be manufactured easily and affordably into useful products. The strength and formability of a material are defined by how it responds to an external force, and when the force is large enough, how it deforms plastically. On a microscopic scale this plastic deformation can be accommodated through both the generation and movement of dislocations through the material or through crystal twinning events. Both of these deformation mechanisms can be detected by analyzing the microstructure using Electron Backscatter Diffraction (EBSD) and Orientation Imaging Microscopy (OIM™).

The crystallographic orientation, which is measured by EBSD, plays a key role for both of these deformation modes. Dislocation motion occurs along specific crystallographic planes and directions called slip systems. Finding ways to activate more slip systems can improve the ductility of a material. Grain boundaries can inhibit dislocation motion. By controlling grain size, engineers can control the yield strength of a material, and even predict properties. Twinning occurs at specific crystallographic orientations as well. EBSD is a key characterization technique for investigating the effects of orientation on deformation behavior.

Nanoindentation is an indentation hardness testing technique where the size of the indenter and the volume of material sampled are small enough that local variations in grain orientation and microstructure can be investigated. This approach has recently been used to determine the crystallographic orientation dependence of hardness and elastic moduli values by coupling nanoindentation with EBSD data (Fizanne-Michel 2014).

(Continued from Page 1)

For this example, a Hysitron PI 87 SEM PicoIndenter® was used to place a series of indents within the microstructure of an Inconel 600 nickel-based superalloy prepared for EBSD analysis. This system is specifically designed for the SEM for in-situ mechanical properties characterization with five degrees of freedom in sample positioning that allows for pre- and post-test EBSD measurements to easily correlate applied stresses with changes in crystallographic orientations.

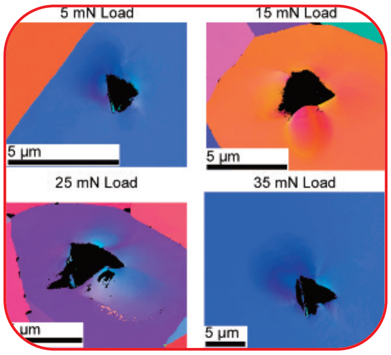


Figure 1. Orientation maps from a set of indents placed with loads varying from 5 mN to 35 mN.

Figure 1 shows EBSD orientation maps, collected with the EDAX TEAM™ EBSD Analysis System, from a set of indents placed with loads varying from 5 mN to 35 mN. Each of these indents was placed within the interior of a specific grain. Note that within this series the scale bar size changes with a constant 5µm reference length. As the load increases, the size of the indent and the surrounding plastic deformation also increases. In these maps, the small changes in color correspond to small changes of local misorientation introduced during the plastic deformation that occurs during the indentation. The effects of grain orientation can also be observed. In three of the indents, a near-(111) oriented (blue) grain was indented, while a near-(100) oriented (red) grain was selected for the 15 mN indent. For the (111) oriented grains, the deformation appears to originate near the corners of the indents. Within the (100) oriented grain, the deformation is located more along the side of the indent.

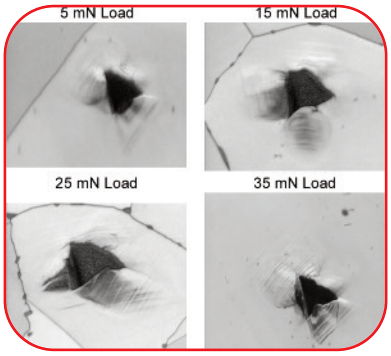


Figure 2. EBSD IQ maps with visible deformation slip bands showing the spatial positioning of the active slip systems.

This spatial relationship between the indenter geometry and crystal orientations can also be visualized using the EBSD image quality (IQ) maps shown in Figure 2. In these images, deformation slip bands are visible showing the spatial positioning of the active slip systems. The specific crystallographic information can be identified by manually drawing along the trace of the slip bands. For the 25 mN load, multiple slip systems are active, explaining why there is a larger region of deformation relative to the 35 mN load indent. It is important to note that the orientations shown in Figure 1 are relative only to the surface normal direction, but that EBSD measures the full three dimensional crystallographic orientation. While three of the grains have near-(111) surface normal orientations, the in-plane orientation information is not shown in this particular map, and these changes in orientations and corresponding slip systems help describe the difference in deformation behavior.

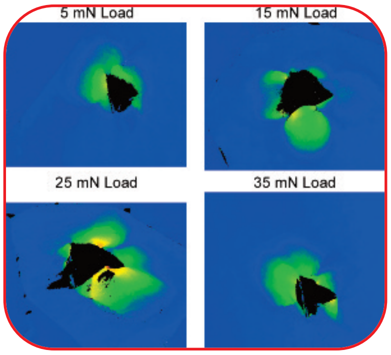


Figure 3. GROD Angle map for the set of indents.

A number of measurements have been developed to measure and visualize the plastic strain fields that develop in materials that are applicable to this type of analysis (Wright 2011). One of these metrics, the Grain Reference Orientation Deviation (GROD) – Angle map, is shown in Figure 3 for the set of indents. In these maps, increases in plastic strain are visualized as increases in the thermal intensity of the map coloring scheme. The orientation precision performance available in the TEAM™ software allows for clear resolution of the deformation fields within the microstructure.

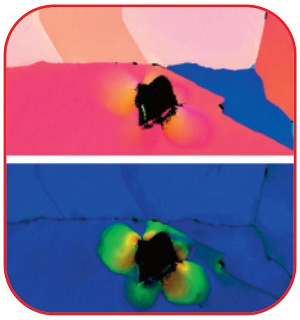


Figure 4. The misorientation relationship between grains is identified before nanoindentation and the corresponding deformation distribution analyzed.

Grain boundaries in a polycrystalline material will inhibit the dislocation motion that occurs during plastic deformation. This slip transfer across grain boundaries will depend on the orientation relationship between the two adjacent grains, and this relationship will determine if regions of damage will nucleate or be suppressed (Bieler 2014). By combining EBSD and nanoindentation, the misorientation relationship between grains can be identified prior to selecting areas for nanoindentation, and the corresponding deformation distribution analyzed. An example of this is shown in Figure 4, where the plastic strain field is attenuated by the local grain boundary.

Combining EBSD and nanoindentation provides researchers with a powerful method of measuring and correlating the mechanical response of the material with the crystallographic orientation. This understanding helps to design materials with improved properties and lifetime performance for a wide range of applications.

References

Fizanne-Michel, C., et. al., Materials Science and Engineering A 613 (2014) 159-162

Wright, S.I. et. al., Microscopy and Microanalysis 17 (2011) 316-329

Bieler, T. R., et. al., Current Opinion in Solid State and Materials Science (2014) In Press

Advanced OIM™

For many people, Electron Backscatter Diffraction (EBSD) analysis has become synonymous with nice looking (color) images, such as Image Quality, IPF, grain size, and many other types of maps, which contain a wealth of information. However, these maps do not do justice to all the data that can be extracted from an EBSD dataset. There is much more that can be done with EBSD data in the EDAX OIM™ Analysis Software. For example, all representations of a single measurement point are linked together and may be highlighted in all displayed maps, plots, and charts interactively for advanced correlative analysis. It is possible to select sub-sections of your data based on properties like orientation, grain properties, or scalar values to separate specific components of your dataset. When a part of the microstructure is selected, that data may be exported for further analysis.

The "common" options are directly accessible through the new quick and right-click options. One level deeper there are many more specialized functions available.

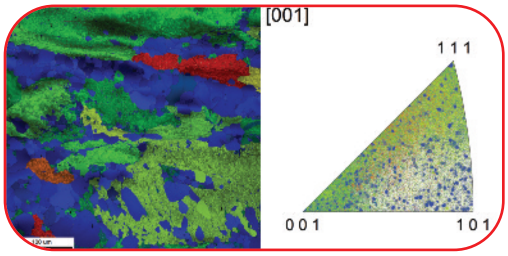


Figure 1. Grain orientation spread map of partially recrystallised steel with corresponding colour-highlighted IPF plot.

The partition properties that allow users to create subsets of an entire dataset for detailed analysis are very powerful. For example, a Confidence Index filter (CI>0.1) may be applied to remove suspect indexed points. In addition to single parameter filters, the mathematical and Boolean operators on the formula tab allow the combination of multiple point and grain parameters to precisely define a fraction of a dataset for separate analysis.

When filter-based partitioning is not possible, the highlighting functionality can be used to create subsets. After creating a highlight, right-click in the map and select "send points to" to generate the subset. Another highlighting function is "apply colors as highlight". With this function users can color-code discrete plots (e.g. IPF, PF, Euler plots) with the coloring used for the "source" map. In Figure 1, the grain orientation spread value is highlighted in an IPF plot. The blue points indicate the individual recrystallized grains and the green background represents the orientation variation due to lattice bending of the deformed grains.

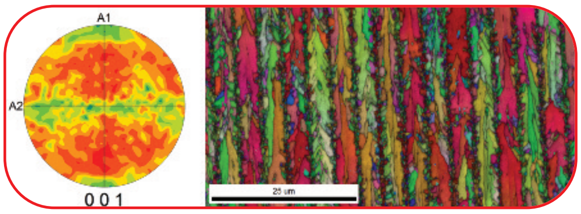
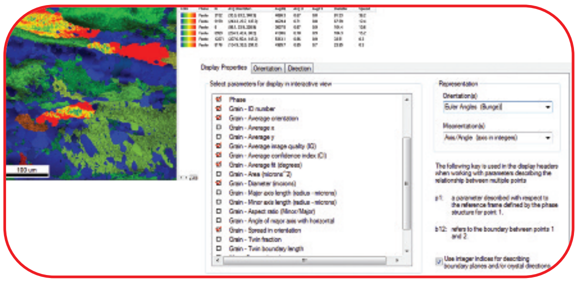


Figure 2. EBSD map of laser crystallised SiGe thin film with Grain Aspect Ratio scalar texture plot (Sample courtesy from Balaji Rangarajan).

The scalar texture function can be used to quantify the occurrence of a scalar value as a function of orientation. Figure 2 shows the grain aspect ratio as a function of orientation. The green zones at the top and bottom of the pole figure show that maximum grain elongation occurs along the [001] axis within ~30 degrees to the lines in the map.



Select parameters for display in interactive view

☑ Phase
☑ Grain - ID number
☑ Grain - Average orientation
☐ Grain - Average x
☐ Grain - Average y
☑ Grain - Average image quality (IQ)
☑ Grain - Average confidence index (CI)
☑ Grain - Average fit (degrees)
☐ Grain - Area (microns^2)
☑ Grain - Diameter (microns)
☐ Grain - Major axis length (radius - microns)
☐ Grain - Minor axis length (radius - microns)
☐ Grain - Aspect ratio (Minor/Major)
☐ Grain - Angle of major axis with horizontal
☑ Grain - Spread in orientation
☐ Grain - Twin fraction
☐ Grain - Twin boundary length

Representation

Orientation(s): Euler Angles (Bunge)

Misorientation(s): Axis/Angle (axis in integers)

The following key is used in the display headers when working with parameters describing the relationship between multiple points

p1: a parameter described with respect to the reference frame defined by the phase structure for point 1.

b12: refers to the boundary between points 1 and 2.

☑ Use integer indices for describing boundary planes and/or crystal directions

Figure 3. EBSD map with interactive grain highlighting results pane and contextual options window.

Quantitative information can also be obtained using the highlighting function, which not only displays the selected data in any open map, chart and plot, but also allows users to record orientation and grain information for the point or grain that is selected. Right-clicking in the interactive tab offers a selection of properties that may be recorded and exported (Figure 3).

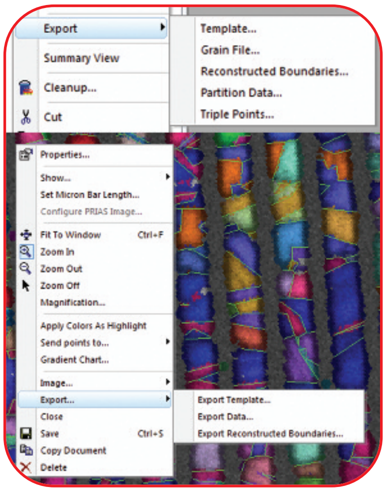


Figure 4. Export data options from map and partition contextual menus.

When highlighting all grains to obtain specific grain properties is impractical, a number of grain property export options are available from the partition contextual menu. However, when the visualization and quantification options in OIM™ Analysis are insufficient for a user's needs, the software offers direct access to virtually all data for your own processing.

This information may be accessed by the various export functions that can be found in the dataset, partition or map contextual menus (Figure 4). In general, when you need something special in OIM™ Analysis, try a right-click.

APPLICATION NOTE

Analyzing Daguerreotype Chemistry Using the Orbis Micro-XRF Elemental Analyzer

In 1839, the French Academy of Sciences initiated what would become the birth of photography by introducing the daguerreotype. Named after its primary inventor, Louis Jacque Mande Daguerre, it was the first mainstream method for developing photographic images. The process utilizes a polished silver plate, which is exposed to a halogen-based fume to create a light-sensitive surface for exposure. The "negative" is developed by exposing the plate to mercury fumes. To reduce the harsh black and white tone, a solution of gold chloride is optionally washed over the image to produce a warmer amber hue (gilding). In addition, various metal oxide solutions such as copper oxide or iron oxide can sometimes be used to introduce color.

Analyzing daguerreotypes for their elemental composition helps shed light on the history of early photochemistry, and also can show which particular chemicals were used for each step of the process. Micro X-ray Fluorescence (micro-XRF) is a non-destructive technique which utilizes an X-ray source to generate characteristic signals from the sample. An EDAX Orbis PC with a 50 mm^2 silicon drift detector, and a poly-capillary optic (~30 µm FWHM at Mo-Ka) oriented normal to the sample was used in this application. Integrated video cameras and an XYZ stage allow sample navigation 100 mm in each direction. One analytical hurdle, as with any cultural or archeological sample, is to avoid destructive sample preparation and analysis techniques. Micro-XRF analysis is non-destructive and is, therefore, ideal for these types of samples. Micro-XRF requires very little sample preparation, such as polishing and mounting. Also, as there is no sample charging associated with X-ray excitation, it is not necessary to coat the sample. Because the spot size of the Orbis is much larger than the spot size of Energy Dispersive Spectroscopy (EDS) micro-XRF is generally more efficient for larger samples and larger areas of interest. In addition, detection for micro-XRF begins at sodium, but is particularly sensitive for heavier elements.

A portion of a daguerreotype supplied from the Detroit Institute of Arts (DIA) is shown in Figure 1 using the Orbis PC internal low magnification 10X camera.

Figure 1. Approximately a 4 x 3 mm video image capture of the daguerreotype. Note that the reflection left of center is from the X-ray optic, which is situated directly above the sample.

While the general chemistry of the plate was known, the Orbis was used to identify all unknown elements and their distribution, and in particular, the elemental composition of the pink area shown on the left cheek of the portrait, as it appears to have been an intentional addition as opposed to discoloration. Again, accomplishing this without destroying the sample is key.

To collect a spectral distribution map, the stage moves in a two-dimensional raster while the X-ray detector collects a series of points and scales the intensities for each element. Because the data is stored for each individual data point on the map, many post-processing options are possible, such as re-building maps "from memory" for elements not initially identified. The running conditions for this sample were 40 kV, 600 uA on a rhodium-anode X-ray tube. The collection area (shown in Figure 1) was approximately 4 x 3 mm. The scan collected 256 x 200 points in the X and Y directions with the 30 µm poly-capillary, for 0.4 seconds per point. Figure 2 shows maps for Au, with M-series on the left and L-series on the right. The ability to work at higher energies with micro-XRF allows comparison of two transition series of the same element.

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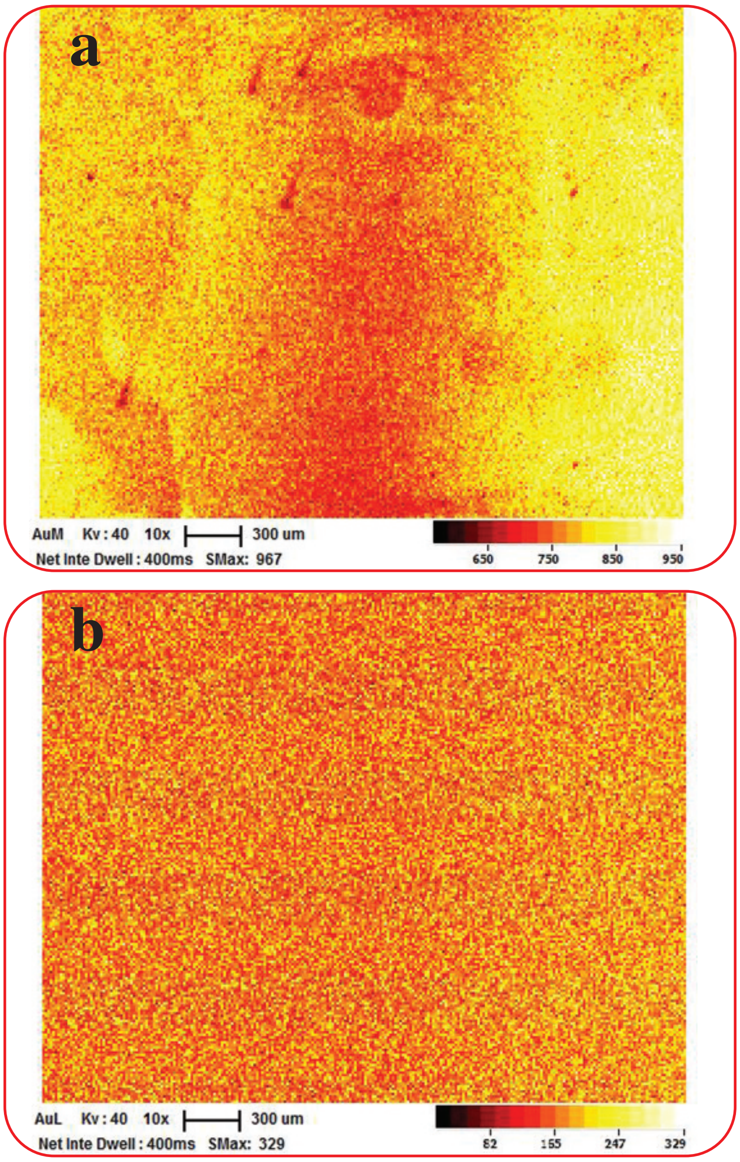

Figure 2. The (a) Au(M) and (b) Au(L) maps for comparison, with the M-series being more indicative of surface distribution.

Because the M-series lines are less energetic, the photons have less escape potential (on the order of tenths of a micron), giving more surface-sensitive information. The L-series, having greater escape potential (on the order of a few microns), would be more indicative of sub-surface composition. Figure 2 shows a discernable Au pattern consistent with the photograph's features on the M-series map, while Au(L) does not, suggesting that the Au is mostly on the surface. This is consistent with the aforementioned gilding process.

In the Fe(K) map shown in Figure 3, a prominent streak of Fe is shown, which correlates with the location of the pink hue in the video image. Iron is clearly the main constituent, which is consistent with early methods of adding color by using materials like iron or copper oxides. There are also isolated Fe(K) inclusions, which are likely contaminants.

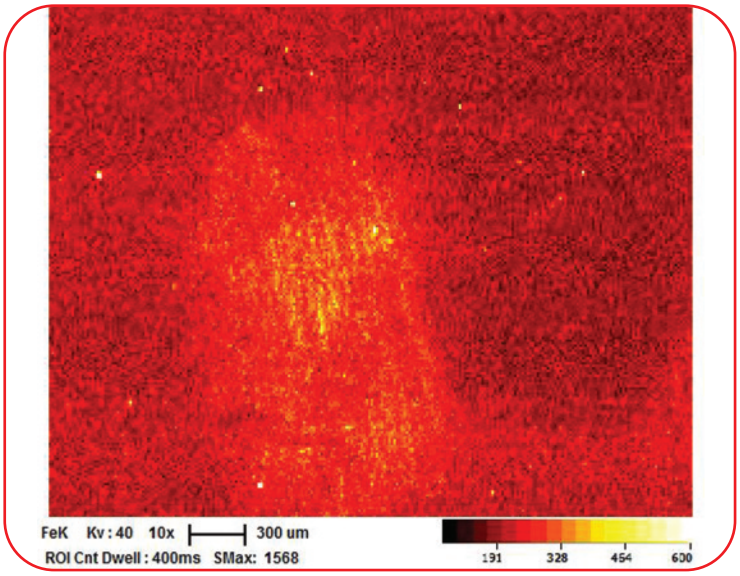

Figure 3. Fe(K) map shows a streak consistent with the pink hue on the cheek.

Various other elements associated with daguerreotypes were detected, and similar to Fe and Au, they each confirm which of the potential chemicals were used for each step of the process. Non-destructive analysis and minimal sample preparation requirements are the main benefits of micro-XRF analysis with cultural items, such as daguerreotypes. In addition, the sampling area is more efficiently covered by a larger X-ray beam instead of an electron beam. Because sensitivity generally improves with higher atomic numbers for micro-XRF, heavier metals like Fe and Au benefit and reveal much about the processes behind creating these early photographs.

Worldwide Events

Date	Event	Location
October 6-10	Midwestern Assoc. of Forensic Scientists (MAFS) & American Society of Trace Evidence Examiners (ASTEE)	St. Paul, MN
October 13-16	The Materials Science & Technology Expo (MS&T)	Pittsburgh, PA
October 16-17	Appalachian Regional Microscopy Society (AReMS)	Knoxville, TN
November 30-December 5	Materials Research Society (MRS) Fall	Boston, MA
December 1-5	Microscopy Society of S. Africa (MSSA)	Pretoria, South Africa
December 4-5	GN-Meba 2014	Paris, France

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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	
November 6-7	Tilburg*
TEAM™ EDS	
September 30-October 2	Tilburg*
December 1-3	Wiesbaden#
EBSD	
November 5-6	Tilburg*
December 3-5	Wiesbaden#
TEAM™ Pegasus (EDS & EBSD)	
December 1-5	Wiesbaden#
TEAM™ WDS	
October 14-16	Tilburg*
Orbis: Course & Workshop	
October 28-30	Wiesbaden#

JAPAN

EDS Microanalysis	
Genesis	
October 9-10	Tokyo
November 13-14	Osaka

CHINA

EDS Microanalysis	
TEAM™ EDS	
November 4	Guangzhou
Decemer 1-4	Shanghai (ACES)
EBSD OIM™ Academy	
October 20-23	Shanghai (ACES)

NORTH AMERICA

EDS Microanalysis	
TEAM™ EDS	
September 23-25	Mahwah, NJ
EBSD OIM™ Academy	
September 30-October 2	Mahwah, NJ
Micro-XRF	
October 21-23	Mahwah, NJ

*Presented in English
#Presented in German

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AMETEK®
MATERIALS ANALYSIS DIVISION

(left to right): Lindsay, Creedence, Jude, Travis and Roxanne Rampton.

Travis Rampton

In July 2013, Travis joined EDAX as an Applications Engineer with a specialty in Electron Backscatter Diffraction (EBSD). He started his career at the company's Draper, UT location and has since relocated to the Mahwah, NJ office. Travis is responsible for supporting customer demos, teaching training courses and testing Energy Dispersive Spectroscopy (EDS) and EBSD products. He also represents EDAX at conferences in North and South America.

Prior to EDAX, Travis was working on his Ph.D. in Mechanical Engineering at Brigham Young University in Provo, UT. He earned a Bachelor of Science degree in Mechanical Engineering from Brigham Young in 2010.

Travis lives in West Milford, NJ with his wife, Lindsay. They have three children, Jude (4), Creedence "CCR" (2) and Roxanne (1), who are all named for famous Rock N' Roll songs and bands. A Los Angeles native, Travis enjoys playing with his kids, watching movies with his wife and obsessing over the Los Angeles Lakers basketball team.

(left to right): Zhang Shuting and Haodong Ding.

Haodong Ding

In October 2010, Haodong joined EDAX as a sales engineer in China. Working out of Beijing, his sales territory is North of China, which includes Beijing, Tianjin and the Inner Mongolia, Hebei, Shandong, Liaoning, Jilin and Heilongjiang provinces. Haodong is responsible for selling EDAX products, developing local markets and maintaining good relationships with all Electron Microscope Manufacturers (EMMs).

Prior to EDAX, Haodong worked at Techcomp Ltd. as a sales engineer in North China from 2008-10. Techcomp is an exclusive agent of Hitachi in China. He was responsible for selling Hitachi's Scanning Electron Microscopes (SEMs) and Transmission Electron Microscopes (TEMs).

In 2007, Haodong earned a master's degree in Physical and Analysis Chemistry from Xiamen University. He received a bachelor's degree in chemistry from Hebei Normal University in 2004.

On September 1, 2014, Haodong's family welcomed a new member, with the birth of a baby boy. Haodong is happily living in Beijing with his wife, Zhang Shuting and newborn son, Ding Yuqun. In his spare time , he enjoys running, playing billiards and spending time at the theater with his wife.

National Renewable Energy Laboratory (NREL) National Center for Photovoltaics (NCPV)

As a part of the United States Department of Energy, NREL focuses on all areas of renewable energy. Some of its major activities are the research and development of devices and processes that produce clean energy, and on renewable energy applied to transportation.

As part of NREL, the NCPV is located in Golden, CO and specializes in research and development of different photovoltaic technologies, such as silicon, III-V materials, CdTe, $CuInGaSe_2$ and organic solar cells. In addition to research, it is also involved in several stages of solar cell fabrication. The laboratory is capable of growing materials using deposition techniques, such as molecular beam epitaxy, close-spaced sublimation and sputtering. The NCPV uses analytical techniques to measure the electro-optical, compositional and structural properties of materials.

To facilitate the fabrication of high-quality materials and high-efficiency solar cells, NCPV scientists analyze the properties of materials at different stages of fabrication. The scientists are looking at grain size and grain structure, minority carrier lifetime, defect levels in the band gap, interface states and various other parameters.

“We believe that the most effective way to fabricate high-efficiency devices is by understanding and optimizing the properties of the materials and interfaces,” said senior scientist Helio Moutinho.

The NCPV purchased a Pegasus Analysis System to perform both EDS and Electron Backscatter Diffraction (EBSD) analysis. The system is mainly used by two scientists with a Ph.D. in Materials Science, one technician with Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) experience and a few Ph.D. students.

“At the time we were looking for a system, EDAX provided the best deal,” stated Moutinho. “The EBSD detector had a high rate of data collection and we liked the software features. Also, the possibility of doing EDS and EBSD (ChI-Scan) to identify different phases was a desired capability.”

The scientists use EBSD to study the crystallographic orientation of thin film materials grown using a wide range of techniques. With EBSD, they can determine the grain size, which is a critical property of films used in photovoltaic devices. EBSD is also utilized to study the epitaxial growth of CdTe and CZTS films, in both plain view and cross section, with much easier sample preparation than would be required with a Transmission Electron Microscope (TEM). The NCPV scientists can also look at cross sections of polycrystalline thin films with EBSD, giving them the ability to study the dynamics of film growth. Figure 1 shows the importance of temperature and the influence of oxygen during the growth of CdTe thin films.

At the NCPV, EDS is used to investigate the composition of films, including film homogeneity, which is a major requirement for the production of high-quality films and devices. Currently, the scientists are using ChI-Scan to look at the formation of different phases in films and to identify different phases where similar crystalline structures exist. The need for the NCPV to perform both crystallographic (EBSD) and elemental (EDS) analysis quickly, easily, and accurately, makes the Pegasus Analysis System the ideal choice for its materials characterization problems.

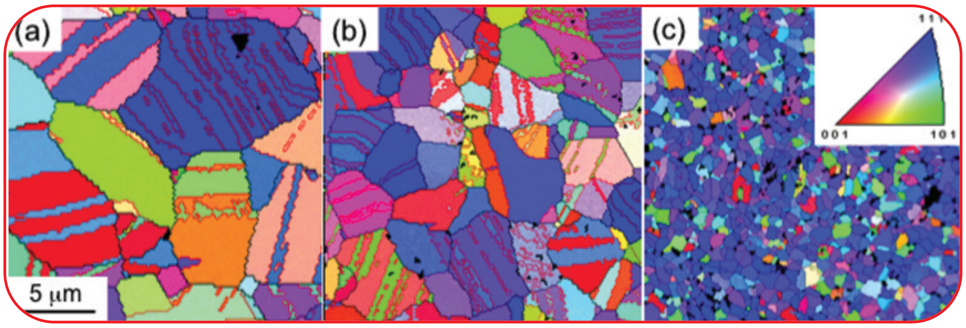

Figure 1. EBSD maps of CdTe films deposited at (a) 550°C, (b) 450°C and (c) 450°C with oxygen. The crystallographic orientation is shown on the inset on the top right. Black lines are grain boundaries and red lines are CSL ∑3 boundaries.

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