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# EDAXinsight

March 2013 Volume 11 Issue 1

# We understand how you see the world.



#### EDS NEWS

## TEAM<sup>™</sup> Pegasus with Octane SDDs Accelerates Development of Lightweight Automotive Materials

Both consumer demands and legislative requirements are compelling automotive manufacturers to develop more fuel-efficient vehicles. A primary method of improving automotive mileage performance is to reduce vehicle weight. The automotive industry is investing heavily in lightweight materials research to meet these demands. Meeting weight and fuel efficiency targets requires the development of structural materials that meet the strength and weight requirements, along with the forming processes needed to manufacture these materials in a costeffective manner and the joining processes to integrate these materials with existing components without compromising performance and safety. TEAM™ Pegasus Analysis Systems provide the materials insight necessary for automotive engineers to meet their goals efficiently and effectively.

The TEAM<sup>™</sup> Pegasus Analysis System is an advanced materials characterization solution used to analyze both elemental composition via Energy Dispersive Spectroscopy (EDS) and crystallographic structure via Electron Backscatter Diffraction (EBSD). Results from TEAM<sup>™</sup> Pegasus enable users to better understand material microstructure and control material properties. The TEAM<sup>™</sup> Pegasus Analysis System consists of the TEAM<sup>™</sup> Pegasus software platform, an Octane Series Silicon Drift Detector (SDD) for EDS data collection and a Hikari XP or DigiView IV camera for EBSD data collection.

The TEAM<sup>™</sup> Pegasus Analysis System provides significant benefits to automotive engineers seeking to integrate lightweight structural materials into automotive systems. The primary benefit is a highquality representation of the internal microstructure of the material. An understanding of microstructure is key to predicting the final mechanical properties of the materials. Different joining processes will also affect microstructure. Analysis of this microstructure is important for optimizing joining parameters in order to provide a consistent quality final result. TEAM<sup>™</sup> Pegasus provides information not easily obtainable using other characterization techniques. Information on crystal orientation, phase content and distribution, grain size and shape, grain boundary character and plastic strain distribution is collected with both high speed and precision.





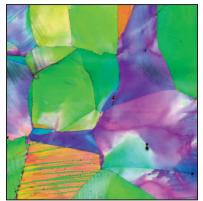
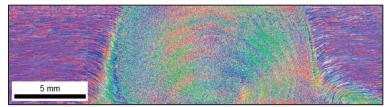


Figure 1: EBSD showing microstructure and twin boundaries present in TWIP steel.

Fast collection allows for a statistically significant number of measurements to be gathered automatically, in contrast with Transmission Electron Microscopy (TEM) measurements, which are manually collected at a slow rate. The Octane SDDs provide superior resolution stability even at high count rates, which enables fast mapping of light alloying elements in lightweight materials to accurately characterize phase distribution.

High strength steels, aluminum and magnesium alloys are three candidates being explored to meet fuel efficiency targets. One example of advanced engineered steel is Twinning Induced Plasticity (TWIP) steel. TWIP steels are designed to absorb more energy in a crash than traditional steels, while maintaining strength and stability. This property is the result of twinning that occurs during the plastic deformation during a crash, thereby increasing the yield strength of the material. As shown in Figure 1, EBSD is used to measure the microstructure and twin boundaries present in the steel before and after deformation. This information is used to improve and optimize the processing conditions to maximize twin formation during deformation.

With a significantly lower density than steel, aluminum offers great potential for weight reduction. Honda has recently announced that the 2013 Accord will feature steel and aluminum components joined using a continuous welding process called friction stir welding. The process enables joining of dissimilar metals with resulting weld strength greater than conventional Metal Inert Gas welding. This new technology reduced body weight by 25% and electricity consumption during welding by 50%. Figure 2 shows an EBSD map of a crosssection of an aluminum Friction Stir weld. EBSD measures the grain size and shape, orientation and plastic deformation from the base metal, the heat affected zone and the center weld nugget. This information provides insight into material flow during the welding process and feedback to optimize welding parameters used to maximize weld quality between different materials.



gure 2: EBSD map of a cross-section of aluminum Friction Stir weld.

#### (Continued from Page 1)

Magnesium also offers an appealing combination of high strength and low density. However, in order for it to be useful, manufacturing challenges must be overcome. In magnesium alloys, the formability is directly related to the slip system orientation distribution. Wrought magnesium alloys generally have a (0001) texture, which creates anisotropic materials properties. Figure 3 shows an orientation map of wrought magnesium exhibiting a strong (0001) texture. Improvements in texture control and formability are needed to increase the use of magnesium alloys in automotive applications. EBSD is used to understand the effects of alloying, deformation rate and temperature on formability in

order to help make magnesium

a viable lightweight material.

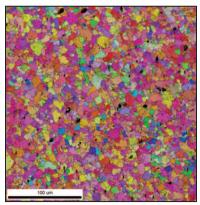


Figure 3: Orientation map of wrought magnesium exhibiting strong (0001) texture.

The TEAM<sup>™</sup> Pegasus Analysis System allows users to collect highquality reliable EDS and EBSD data easily. The TEAM<sup>™</sup> Pegasus software platform is built around revolutionary Smart Features. These features use analytical intelligence to optimize systems settings for specific analytical measurements to provide consistent high-quality data for all users. Novice operators can rely on these Smart Features to achieve reliable data, while expert-level users will find them useful timesavers. These Smart Features include:

Smart Camera – Automatic optimization of the EBSD camera settings based on the desired acquisition mode to maximize data quality while minimizing data collection time.

Smart Indexing - Accurate EBSD crystal orientation determination through a unique triplet indexing algorithm to provide superior indexing success rates.

Smart Phase – Automated phase mapping with no prior user input to remove the requirements for accurate phase determination.

Smart EXpert ID – Analytical intelligence combining traditional EDS peak ID routines with real world analysis techniques to provide accurate quantitative EDS analysis.

Smart Features in TEAM<sup>™</sup> Pegasus Analysis Systems allow users to better focus thier lightweight materials engineering efforts, enabling them to more quickly hone in on key areas to improve efficiency while maintaining crash safety performance.



# Optimizing Spatial Resolution for EDS Analysis

One common theme in the development of advanced materials is the continued reduction in critical feature size. As scientists and engineers push microstructural development to meet performance and cost goals, the ability to identify and characterize smaller and smaller chemical features becomes more important.

Spatial resolution in EDS is dominated by the size of the interaction volume, the region of X-ray excitation in the sample. This interaction volume is determined by the accelerating voltage of the SEM and by the density of the sample itself. Traditional EDS was often done at an accelerating voltage of 15 kV. Figure 1 below is a Monte Carlo simulation illustrating the interaction volume for a 15 kV beam on a pure Aluminum sample. The depth of X-ray generation is well over a micron and the lateral spread is more than 2 microns. From this, it is apparent that EDS spatial resolution is limited to 2 microns under these conditions.

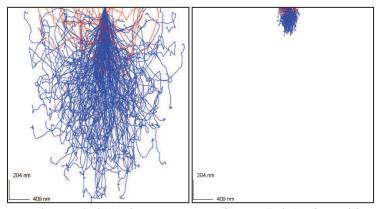


Figure 1: Monte Carlo SimulationFigure 2: Monte Carlo Simulation of theillustrating the interaction volume for ainteraction volume of a 5kV beam with the15 kV beam on a pure Aluminum sample.same Aluminum sample.

By comparison, Figure 2 shows a Monte Carlo Simulation of the interaction volume of a 5 kV beam with the same Al sample. In this case, the area of X-ray generation is reduced to approximately 200 nm x 200 nm, and the EDS spatial resolution would be of similar size.

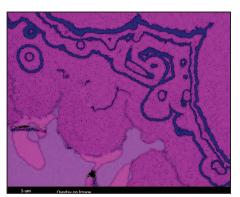
Knowing that the size of the interaction volume increases with accelerating voltage, and in turn, the spatial resolution of the EDS analysis decreases, it becomes obvious that the user should work at as low a kV as possible for his/her particular sample. To determine that value, a user must know what elements are present in the

sample and which X-ray lines are needed. From that point, the Critical Ionization Energy - the beam energy needed to excite the desired X-ray emission - can be determined. In reality, it is necessary to overshoot the Critical Ionization Energy by a margin, usually one and a half to three times the highest energy X-ray line. This value over the Critical Ionization Energy is called the overvoltage.

Figure 3 below shows a SiC sample with a thin Carbon ribbon-like structure layered into the matrix. This C layer can be very difficult to measure, both due to its extreme thinness and to the difficulty in generating and collecting the low energy C X-rays. However, when analyzed under ideal conditions, TEAM<sup>™</sup> EDS with Octane SDDs can clearly resolve sub-50 nm thicknesses.

It should be noted that electron beam current is not a direct factor on EDS resolutiondepending on the SEM being used. While tungsten filament SEMs inherently have a direct relationship between current and probe size, i.e., an increase in current will result in an

increase in probe size,



*Figure 3: SiC sample with a very thin carbide structure present.* 

modern field emission SEMs are capable of supplying high beam currents in very small spot sizes at low kVs. These higher currents lead to higher count rates, better statistics and faster analysis.

EDAX Octane SDDs offer the ideal combination of resolution stability, efficiency and low energy performance to maximize your results at lower kVs. The Octane SDDs provide the latest evolution in SDD technology with low noise electronics that clear the way for analysis using the extreme low energy region while providing a guaranteed 90% resolution stability that provides industry best resolution at low count rates and at high throughput conditions. Additionally, Octane detector design provides three times the X-ray throughput of traditional SDDs in the marketplace for high efficiency operation at low deadtimes.



## Smart Phase Mapping Provides the Next Level of Materials Insight

#### **Comparison with Existing Solutions**

Energy dispersive spectroscopy (EDS) and elemental mapping have traditionally been used to characterize elemental composition and spatial distribution of the elements in the sample microstructure. These techniques provide an accurate method to quantify elemental ratios and distribution; however, they provide no insight into how the elements are combined into phases or into the distribution of phases in the sample.

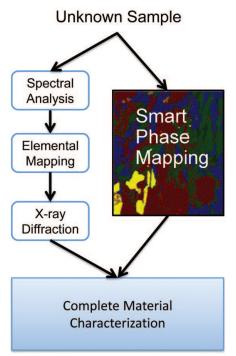


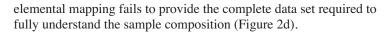
Figure 1. Materials Characterization Flowchart

In contrast, EDAX's Smart Phase Mapping feature in the TEAM<sup>™</sup> EDS Analysis Software provides elemental weight percentages, spatial distribution, and phase information with all three results collected in the same time frame as a typical EDS map of the past.

Element and phase maps are collected automatically and simultaneously, allowing the user to see how an element is distributed individually or how the phases are spatially related. See Figure 1.

#### **Microanalysis Results**

The elemental maps seen in Figure 2a and 2b show obvious differences in the distribution of the major elements, Aluminum (blue) and Oxygen (yellow). A basic image overlay (Figure 2c) shows the general spatial relationship of these elements clearly. However, when it comes time to consider the minor constituent elements present – Magnesium, Calcium and Sulphur - or when trying to understand the small variations in the Al to O ratio,



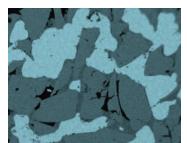


Figure 2a. Aluminum map



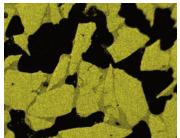


Figure 2b. Oxygen map

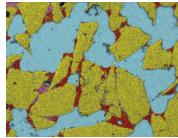


Figure 2c. Aluminum, Oxygen overlay map

Figure 2d. Aluminum, Oxygen, Sulphur, Magnesium, Calcium overlay map

Figure 3a is an enlargement of figure 2d, and, at first glance, appears to accurately explain the compositional nature of the sample. However, to completely characterize the microstructure and to be able to understand a material well enough to propose modification to impact its material properties, further insight is required. The Smart Phase Mapping feature provides the tool to reach this level of understanding.

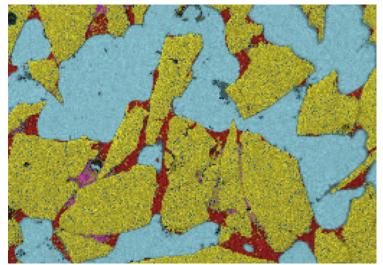


Figure 3a. Image overlay of five elemental maps. When multiple elements are present, each element can be difficult to distinguish visually.



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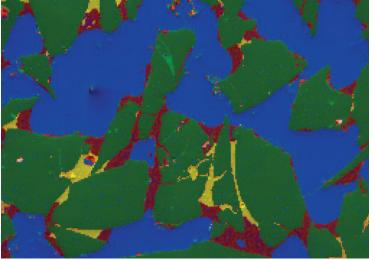


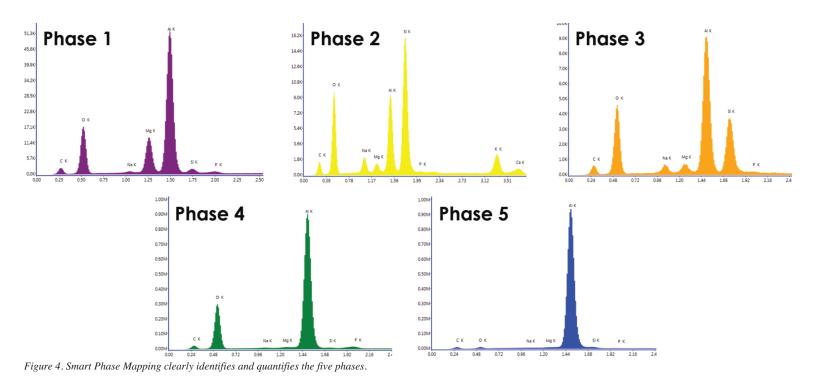
Figure 3b. One minute Smart Phase Map collected at 200 kcps throughput and 131 eV resolution, clearly showing the five different phases in the sample.

Figure 3b is a phase map of the same area used in figure 3a. With elemental mapping, it is impossible to determine the the number of phases present, while Smart Phase Mapping clearly identifies and quantifies 5 phases (Figure 4).

The result is a single Phase Map, collected in the same time as an elemental map, which clearly describes the overall chemical makeup of the material.

#### **Recommended EDAX Solution**

Smart Phase Mapping, a key component in TEAM<sup>™</sup> EDS Enhanced Software, is recommended to help engineers and scientists fully understand the true microstructure of their materials. Paired with Octane Silicon Drift Detectors, this powerful data is collected at blazing speeds and high quality, providing the ultimate in materials insight.



EDAX Smart Insight

## 6 EDAXinsight

#### EVENTS AND TRAINING

# Worldwide Events

April 16-18		June 9-14	
SAE 2013 World Congress & Exhibition	Detroit, MI	Lehigh School	Bethlehem, PA
April 18-20		June 10-14	
Texas Society of Microscopy (TSM)	Dallas, TX	Scandem	Copenhagen, Denmark
May 12-16		June 19-21	
EMAS 2013	Porto, Portugal	Microscopial Society of Canada (MSC)	British Columbia, Canada
May 22-24			
The Southeastern Microscopy Society (SEMS)	Greenville, SC		

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

# 2013 Worldwide Training

To help our present and potential customers obtain the most from their equipment and to increase their expertise in EDS microanalysis, WDS microanalysis, EBSD/OIM<sup>™</sup>, 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				
June 13-14, 2013	Tilburg			
September 12-13, 2013	Tilburg			
November 7-8, 2013	Tilburg			
TEAM™ EDS				
May 28-30, 2013	Tilburg			
June 11-12, 2013	Wiesbaden			
September 24-26, 2013	Tilburg			
November 11-13, 2013	Wiesbaden			
Genesis				
April 9-11 2013	Tilburg			
November 19-21, 2013	Tilburg			
EBSD				
June 10-12, 2013	Tilburg			
September 9-11, 2013	Tilburg			
November 4-6, 2013	Tilburg			
November 13-15, 2013	Wiesbaden			
EDS & EBSD (Pegasus)				
November 11-15, 2013	Wiesbaden			
WDS				
April 16-17, 2013	Wiesbaden			
October 8-9, 2013	Tilburg			
Orbis: Course & Workshop Presented in English				
October 29-30, 2013 Wiesbaden				

JAPAN

EDS Microanalysis		
Genesis		
April 11-12, 2013	Osaka	
October 10-11, 2013	Tokyo	
November 14-15, 2013	Osaka	
TEAM™	EDS	
June 13-14, 2013	Tokyo	
July 11-12, 2013	Osaka	

### CHINA

EDS Microanalysis				
TEAM™ EDS				
March 19-21, 2013	Shanghai			
May 8, 2013	Beijing			
June 4-6, 2013	Shanghai			
September 24-26, 2013	Shanghai			
October 10, 2013	Xi'an			
December 3-5, 2013	Shanghai			
EBSD				
April 16-18, 2013	Shanghai			
October 15-17, 2013	Shanghai			
Particle Analysis				
June 4-6, 2013	Shanghai			

## NORTH AMERICA

EDS Microanalysis			
TEAM™ July 16-18, 2013 September 17-19, 2013	Draper, UT		
EBSD			
August 20-22, 2013 November 12-14, 2013	Draper, UT Mahwah, NJ		
EDS & EBSD (Pegasus)			
May 13-17, 2013	Mahwah, NJ		
Micro-XRF			
April 9-11, 2013 October 1-3, 2013	Mahwah, NJ Mahwah, NJ		

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



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## **EDAXinsight**

#### EMPLOYEE SPOTLIGHT



Jeff Hendrickson

Jeff joined EDAX in November 2011 as Electrical Engineering Manager. He is responsible for the electrical engineering team, including systems, mechanical and software engineering resources.

In 1996, Jeff completed his Bachelor of Science degree in Applied Physics at The College of New Jersey. He also received a Master of Engineering degree from Stevens Institute of Technology in 1999.

Jeff has extensive experience as an electrical engineer and as a project manager. His background is in RF design. Prior to joining EDAX, he designed communication systems for the United States Department of Defense.

Jeff currently lives in Rockaway, NJ with his wife, Kristy, and their three children, Tyler (21), Caden (eight) and Zeven (three). Outside of work, Jeff enjoys a multitude of outdoor activities, including hiking, backpacking, soccer, surfing, snowboarding and wakeboarding.



## John Mendoza

John began working at EDAX in 1987. He is a Senior Service Engineer with experience in all aspects of X-ray microanalysis including: EDS, EBSD, WDS and micro-XRF. John is involved in installing, training and maintaining systems throughout the southwestern United States and assisting customers in Puerto Rico. He enjoys supporting the sales team and pays special attention to establishing lasting relationships with customers.

John was born and raised in Southern California, where he enjoyed being part of the excitement of the 'dot com era'. He began his studies in electronics at DeVry University in Phoenix, AZ and continued at ITT Technical Institute in California.

In 1985, John joined Philips Electronic Instruments to provide service support in the Los Angeles area in the field of X-ray security instruments, such as those found in airports, U.S. customs, prisons and courthouses. It was a hectic and demanding schedule but entailed exotic trips, such as visits to Guam, Alaska and his favorite service experience Saipan, where the locals invited him to cook a pig in the middle of the jungle. While transitioning into EDS, he also did a brief venture into PYE Unicam, Philips' line of spectrophotometers based out of Oxford, England.

John has been married to his wife, Eloise, for 22 years and they have two sons, Nic (18) and Alec (14). He likes restoring architecture, working on art pieces and being involved with the neighborhood historical district. Together, the couple enjoys family, neighbors, nightlife and everything California has to offer.



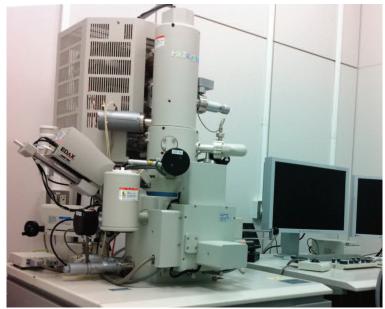
# Center of Advanced Instrumental Analysis Kyushu University, Fukuoka, Japan

Kyushu University is the fourth oldest national university in Japan, with a long history and tradition. The School of Engineering, established in 1911, is a leading educational and research hub in Japan with more than 300 academic staff members and cutting edge research facilities. It has produced over 43,000 graduates working in all areas of society, both in Japan and overseas.

In order to progress in science and technology, analytical equipment for material and structural analysis has become essential. This led to the creation of the Center of Advanced Instrumental Analysis at Kyushu University in 1982. The center provides multiple users shared access to sophisticated analytical equipment, which would not be possible by on a one-on-one basis.

At the University, there is a growing demand for high magnification observation with elemental analysis. The latest scanning electron microscopes are able to observe these images with low acceleration voltage and low beam current. This is very useful in analysis of electron beam-sensitive samples and nano-sized samples. However, this observation condition is challenging for elemental analysis due to the limited number of X-rays generated.

The first EDAX system was installed over 20 years ago and the lab currently uses EDAX equipment exclusively for its SEM. With a growing need by professors at the University to perform elemental analysis, the Center of Advanced Instrumental Analysis introduced several Genesis and Trident systems with a variety of detectors. Dr. Watanabe, an assistant professor at Kyushu, supervises the use of all the systems.



An Ultra-High Resolution Scanning Microscope SU8000 with an EDAX Octane Super SDD in the Center of Advanced Instrumental Analysis at Kyushu University.

The equipment is shared by students, researchers and professors who perform sample analysis and educational experiments. The recent purchase of the Octane Super SDD (previously, Apollo XF) detector is important because it allows the users to maximize X-ray collection under difficult analysis conditions, such as low accelerating voltage or low beam current.

## Also available from AMETEK-

TMC designs and manufactures the world's most advanced precision floor vibration isolation systems for ultra-precision research and manufacturing processes.

SEM-Base<sup>™</sup> uses a patented, piezoelectric vibration cancellation system to dramatically lower the effect of floor vibration and improve image resolution. SEM-Base allows SEMs to be successfully installed on floors that otherwise do not meet spec.

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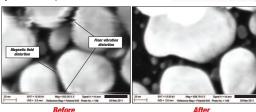


When stray magnetic fields exceed spec, Mag-NetX<sup>™</sup> actively cancels background magnetic fields. Mag-NetX is designed for both point-of-use and OEM applications and is ideal for SEMs, TEMs, and other charged beam tools.

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#### For the highest resolution even when your environment does not meet spec

The before and after photos below are actual images taken on the FIB-SEM (right) installed in a non-ideal environment. The image on the left was taken with the newly installed TMC STACIS<sup>®</sup> iX SEM-Base<sup>™</sup> Floor Platform and Mag-NetX<sup>™</sup> Magnetic Field Cancellation systems powered-off. The image on the right was taken immediately after both active systems were powered-on



Before



Art and Layout Jonathan McMenamin

**Contributing Writers** Toru Ae Mike Coy Matt Nowell

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**METEK**° MATERIALS ANALYSIS DIVISION