EDAX FOCUS

Orbis Vision Software Version 1.6 Release

Inside This Issue

With the release of Orbis Vision Software Version 1.6, EDAX has introduced coating thickness and composition of analysis software onto the Orbis micro-XRF elemental analyzer. With the Orbis coating analysis package, the Orbis micro-XRF spectrometer can make simultaneous measurements of layer thickness and composition on a multi-layered material structure. XRF analysis is an ideal method for many types of coating structures due to the fact that the technique is non-contact, non-destructive, and requires little, if any, sample preparation. Micro-XRF, with its capability of measuring thickness/composition parameters within a very small area, can make measurements on individual small components or multiple measurements on larger parts, to determine thickness and compositional homogeneity. Coating thickness measurement limits are dictated by the X-ray signal energy and hence have the advantage of the penetrating power of X-rays.

Features of Orbis Coating Software

- Measurement of thickness and composition on up to 5 layers with maximum 10 elements per layer
- Thickness measurements from 1 nm to 50 µm depending on elemental line series available
- Fundamental Parameter algorithm allows for "No Standards" measurement mode and calibration with minimal standards for improved accuracy
- Consistent with coating thickness measurement standards EN ISO 3497 and ASTM B568

Applications

The use of coatings in the modern manufacturing world continues to increase in an effort to improve product performance and reduce product cost. Orbis coating analysis software can be used to measure coating structures employed in a wide variety of fields including:

- Microelectronics packaging and interconnect
- Optical filters
- Photovoltaics
- Anti-corrosion coatings
- Wear resistance
- ROHS applications
- Semiconductors

Example

As an example of the power and utility of using the Orbis micro-XRF system for coating analysis measurements, a CIGS/Mo photovoltaic cell was measured to determine the homogeneity of coating thickness and composition in the solar cell. The CIGS layer is comprised of Cu, In, Ga and Se coated on a Mo layer which in turn is supported on a glass substrate. Measurements made with the Orbis system clearly show how the CIGS layer thickness (Figure 1) varies over this portion of a solar panel, while the Se composition (Figure 2) is relatively homogeneous except for extreme edge effects.

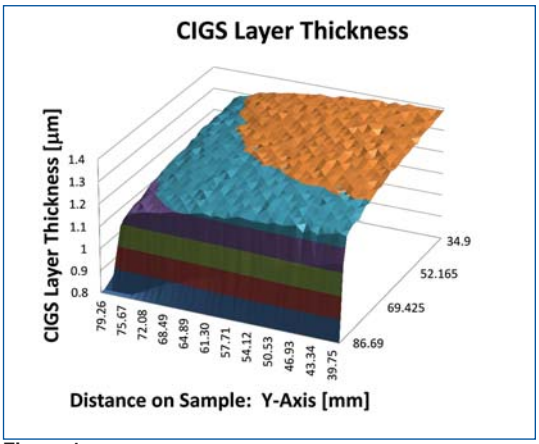


Figure 1:

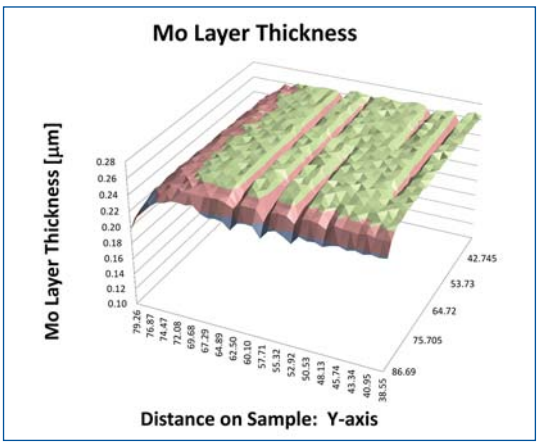


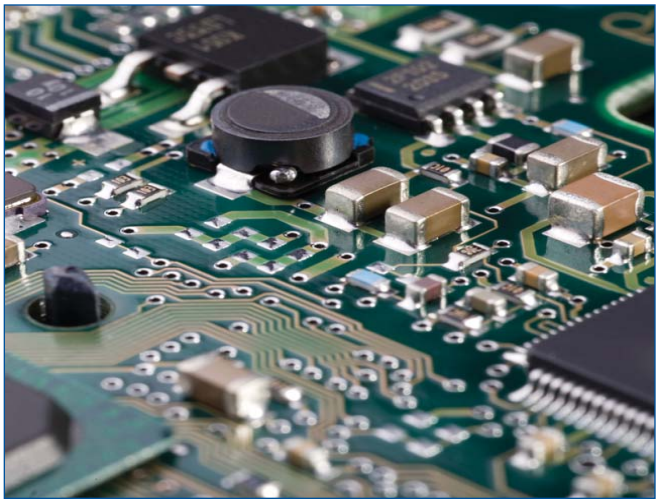
Figure 2:

(Cont'd on page 2)

Orbis Vision Software Version 1.6 Release (Cont'd. from Pg. 1)

Additional Features of Orbis Software Version 1.6

Peak Thresholding: For critical trace element calculations, this allows the user to set a threshold assigning statistical significance to the calculated peak intensity. Elements which fail the thresholding test are flagged as being statistically insignificant for the peak deconvolution routine.



Sample Navigation Tool: This feature allows for a sample image captured from another imaging technique to be used as a navigational map to set target points for analysis in the Orbis. The sample image need only contain at least two reference points visible in the Orbis for calibration of the Orbis stage. The purpose of this software tool is to allow for easy analysis of sample features observed in other imaging modalities, e.g. electron microscopy, optical microscopy, Raman, and FTIR, which may not be easy to target using the Orbis video system.

Windows 7 Compatibility: Vision Software application now operates under Windows 7 or Windows XP (Feature in Orbis Software Version 1.6.1)

Summary

With the introduction of coating analysis software, the Orbis micro-XRF analyzer can now be used to make simultaneous measurements for coating thickness and composition of complex coating structures. Measurements made with the Orbis system are non-contact, non-destructive and require little sample preparation. The Orbis analyzer can be used to make measurements on small individual components or to analyze the homogeneity of coatings on larger components.

Copyright© 2011 All rights reserved EDAX Inc.

Optimizing Structure Files in OIM™ Data Collection

When indexing Electron Backscatter Diffraction (EBSD) patterns, the phase and structure of the sample being investigated must be entered into the OIM™ software. The crystal symmetry and EBSD reflector list, is stored in an EBSD structure file.

The TSL materials database, for the most commonly analyzed phases, is included with each EDAX OIM™ EBSD system. These structure files have been verified against actual patterns and can be used directly. If the phase needed is not available in the TSL database, the user can create it manually or load it from an optional crystal structure database such as the AMCS or ICDD-PDF4 database. However, the reflector lists taken from these databases are often not ideal for EBSD and the operator may need to optimize such imported structure files before they can be used to successfully index EBSD patterns. In OIM™ Data Collection, several tools are available to assist the user in this structure file optimization.

Editing the reflector list

After loading the new structure file, visually verify that the correct orientation has been selected (Figure 1). A missing reflector can be added by drawing a line on the pattern while holding the shift key. It is easier to draw a line between two visible zone axes that are present on the unlabeled line. OIM™ Data Collection will then propose a lattice plane that matches the drawn line. Accepting this plane will display all crystallographic equivalent planes on the pattern (Figure 2).

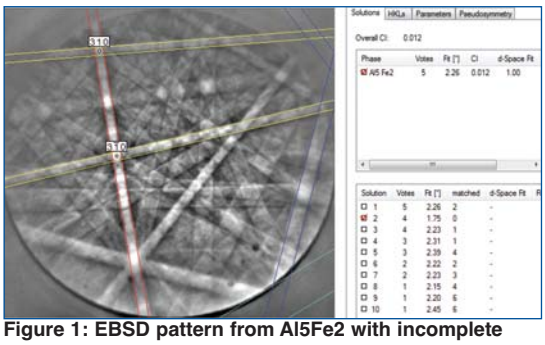


Figure 1: EBSD pattern from Al5Fe2 with incomplete reflector list. The best orientation solution was the 2nd in the table.

When a band that is not visible in the actual pattern is indicated in the solution, it can be removed by shift-drawing a line on top of the colored band. This will disable all equivalent planes in the structure file.

The reflector list can be optimized using this tool to include all visible bands in the pattern. All selected bands will now be used in the triplet voting indexing procedure to determine the crystal orientation.

When the optimization is done on a high resolution pattern, many reflectors can be recognized that will not be visible on the lower resolution images normally used for mapping. For best results these invisible bands should not be used for high-speed indexing.

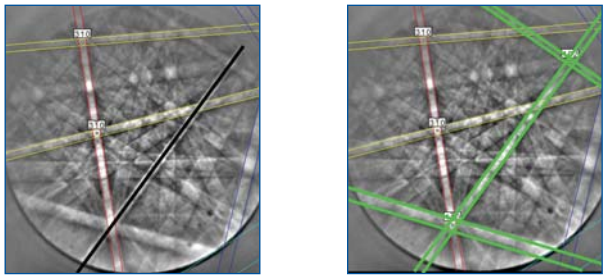


Figure 2: Manual selection of unidentified band with proposed lattice plane and added lines.

Selecting which bands to use for indexing

Another useful tool that can assist the user in identifying which reflectors should be used can be found in the HKL tab on the indexing page. After indexing a pattern, a table is displayed showing how often each reflector was visible in the pattern and how often it was actually detected (Figure 3). These results can be added to the cumulative column by clicking “add to sum”. When this has been done for a number of patterns at the camera preset to be used for scanning, the cumulative column will show which bands have never been detected and used for indexing. These reflectors can now be switched off by deselecting the red checkmark to the left of the reflector. When the blue checkmark is on, the band will be indicated on the pattern, but not used for indexing. Deselecting the blue mark will hide the corresponding reflector.

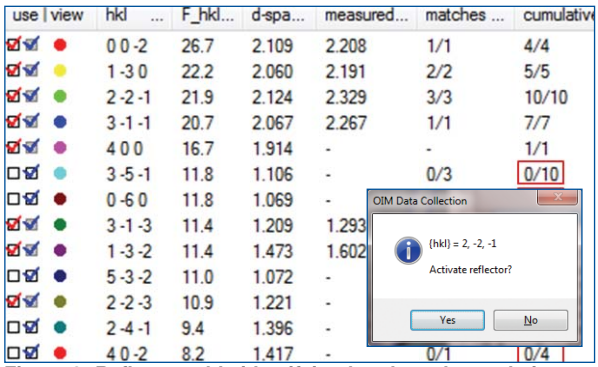


Figure 3: Reflector table identifying bands and cumulative band detection over multiple patterns. The reflectors outlined in red can be switched off as they are never detected.

Copyright© 2011 All rights reserved EDAX Inc.

EDS NEWS

Pharmaceutical Impurity and Compound Analysis with EDS

The pharmaceutical industry is involved in the discovery, development, and manufacture of drugs and drug products. With approximately 3,000 drugs in research in the US, health and safety is a primary concern for this heavily regulated industry. Since the 1937 incident when a toxic drug product was released into the market without safety testing and was responsible for 100 deaths, the FDA has been involved with government oversight in the US. Currently there are several worldwide groups and agencies responsible for setting guidelines for the safety, efficacy, quality, and purity of drug products during the development process and incorporated into Quality Control (QC) monitoring processes.

Analytical methods are a primary component in all aspects of drug design, development, QC, and stability testing. While chromatographic techniques such as HPLC and GC are heavily relied upon to provide very low level and accurate compound analysis, they lack the ability to provide site specific localized chemical information, as in a final product. The ability to analyze drug products in their finished form is particularly important for the investigation of impurities, which may influence the safety and efficacy of the drug product and have regulatory allowable limits. Impurities are the unwanted chemicals that remain with the drug ingredients or those that develop during product formation or aging. They can be process and drug related by-products, degradation products, inorganic residual heavy metals or salts and solvents. These unwanted compounds could be formed as a result of methodology or from environmental conditions, such as temperature, humidity, light exposure, and time.

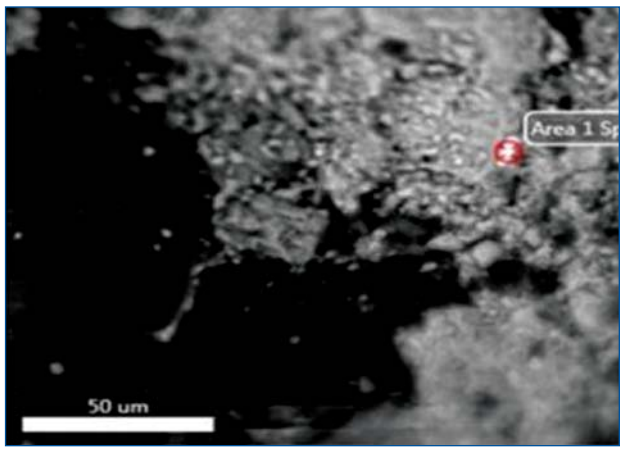


Figure 1: BSE image of Iron supplement tablet.

EDAX EDS analysis has proven to be a powerful technique in studying impurities in their local environment in tablet form. EDAX TEAM™ EDS highlights where the impurities are located, what their chemistry is, and what compounds they are near, all of which provides valuable information on why they are formed. The first example, Figure 1, shows an electron image of an iron supplement tablet containing the active ingredient, FeSO4 among the primary filler, Calcium Carbonate. Spectrum collection shows a typical FeSO4 spectrum (Figure 2) and a spectrum from an impurity that is approximately 10µm in size. The data shows that the impurity product, Figure 3, is a variant of the main constituents of the active ingredient, specifically higher in Fe. Also of interest was that this type of breakdown product was not seen in a different brand tablet, which uses Dicalcium Phosphate as the primary filler.

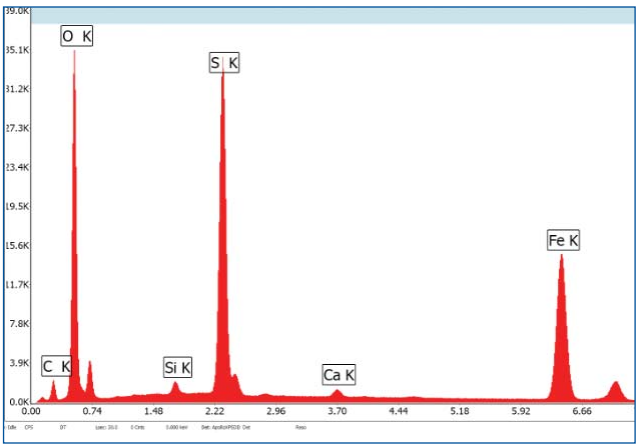


Figure 2: Typical spectrum of active ingredient FeSO4.

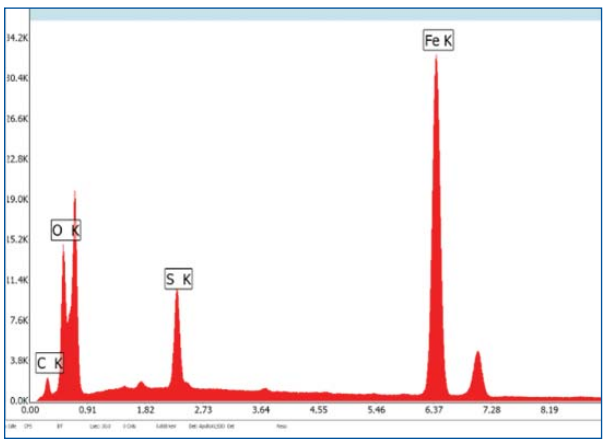


Figure 3: Spectrum of impurity showing variation of active ingredient elements.

(Cont'd on page 5)

Copyright© 2011 All rights reserved EDAX Inc.

Pharmaceutical Impurity and Compound Analysis with EDS

(Cont'd. from Pg. 4)

Figure 4 shows one of several Bismuth and Chlorine containing particulates on the surface of the cross sectioned tablet. These particulates seemed to become more prevalent the longer the tablet was left under SEM vacuum, with many new particulates appearing when the sample was left overnight. As solvents are another type of impurity, this may be explained as being crystallized solvent from the tablet. The EDAX peak deconvolution routine was used to confirm presence of all elements, S, Bi, and Cl, shown in Figure 5, as the best fit with all three identified.

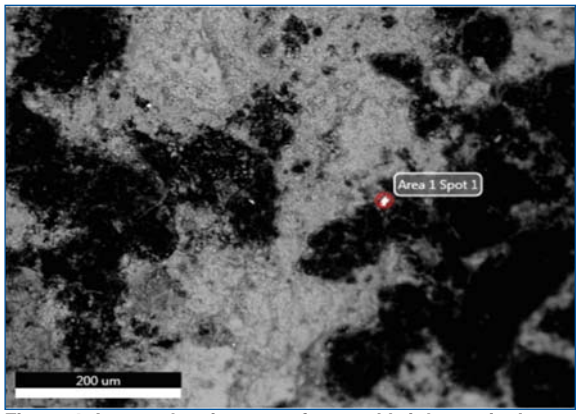


Figure 4: Image showing one of several bright particulates on surface of the tablet.

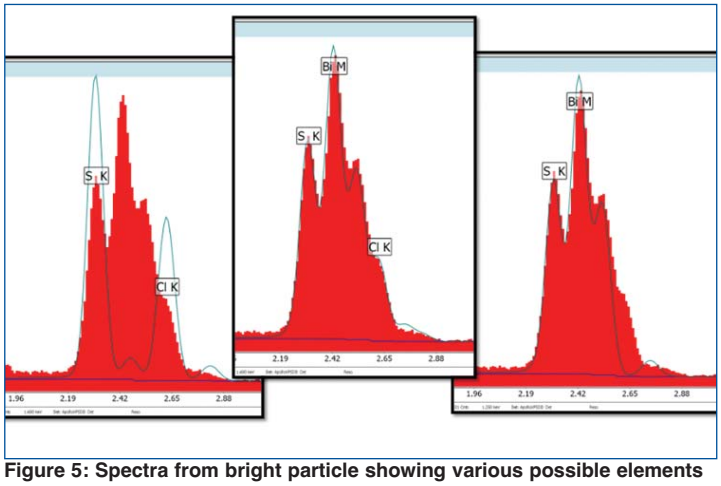


Figure 5: Spectra from bright particle showing various possible elements with peak deconvolution, the best fit achieved with all three elements.

Traditional elemental maps display individual element distribution throughout the area of interest. However, most compounds in pharmaceutical tablets are made up of more than one element, for example, Magnesium Oxide (MgO) or Titanium Dioxide (TiO_2). Side by side comparison of individual maps may allow users to visualize where phases or compounds are present by finding common areas. For example the Calcium and Phosphate maps in Figure 6 allow the assumption of the compound Dicalcium Phosphate. However, some compounds become too complex to visualize comparatively in this way. Therefore a fully automated phase routine with TEAM™ EDS is used to determine all phases present and display them in one single-phase map image, Figure 7.

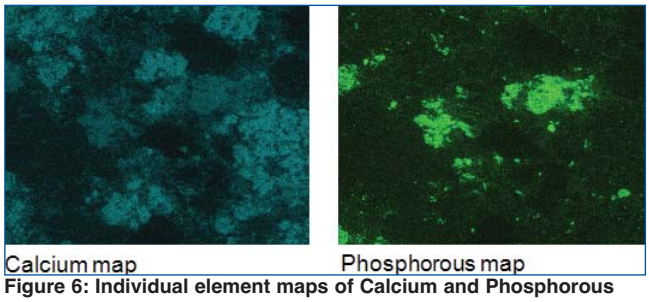


Figure 6: Individual element maps of Calcium and Phosphorous allow some phase or compound visualization be seeing common areas.

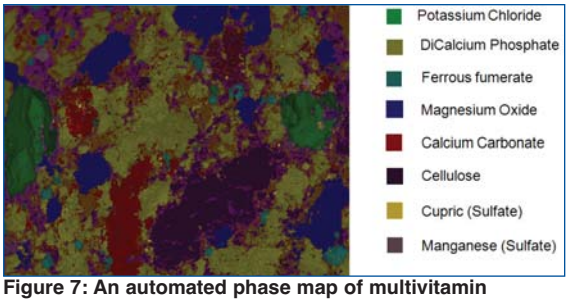


Figure 7: An automated phase map of multivitamin generated with EDAX TEAM EDS software.

Using EDS for local chemical imaging of drug product components in finished form allows insight into the mechanisms of impurity formation. Pharmaceutical companies incorporate QC measures to monitor the presence and extent of impurities. EDS allows them to characterize impurities in finished form better than traditional methods of analysis. This enables them to take more effective measures to prevent impurities in the design process. Reducing the likelihood of impurity formation and improving quality by design can achieve a more streamlined and cost effective overall drug manufacturing process.

Copyright© 2011 All rights reserved EDAX Inc.

TRAINING AND EVENTS

World-Wide Events

December 12-17	The 16th International Conference of the Textures of Materials (ICOTOM 16)	Mumbai, India

***Please see our website www.edax.com for a complete list of our tradeshows

2011 & 2012 World-Wide 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

Tilburg = (T) (in English)
Wiesbaden = (W) (in German, unless stated otherwise):

Tilburg

EDS Microanalysis:

- February 14-16, 2012
- March 15-16, 2012
- March 27-29, 2012
- June 14-16, 2012
- September 13-14, 2012
- October 2-4, 2012
- November 8-9, 2012
- November 20-22, 2012

EBSD (OIM™):

- March 12-14, 2012
- June 11-13, 2012
- September 10-12, 2012
- November 12-14, 2012

WDS LEXS:

- April 24-26, 2012
- October 23-25, 2012

Wiesbaden

EDS:

- Nov. 29 - Dec. 1, 2011
- June 12-14, 2012

Pegasus: (EDS/EBSD)

- Feb. 27 - Mar. 1, 2012
- November 5-8, 2012

EDS (TEAM™) & WDS TEXS

- May 8-11, 2012

Orbis: Course & Workshop Presented in English

- March 6-7, 2012
- March 8-9, 2012
- October 23-25, 2012

Japan

EDS Microanalysis:

2012

February 9-10	Tokyo
April 12-13	Osaka
June 7-8	Tokyo
July 12-13	Osaka
October 11-12	Tokyo
November 8-9	Osaka

EBSD (OIM™):

Dec. 7-8, 2011	Tokyo
February, 2012	Tokyo
May, 2012	Tokyo
June, 2012	Osaka
August, 2012	Tokyo
December, 2012	Tokyo

China

EDS:

- March 13-15, 2012
- June 19-21, 2012
- September 4-6, 2012

Particle Analysis:

- December 6-8, 2011
- December 4-6, 2012

For more information on our training classes, please visit our website at:
www.edax.com/support/training/index.aspx

North America

EDS Microanalysis:

2012

April 23-27	Mahwah, NJ
May 22-24	Mahwah, NJ
July 10-12	Draper, UT
September 25-27	Mahwah, NJ

EBSD (OIM™):

May 29-31	Draper, UT
October 23-25	Mahwah, NJ

Pegasus: (EDS/EBSD)

March 26-30	Mahwah, NJ

EDS Particle Analysis:

January 24-26	Mahwah, NJ

WDS:

November 13-15	Mahwah, NJ

Micro-XRF:

April 10-12	Mahwah, NJ
October 2-4	Mahwah, NJ

Copyright© 2011 All rights reserved EDAX Inc.

EDAX's Summer Student Learning Program

Aneeka Ayyar

Brandon Ilie


Shannon Oscher

This summer EDAX opened its laboratory doors to a different group of scientists-talented local high school students with an interest in exploring science projects with SEM and EDS. The learning opportunity was created to give students a unique experience in laboratory sciences and also to remind EDAX of how first time, novice users approach the use of TEAM™ EDS software. Within a few short sessions, the students were able to design a scientific project and gather the data needed to make a presentation of what they learned from their study.

The family members of several employees were able to take part in this project, despite busy summer academic and athletic pursuits. Aneeka Ayyar and Brandon Ilie, tenth graders, and Shannon Oscher a ninth grader, participated and brought several interesting ideas for the project. Each student was asked to pick a topic of interest from several provided topics or to suggest their own. Project topics included:

- Measuring the variations of copper in a penny and the comparison of a 1956 penny to a 2011 penny
- Testing for heavy metal or harmful elements by comparing basil leaves treated with organic fertilizer and leaves treated with harsher chemicals. Both leaves were from the same family garden
- A study of the difference between an extended release Iron supplement tablet and a regular release tablet to understand how dosage differences may affect drug delivery
- A study of variations of rock chemistry from a rock collection and an understanding of how different minerals appear different to the eye and under the SEM electron beam

The students were asked to design an experiment using proper scientific method and to include a hypothesis, background information on their topic, data collection with SEM/EDS, and a logical conclusion.

During the first session, laboratory managers Tara Nylese and Bob Anderhalt discussed basic atomic and electronic concepts with the students. Subsequent sessions gave the students hands on time with the variable pressure SEM for non-conductive work and the FESEM for high resolution capability. The students spent time on their own samples focusing the SEM, increasing the electron beam current for high X-ray count production and collecting images at various magnifications. Of particular interest was how rough and dirty an old penny looked close up.

With the TEAM™ EDS software, the students quickly navigated their way through many of the functions of multi-area quantitative analysis and phase mapping for visual chemical distribution. Being an avid math scholar, Shannon enjoyed gathering a series of quantitative values for the copper and zinc weight percentages, which she ultimately summarized in charts for an easy to understand explanation of the data. Brandon was able to identify the chemistry components of Iron supplement tablets and use the phase mapping software to characterize chemical compounds including Iron Sulfate ($FeSO_4$) and Dicalcium Phosphate, which matched up to the ingredients labels. With the use of the standard report template, he exported all of his data into the images, maps, and spectra that he used in his presentation. Several of Aneeka's rocks contained Lead, Carbon, and Oxygen. A look at the quant numbers showed atomic ratios near 1:1:3, which forms a $PbCO_3$, compound which is a substitution of Ca from Calcite, $CaCO_3$.

Finally, with the students' data compiled and conclusions drawn, they returned to EDAX to present their findings and to receive certification as EDAX certified microanalysts. Their presentations to a small group of EDAX employees were well received and showed the amount of thought and preparation each had spent creating a high quality scientific study. The dedication to their work and engagement in all aspects from planning through research to presentation ensures they will have strong futures in math and science.

Copyright© 2011 All rights reserved EDAX Inc.

CUSTOMER NEWS

Oak Ridge National Laboratory ShaRE Scientific User Facility

The Shared Research Equipment (ShaRE) User Facility at the Oak Ridge National Laboratory (ORNL) is located in Oak Ridge, TN. It is a multidisciplinary scientific user facility supporting high-impact science both within the Oak Ridge National Laboratory and for external users from all over the world. ShaRE's user base includes materials researchers focused on metallurgy, ceramics, nuclear materials/radiation materials science, fuel cells, and battery materials.

ShaRE has the following systems on site:

- EDAX Si(Li) on Philips CM200 analytical TEM/STEM
- EDAX Si(Li) on Hitachi S3400N SEM
- EDAX Apollo SDD + DigiView IV on Hitachi NB5000 FIB-SEM
- EDAX Apollo SDD + Hikari on JEOL J6500F SEM
- EDAX Apollo XL + Hikari S-4800

Chad M. Parish, Ph.D., a Staff Scientist with the EDAX Apollo SDD + Hikari EBSD camera on a JEOL J6500F SEM.

One of the principal users of these systems is Chad M. Parish, Ph.D. Chad is a Staff Scientist at ShaRE. He has a doctorate from North Carolina State University, where he studied under Professor P. Russell, one of the foremost practitioners and teachers of SEM in the US. Chad also performed post-doctoral studies at Sandia National Laboratory, a leading SEM and STEM spectrum imaging facility.

ORNL's ShaRE User Facility instrument user base includes professional microscopists within the ShaRE microscopy group and from other external facilities, experienced microscopists in the ORNL Materials Science and Technology Division and also scientists without a microscopy background both from the facility and from around the world.

ShaRE users study a diverse array of problems in fields ranging from materials engineering to solid-state physics. The Apollo EDS detector + Hikari EBSD system on the 6500F SEM, for example, is used in studying texture and phase evolution of metals under extreme processing conditions.

ShaRE users have also used the Apollo + Hikari system for simultaneous EBSD and EDS analysis to quantify cubic carbides and retained austenite in martensitic stainless steel matrices. When these results were combined with the measurement of mechanical properties, it was possible to optimize processing parameters of the steel to enable maximum performance with minimum manufacturing energy expense. This enabled the microscopy user facility to support other Department of Energy programs.

As the Apollo + Hikari system at ShaRE is very heavily used, the fact that users could collect data on the microscope and then analyze their EDS+EBSD results offline was essential to the timely solution of the martensite + austenite + carbide resolution problem.

ORNL's Shared Research Equipment (ShaRE) User Facility is sponsored by the Office of Basic Energy Sciences, U.S. Department of Energy. www.ornl.gov/share

Art & Layout:
Beverlee Boddy

Contributing Writers:
Rene de Kloe
Tara Nylese
Bruce Scruggs
Craig Theberge

Please visit our new website at www.edax.com

Copyright 2011 EDAX Inc. No part of this publication may be reproduced, stored in a retrieval system, or transmitted in any form or by any means without prior written permission of EDAX Inc.

EDAX
advanced microanalysis solutions

AMETEK
MATERIALS ANALYSIS DIVISION

EDAX Inc. ● 91 McKee Drive Mahwah, NJ 07430 ● Phone: (201) 529-4880 ● E-mail: info.edax@ametek.com ● www.edax.com

Copyright© 2011 All rights reserved EDAX Inc.