July 2022
Volume 20 Issue 2

EDAXinsight

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NEWS

Characterizing directionally solidified Al-Cu-Mg eutectic alloys with combined EDS-EBSD

The role of solidification in the development of microstructures and material properties is becoming more critical as processing techniques, including additive manufacturing, that involve solidification are becoming more prevalent. In this work, based on a paper by Ezemenaka and Genau (*Journal of Crystal Growth* **577** (2022) 126389), an Al-Cu-Mg alloy processed via directional solidification has been characterized using simultaneously collected Energy Dispersive Spectroscopy (EDS) and Electron Backscatter Diffraction (EBSD) to investigate the phase and orientation relationships that develop in this ternary eutectic alloy.

Samples with a eutectic composition (Al – 15.5 at% Cu – 10.6 at% Mg) were melted in an electric furnace and directionally solidified using a Bridgman-type furnace, where solidification conditions were controlled to establish a stable microstructural growth pattern. Examples shown here are from a sample with a 49 mm growth height. Transverse sections parallel to the solidification direction were prepared for EBSD analysis, first by mechanical polishing down to 0.1 µm diamond paste, followed by 2 hours of polishing with 0.05 µm colloidal silica on a vibratory polisher. Final EBSD preparation was performed using a Gatan PECS™ II Broad Beam Ion Polisher via a 2-step milling routine. The sample was first milled with a 4 kV argon beam with a 4° glancing angle for 10 minutes, followed by a 2 kV beam at 4° for 60 minutes.

EBSD data was collected using a Clarity™ Direct Detector operating at 20 kV acceleration voltage and approximately 1 nA beam. EDS data was collected simultaneously with the EBSD data using an Octane Elite Detector. The microstructure is visualized by the three PRIAS™ images shown in Figure 1.

AMETEK®

NEWS

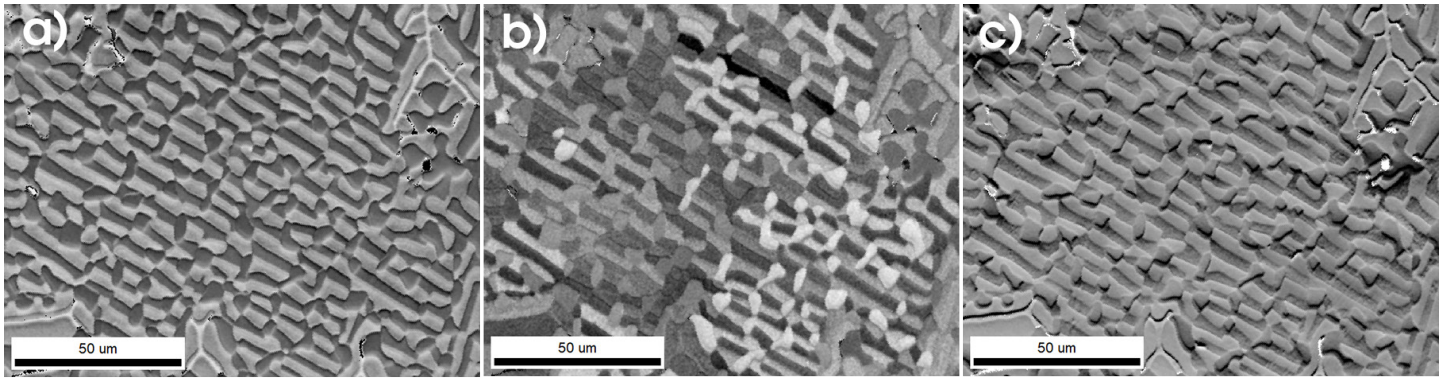

Figure 1. a) PRIAS top ROI image showing atomic number contrast, b) PRIAS middle ROI image showing orientation contrast, and c) PRIAS bottom ROI image showing topographic contrast for the directionally solidified Al-Cu-Mg alloy.

Figure 1a shows atomic number contrast from the sample in the top region of interest (ROI) from the PRIAS image. Figure 1b shows the center ROI PRIAS image, which displays orientation contrast from the different grains and phases. Figure 1c highlights the surface topography present on the sample after preparation in the bottom ROI PRIAS image. This multi-phase sample exhibits surface topography due to the differential polishing rates of the constituent phases.

Figure 2 shows a composite EDS map from the chemical data collected simultaneously with the EBSD data. The red channel corresponds to the copper elemental information, the green channel corresponds to aluminum, and the blue channel corresponds to magnesium.

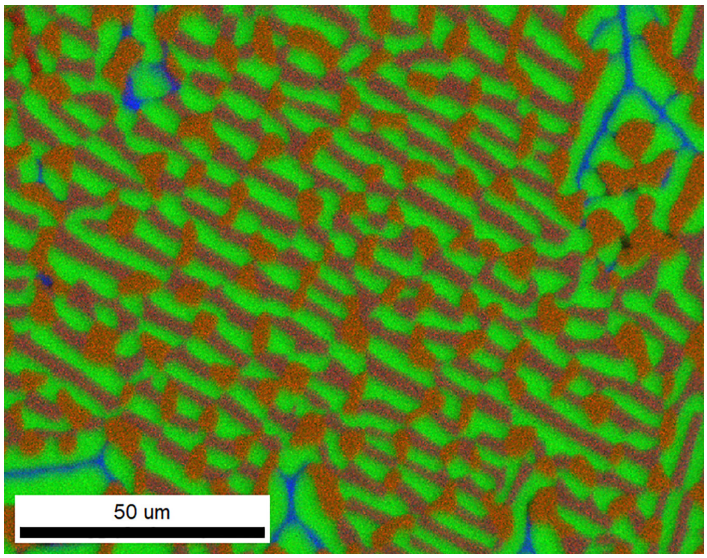

Figure 2. Composite EDS RGB map where the red channel corresponds to the copper signal, the green channel corresponds to the aluminum signal, and the blue channel corresponds to the magnesium signal.

The EDS information was used for Chl-Scan™ analysis for the most accurate and efficient phase mapping, as shown in Figure 3. NPAR™ was also applied during the Chl-Scan indexing to improve the signal-to-noise ratio of the saved EBSD patterns and subsequent indexing results. The phase map shows the expected two-lamellar/one-rod pattern standard in this ternary eutectic alloy. The aluminum and Al_2CuMg phases form the lamellar phases, and the Al_2Cu forms the rod phase. The grain size and morphology of each phase are easily measured with this EBSD data. A cored Al-Si-Mg phase was also detected and is attributed to contamination from an unknown source.

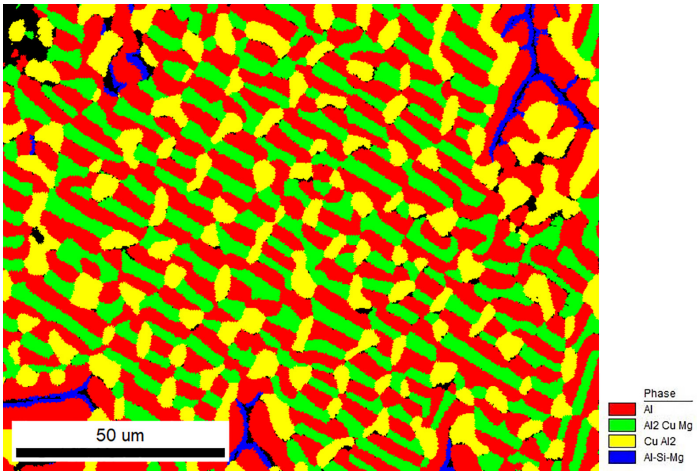

Figure 3. Phase map of the ternary eutectic Al-Cu-Mg alloy measured using combined EDS-EBSD data via Chl-Scan analysis.

Inverse Pole Figure (IPF) maps for the aluminum, Al_2CuMg, and Al_2Cu phases are shown in Figure 4, with the orientations colored relative to the normal direction of the analysis surface. These orientation maps show a strong preferred orientation for each constituent phase.

NEWS

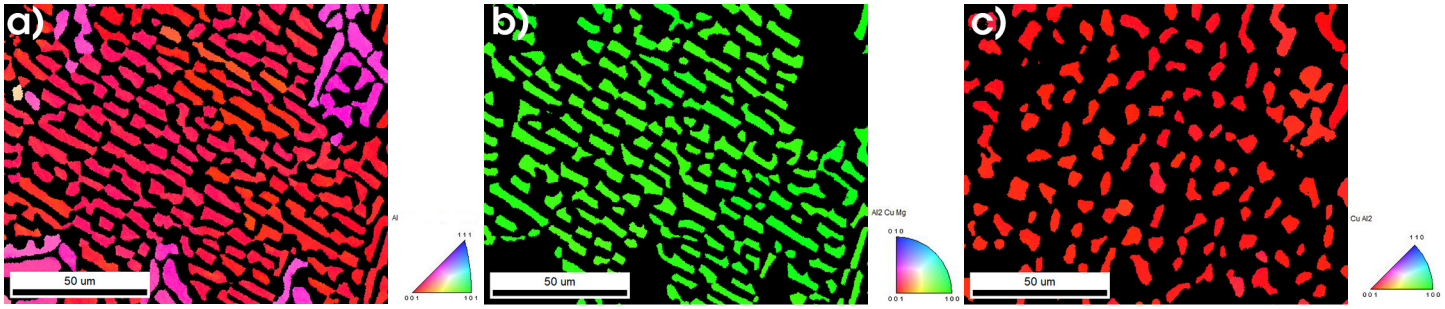

Figure 4. IPF orientation maps for the a) aluminum, b) Al_2CuMg, and c) Al_2Cu phases, with the orientations colored relative to the surface normal direction of the measured surface.

Analysis of these orientations shows that the (001) crystal direction of the aluminum phase is parallel with the (001) direction in the Al_2Cu and also parallel with the (100) direction in the Al_2CuMg phase. These orientation relationships can be visualized by plotting the colored orientations in inverse pole figures for each phase, as shown in Figure 5. It is also interesting to note that by comparing Figures 3 and 4, it is observed that aluminum grains adjacent to the cored Al-Si-Mg phase have an orientation that deviates from both the other grains and the expected orientation relationships.

This work shows how combined EDS and EBSD can be used to fully characterize the orientations and orientation relationships present in a complex ternary eutectic alloy prepared by directional solidification. The application of the Clarity, Octane Elite, ChI-Scan, and NPAR processing allows for optimal EBSD pattern indexing from each phase present for accurate results. We want to acknowledge the kind permission from Dominic Ezemenaka, previously at the University of Alabama at Birmingham and now at the University of Alabama, for providing the samples and sharing the EBSD data.

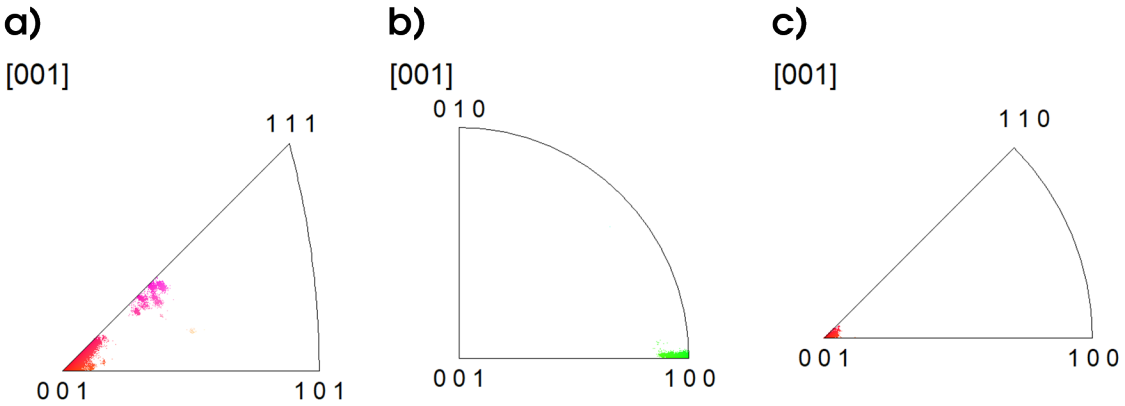

Figure 5. IPF plots for the a) aluminum, b) Al_2CuMg, and c) Al_2Cu phases, with the orientations colored using the same color scheme from Figure 4.

TIPS & TRICKS

Reindexing data using ChI-Scan with non-ideal experimental conditions – how to work with shadowing and no EDS data

ChI-Scan™ can be an essential tool to help index patterns for Electron Backscatter Diffraction (EBSD) on some samples. ChI-Scan generally uses Energy Dispersive Spectroscopy (EDS) data as a filtering agent to guide the indexing process. In some situations, combining EBSD and EDS data can be tricky. Adding an *in-situ* accessory, like a heating or cooling stage, can compound these issues. Recently, a data set was collected using a Velocity™ Super EBSD Camera and an Octane Elite EDS Detector. The sample was heated to 800 °C using a Gatan Murano heating stage so that a phase change could be analyzed by EBSD. When a sample is heated to 800 °C, blackbody radiation at infrared wavelengths is emitted. Typically, to lower the IR signal, which would cause problems with the EDS signal, the line of sight is limited, or an IR filter is placed on the EDS detector. In this situation, neither option was feasible. Additional issues can be caused by shadowing of the phosphor screen.

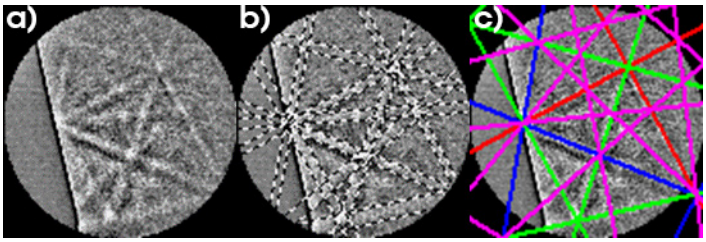

Figure 1. a) Pattern as collected shows shadowing of the phosphor screen on the left side. b) The sharp contrast caused by the shadowing is usually not identified as a line by the Hough Transform. c) If a line is found, the indexing algorithm will often find the correct solution.

For this data set, data was collected at 1,000 frames per second at the standard 4 x 4 binning. Patterns were saved to post-process the data in OIM Analysis™ using a combination of NPAR™ and ChI-Scan. Since EDS data was not collected, non-elemental data was required to filter the data accordingly.

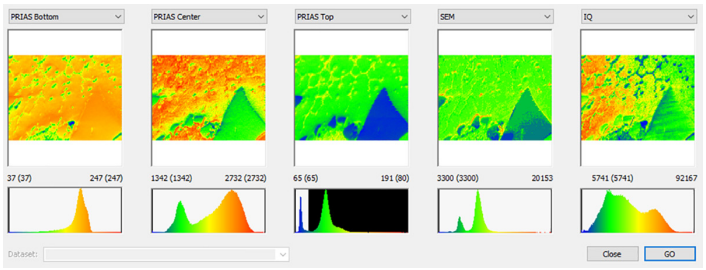

Figure 2. Non-elemental datasets are collected at the same time as EBSD scans. These five datasets show differences in signal related to electron channel contrast, collection angle, and density of the material.

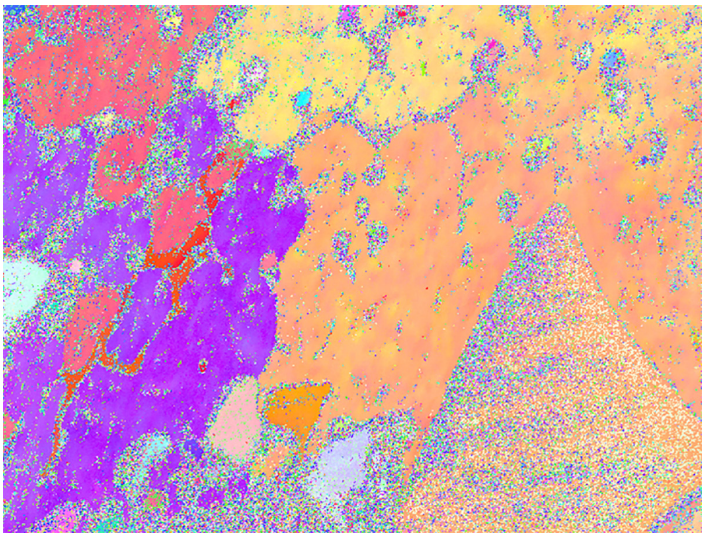

Figure 3. The as-collected IPF map shows issues with grain boundaries and some precipitates. The field of view is approximately 50 µm.

By default, five non-elemental datasets were saved with a standard EBSD dataset, PRIAS top, PRIAS bottom, PRIAS center, Image Quality (IQ), and Scanning Electron Microscope (SEM) signal (Figure 2).

The as collected Inverse Pole Figure (IPF) maps (Figure 3) showed some issues, especially where image quality was lower and with smaller grains.

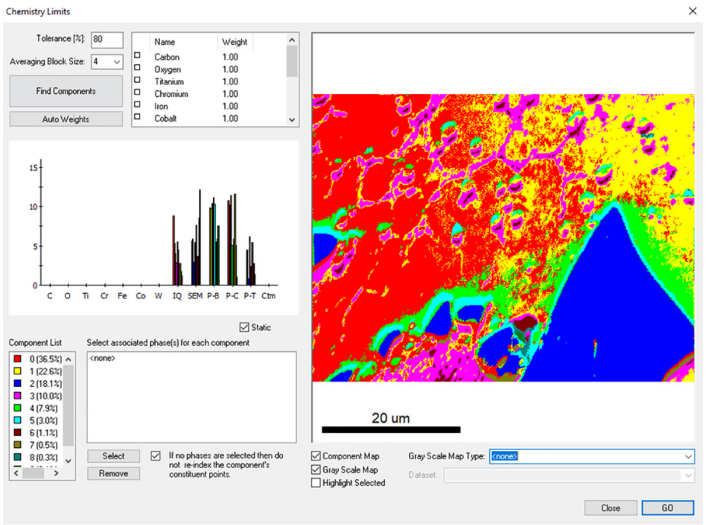

Figure 4. Using the Phase Cluster approach on this sample lead to some grain boundary effects that made the process more difficult. In a case like this, it may be better to use the manual approach.

TIPS & TRICKS

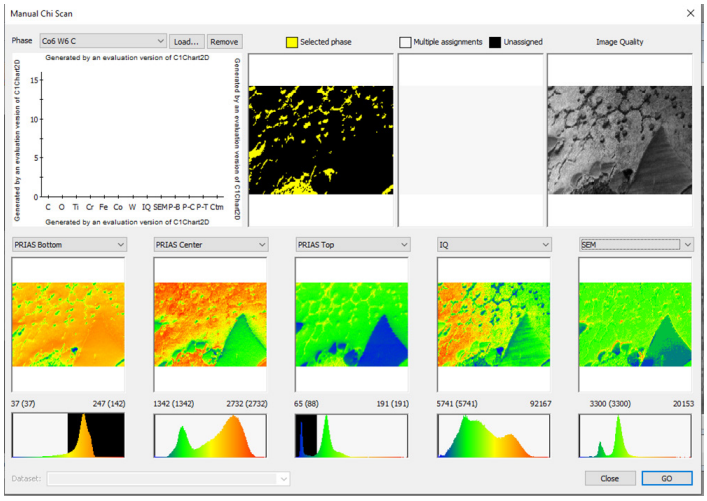

Figure 5. Manual mode allows the user to manually select the data sets and the upper and lower limits used to filter that data. Here, two channels were used by selecting the lower tail of the PRIAS bottom signal and the higher values of the PRIAS top filter the matrix material out to be assigned to a phase.

ChI-Scan has two modes: Phase Cluster Analysis (PCA) and Manual mode. PCA is an algorithm-based method that groups the pixels together, and then the user assigns the phase or phases (Figure 4). The Manual mode (Figure 5) allows the user to manually choose the upper and lower values to filter the data. The user can use any combination of the five selected signals for that phase. Depending on the filtering chosen, each pixel can be used for single or multiple phases.

In this sample, the PCA has issues due to some grain boundary effects, probably caused by the still ongoing diffusion in the sample. Manual mode was selected for each phase instead. Each phase was assigned by using up to three data channels. After all the phases were set for filtering, the data was reprocessed by clicking the ChI-Scan button (Figure 6).

Figure 6. ChI-Scan is run by pressing the larger button with the X in it. The PCA checkbox toggles between PCA and Manual mode. The button below allows ChI-Scan to be run in batch mode.

After the data was processed, the new IPF map (Figure 7) appeared in the project tree, allowing further analysis of the data set or allowing the user to iterate the process as needed. In the end, using non-EDS signals for phase filter via ChI-Scan can be just as useful as the EDS signal itself. In cases when no EDS data is available, the ability to do so can make the experiment successful.

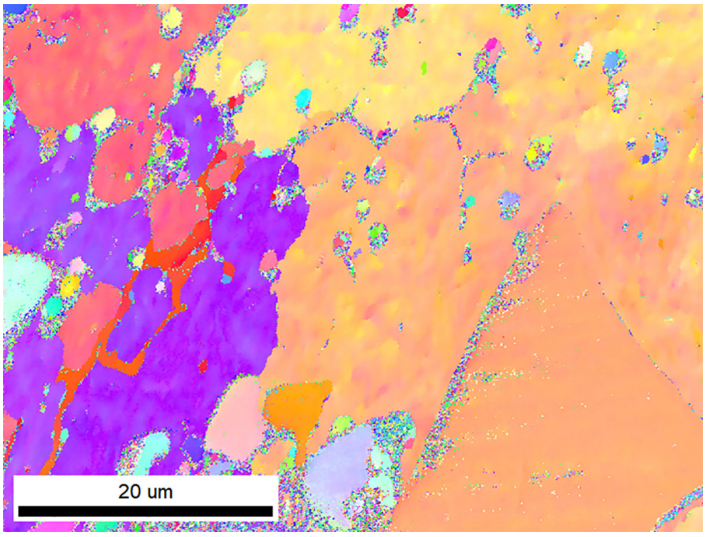

Figure 7. The data after processing via NPAR and ChI-Scan is improved and suitable for most analytical work. Further refinements or cleanup steps could be used on the data if needed.

EXPERIMENT BRIEF

Fast-track the search for inclusions and trace elements with APEX Software

Instruments Used

APEX™ Software for EDS and Octane Elite Super EDS Detector

Background

A common goal of microanalysis by energy dispersive spectroscopy (EDS) is to reveal inclusions within a sample. Often, the composition of an inclusion is not known before analysis and will only form a small fraction of the area analyzed. Consequently, they can be challenging to uncover by conventional EDS mapping even for an experienced microanalyst due to the relatively few X-ray counts produced. During EDS mapping, a cumulative spectrum (summed over all points) is displayed to identify elements present in the sample and their elemental maps. This is a powerful means of identifying major elements and phases but hides impurities or inclusions that only make up a small contribution to the total signal. However, APEX Software for EDS enables users to quickly identify impurities even if they are of unknown constituent elements.

Materials and Methods

A phenocryst in a granite sample was analyzed using an Octane Elite Super EDS Detector and the Montage Map optional module of APEX Software; a 6 cm^2 area was examined in ~120 min. The cumulative spectrum (Figure 1a) revealed >14 major elements incusing Al, Si, Ti, O, P, S, Ca, K, and Fe; at least three major phases were identified (Figure 1b). With APEX Software for EDS, a 'maximum pixel' spectrum was built (Figure 1a), displaying the highest number of counts for each energy channel in any spectrum, and revealing the presence of Mn. With this information, the elemental map of Mn was rebuilt (Figure 1c), showing small iron-titanium oxide inclusions containing ~5 wt. % Mn that were completely overlooked previously. Since each data point is based on a single pixel, the maximum pixel spectrum may appear noisy. Still, it highlights elements that are present below the signal-to-noise ratio in the cumulative spectrum. In this example, Mn contributed <0.01 % of the total number of X-rays collected but was detected quickly, easily, and reliably using APEX Software.

Summary

With the Maximum Pixel Spectrum feature of APEX Software for EDS, impurities and inclusions can be identified quickly and reliably, making it easy for users to find the needle in the haystack.

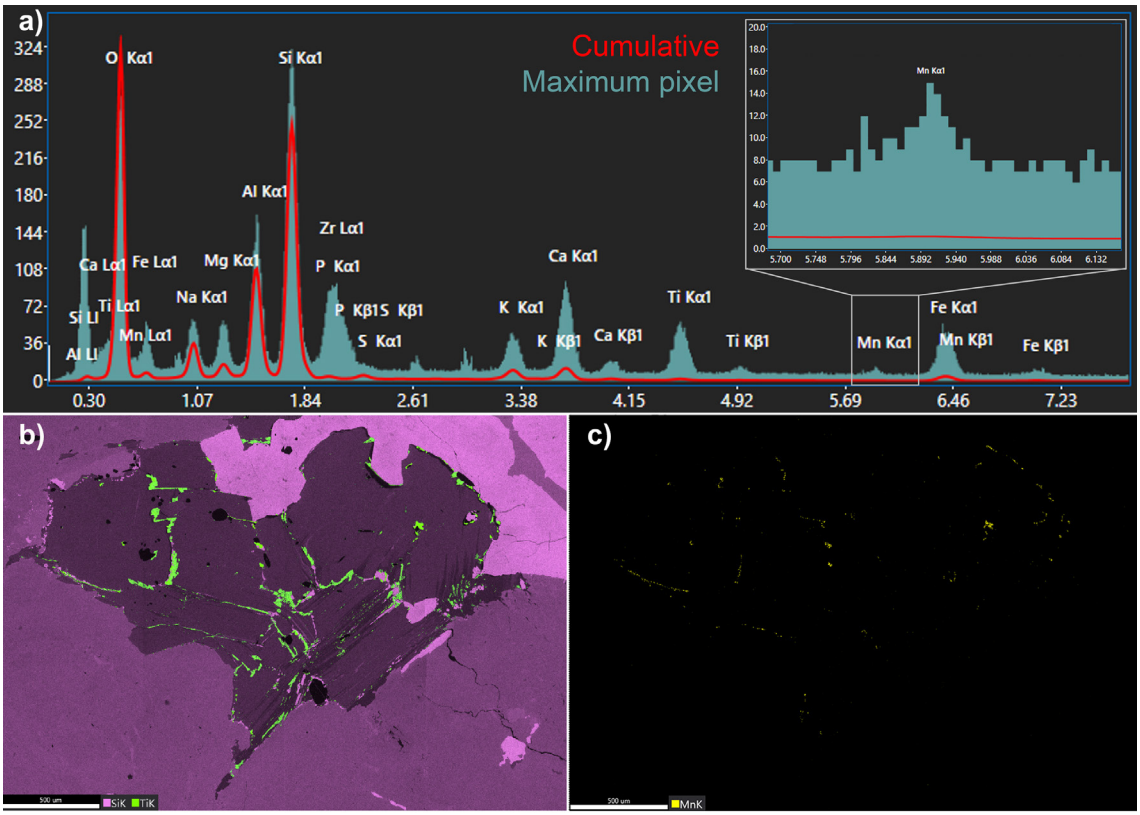

Figure 1. a) Comparison of the cumulative (red line) and maximum pixel (teal) spectra with inset showing a narrower energy range highlighting that manganese can only be observed in the maximum pixel spectrum. b) A colorized overlay of the Si K (purple) and Ti K (green) elemental maps of the granite sample. c) The Mn K map was rebuilt from the maximum pixel spectrum revealing the presence of small iron-titanium oxide inclusions.

EVENTS AND TRAINING

2022 Worldwide Events

Microscopy & Microanalysis (M&M)
August 1 – 4 *Portland, OR*

Ceramics Expo
August 29 – 31 *Cleveland, OH*

Multinational Congress on Microscopy (MCM)
September 4 – 9 *Brno, Czech Republic*

International Conference on Microscopy (ICM)
September 6 – 7 *Prague, Czech Republic*

Midwestern Association of Forensic Scientists
September 11 – 16 *Des Moines, IA*

SEMICON China
October 5 – 7 *Shanghai, China*

MS&T/The Nanotechnology Show/Advanced Materials Show
October 11 – 12 *Pittsburgh, PA*

ISTFA
November 13 – 17 *Pasadena, CA*

Visit https://www.edax.com/news-events/conferences-tradeshows for a complete list of events.

2022 Worldwide Training

Europe

APEX EDS
October 10 – 11 *Unterschleissheim**
November 7 – 8 *Unterschleissheim#*

APEX EBSD
October 12 – 14 *Unterschleissheim**
November 9 – 11 *Unterschleissheim#*

APEX Pegasus (EDS & EBSD)
October 10 – 14 *Unterschleissheim**
November 7 – 11 *Unterschleissheim#*

**Presented in German*
#Language to be determined

Japan

EDS Microanalysis (APEX™ EDS)
July 22 *Virtual (Basic)*
October 7 *Virtual (Advanced)*
November 11 *Virtual (Basic)*

China

EDS Microanalysis
September 6 – 8 *Shanghai (ACES)*
December 6 – 8 *Shanghai (ACES)*

EBSD OIM Academy
August 16 – 18 *Shanghai (ACES)*
December 15 – 17 *Shanghai (ACES)*

North America

EDS Microanalysis
August 16 – 17 *Virtual*
November 8 – 9 *Virtual*

Pegasus (EDS & EBSD)
October 11 – 14 *Virtual*

Visit https://www.edax.com/support/training-schools for a complete list and additional information on our training courses.

EVENTS AND TRAINING

Visit the Gatan/EDAX booth at Microscopy & Microanalysis 2022

Booth #2071

Visit the official EDAX and Gatan M&M 2022 webpage to get all the latest news and to book demos: edax.com/mm2022

Demonstrations

- Octane Elect
- Octane Elite
- Velocity™
- Clarity™
- Pegasus
- APEX™
- OIM Analysis™

Lunch & Learns

Monday, August 1 12:30 – 1:00 p.m.
Cutting Edge EBSD Detector Technology
Presenter: Matt Nowell

Wednesday, August 3 12:30 – 1:00 p.m.
Recharge Your Lithium Research – Workflow and Tools for the Characterization of Lithium Ion Batteries
Presenter: David Stowe

Vendor Tutorial

Tuesday, August 2 5:45 – 6:45 p.m.
Get More from Your EBSD Data with OIM Analysis
Presenter: Matt Nowell

Posters/Presentations

Monday, August 1 3:00 – 5:00 p.m.
The Promises When WDS Supports the EDS X-ray Analysis in SEM and the Evaluation Algorithms Do Merge
Presenter: Frank Eggert
Session: A05.P1
Poster: 809

Tuesday, August 2 11:00 – 11:15 a.m.
SEM EDS Mapping of Ultra-Low Energy X-rays Using a Silicon Nitride Window Silicon Drift Detector
Presenter: Dr. Shangshang Mu
Session: A05.3
Presentation: 115

Tuesday, August 2 3:00 – 5:00 p.m.
Electron Backscatter Diffraction Analysis of Beam Sensitive Samples Using Direct Detection Technology
Presenter: Matt Nowell
Session: P08.P2
Poster 1003

EMPLOYEE SPOTLIGHT

Huaping Sheng

Huaping joined EDAX/Gatan as an Applications Specialist in 2022. Based in Shenzhen, China, he works with customers in South China. Huaping's responsibilities include giving product presentations and demonstrations to highlight features and capabilities, technical support, post-sales customer training and applications support, seminars, workshops, and promoting products at professional conferences. He helps train colleagues in sales, service, and applications.

Before joining AMETEK, Huaping was a postdoctoral researcher at the Erich Schmid Institute of Materials at the Austrian Academy of Sciences in Leoben, Austria, from 2019-2021. He focused on Transmission Electron Microscope (TEM)-based investigation of microstructures and mechanical behaviors in metallic glass. In 2018, he served as a Failure Analysis Engineer at Yangtze Memory Technologies Co., Ltd. (YMTC) in Wuhan, China.

Huaping graduated with a bachelor's degree in Materials Physics from Wuhan University in 2013. He was part of the materials characterization joint program at Argonne National Laboratory in Lemont, IL, from 2015-2017. Huaping received his doctoral degree in Materials Physics and Chemistry from Wuhan University in 2018. As a Ph.D. student, he worked with EDAX equipment on the TEM.

Huaping is married to his wife, Judy. In his spare time, he enjoys taking walks and hiking. Huaping likes listening to various styles of music, especially heart-touching pop songs by Michael Jackson.

Lindsay Stack

Lindsay joined EDAX and Gatan as Human Resources Manager in February 2022. Located in the Warrendale, PA office, her responsibilities include recruiting and hiring, training and development, retention, payroll, benefits, compliance, exit formalities, employee relations, and conflict resolution.

Before joining EDAX and Gatan, Lindsay worked at PPG Industries in Pittsburgh, PA, from 2012 – 2020. She held several roles, including supporting the company-owned paint stores and benefits department and developing the employee relations structure. Her most recent position was Strategic HR Partner supporting global IT and digital.

A lifelong learner, Lindsay received her bachelor's degree in English Writing from the University of Pittsburgh in 2007. She earned three master's degrees in Elementary Education from Chatham University in 2008. Lindsay completed a dual Master of Business Administration and Master of Science in Human Resources from Robert Morris University in 2020.

Lindsay and her husband, Tom, have two children, Henry and Gianna. Lindsay enjoys traveling, yoga, and hiking with her dog in her spare time.

CUSTOMER NEWS

A brief review of EBSD sample preparation

Pranabananda Modak[1*]

[1]Mars Scientific – Authorized Service Partner for EDAX,
*pranabm@mars-scientific.com

Introduction

Electron backscatter diffraction (EBSD) has emerged as a valuable analytical technique for materials science and earth sciences. EBSD permits statistical data on crystallographic orientation/texture, grain size, grain boundary, strain, and phase, which are critical parameters in determining the strength of crystalline materials. At the same time, sample preparation is an absolute prerequisite for the technique. As the backscattered electrons come from the first few nanometers (20 – 50 nm) of the sample's surface, sample preparation is crucial to achieving a useable and representative EBSD pattern. Making the sample surface clean, smooth, and damage-free is necessary. Rough or damaged surfaces and contamination could produce a weak pattern. However, some samples, such as fracture samples and crystal facets, do not require any sample preparation. Sometimes coating is required to make the sample conductive. A non-conducting sample may result in drift due to charging. Another vital challenge in sample preparation is that no single technique can be used for all materials. Hence, the selection of a suitable technique and the optimization of relevant parameters are very important. Several sample preparation techniques are used for EBSD, which are discussed below.

Conventional technique

Mechanical and electro-polishing are the conventional, as well as most popular, techniques for sample preparation. But the process involves many steps, including cutting, multiple rounds of emery paper grinding at different grit sizes, diamond polishing, colloidal silica polishing, ultrasonic cleaning, vibratory polishing, and electro-polishing[1]. To get the desired surface smoothness, additional time is required to repeat several steps. The grinding and polishing procedure is summarized in Table 1. In addition, the method requires excellent operator skills[2].

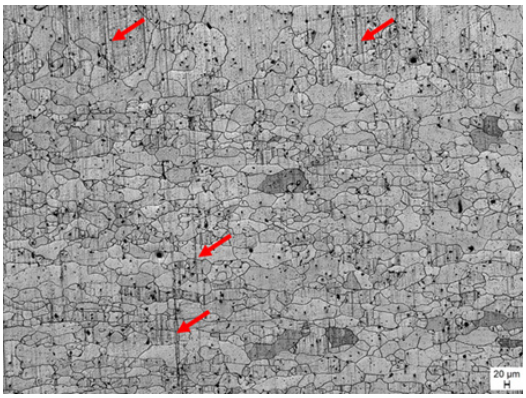

Figure 1. SEM image showing scratches due to improper mechanical polishing in a ferritic stainless-steel sample.

A little extra pressure during polishing can produce mechanical damage on the sample surface, as shown in Figure 1. Soft materials are especially prone to scratching.

Problems with the conventional technique[2,3]

1. Mechanical grinding and polishing may create an amorphous layer on the surface, called the Beilby layer.
2. The grains on the surface can be deformed during diamond polishing.
3. Colloidal silica polishing is a time-consuming process. In addition, this process can result in embedded residual polishing material on the surface.
4. Electro-polishing is a complex process with variable results.

		Steps						
		1	2	3	4	5	6	7
Consumables	Abrasive (P-[grade])	320 Grit (P-400)	600 Grit (P-1200)	800 Grit (P-2400)	1200 Grit (P-4000)	3 µm	1 µm	0.05 µm
	Type	Silicon carbide	Silicon carbide	Silicon carbide	Silicon carbide	Polycrystalline diamond	Polycrystalline diamond	Colloidal suspension
	Polishing cloth	–	–	–	–	Gold Label	Kempad	Final A
	Coolant	Water	Water	Water	Water	GreenLube	GreenLube	Water*
Settings	Platen speed/ direction	300 rpm/ comp[1]	300 rpm/ comp	300 rpm/ comp	300 rpm/ comp	150 rpm/ comp	150 rpm/ comp	150 rpm/ contra[1]
	Specimen speed	150 rpm	150 rpm	150 rpm	150 rpm	150 rpm	150 rpm	150 rpm
	Force[1]	5 lbf/22 N	5 lbf/22 N	5 lbf/22 N	5 lbf/22 N	5 lbf/22 N	5 lbf/22 N	3 lbf/13 N
	Time[1]	1 minute	1 minute	1 minute	1 minute	10 minutes	10 minutes	30 minutes

Table 1. Grinding and polishing procedure[4].

CUSTOMER NEWS

Focused ion beam (FIB)

A FIB is generally used for the preparation of very small samples. The main advantage of ion beam sample preparation is that valuable diffraction data can be achieved from secondary phases with small volume fractions. Apart from that, the method is less sensitive to the chemical composition of the material[5]. The basic principle of the technique is the sputtering of atoms from the surface. During ion milling, the incident ions transfer energy to the target's atoms through collisions, resulting in the escape of atoms if sufficient energy is achieved. The sputtering rate depends on the material of interest (via surface binding energy) and the incident ions' mass, energy, and direction[5]. This technique can require high-energy Ga ions (30 kV), which can introduce lattice defects/strain within the sample or implant Ga ions into the lattice[6]. As a result, the crystal structure of the sample can be damaged and might produce bad EBSD signals. Lower energy ions can be used, resulting in lower material removal rates.

Broad argon/ion beam

A broad argon ion milling process can be used to prepare the surface and a sample cross-section for EBSD characterization. Furthermore, the challenges observed in the conventional technique can be avoided with this technique. The sample preparation involves milling using a high-energy Ar+ ion gun initially with simultaneous rotation and oscillation of the sample. The sample is fixed on a rotary stage, and beams of heavy ions bombard the rotating sample surface. The artifacts related to grain boundaries are removed. Then a subsequent cleaning with a low-energy Ar+ ion gun is carried out. An iPrep II system schema shows a sample transfer to the home position on an SEM stage attached to the polishing unit (Figure 2). A plot showing the change in image quality (IQ) against Ar ion polishing time is shown in Figure 3. Corresponding inverse pole figure (IPF) maps are also displayed inset. It is clear from the figure that EBSD data quality can be significantly improved through Ar ion polishing.

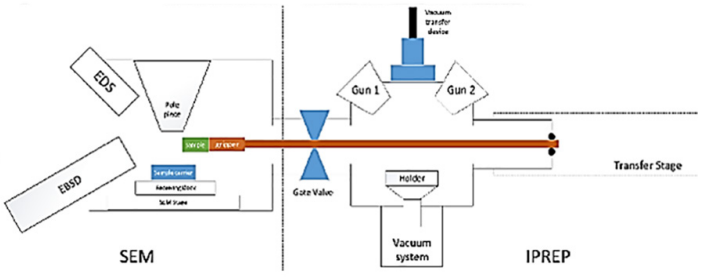

Figure 2. The iPrep II system schematic shows a sample's transfer to the home position on an SEM stage[7].

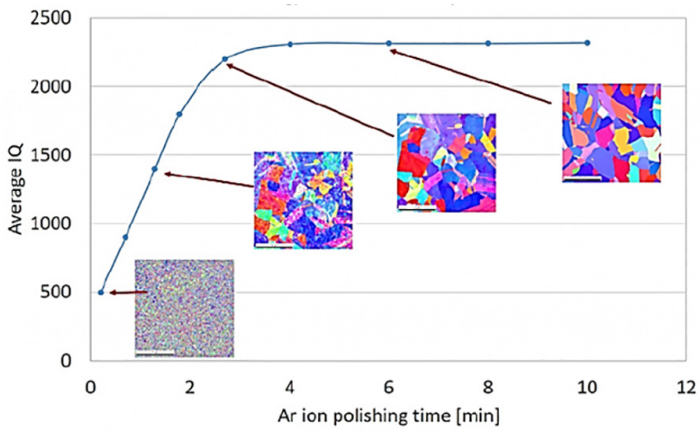

Figure 3. Average IQ in EBSD scan against Ar ion polishing time[3].

The technique is helpful for both SEM and TEM sample preparation operated at much lower energies (~4-6 keV for SEM and 100 V for TEM). For very soft materials, the operating voltage is kept even lower (~1-2 keV)[2]. The chamber is kept at vacuum to avoid any contamination of the ion source.

Advantages of the broad argon beam technique over others

- Several steps (involved in conventional polishing) can be avoided in this advanced method.
- A highly skilled operator is not necessary.
- Allows a wide range of samples that are difficult to prepare through conventional techniques, such as soft or porous materials.
- Permits samples as large as 32 mm in diameter.
- High milling rate.
- Reduced cost as the tool costs around ten times less than a FIB.
- Easy-to-use and effective on surfaces containing oxides, small damages, or irregularities.

Importance of argon

- A noble gas, argon is used in this process, unlike Ga in FIB, which ensures no chemical interaction with the sample.
- The gas is easily ionized after reaching an energy threshold of 15 eV.
- The inclusion of lattice defects (strain) can be avoided using low-energy argon ions.

- When the ions interact with the sample, there is a chance of an amorphous layer forming on the surface due to the movement of the atoms from their actual location. Argon is the best option from the low energy and economic points of view.

The thickness of the amorphous layer depends on the milling voltage and milling angle. The thickness is reduced with lower energy and a decrease in beam angle. The angle is generally kept between 4 – 7 °. Lower angles require a longer milling time (more than 30 minutes), whereas higher angles result in bumpiness on the sample surface[5].

Products

Gatan offers two ion beam polishing products.

1. PECS™ II System – Broad argon ion beam system designed to polish and coat samples for SEM imaging and analytical techniques.
    - Polish, etch, or coat samples with a single pump down.
    - Etch at voltages as low as 100 V for rapid sample preparation.
    - Permit samples as large as 32 mm in diameter.
    - Transfer samples to SEM/FIB chamber without exposure to air (optional).
    - Store and analyze the image in DigitalMicrograph® software.
    - Display and control all parameters using the integrated 10-inch color touch screen.
2. Ilion™ II System – Ideal for low-energy surface preparation for your SEM cross-section viewing.
    - Vacuum load-lock and liquid nitrogen cold stage to provide rapid workflows on beam-sensitive samples.
    - Real-time observation of the polishing process, including an optical microscope with digital imaging.
    - Stored and analyzed with DigitalMicrograph software.
    - Operation via a 10-inch color touch screen.

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