APPLICATION NOTE

# Metallic sample preparation for EBSD by mechanical method and argon ion beam milling

*The preparation of samples for electron backscatter diffraction (EBSD) analysis is similar to the preparation techniques used for ordinary metallographic samples. The quality of the information obtained during analysis is dependent upon absolute removal of all surface defects on the sample due to mechanical preparation steps. The electron beam used during EBSD analysis needs to be at an acute angle (60 to 65°), which makes it necessary for the sample to have superior surface flatness. This can be achieved with little effort using table-top sectioning, grinding, and polishing systems. Subsequent ion milling removes any residual lattice damage on the nanoscale, including oxidation and hydrocarbon contamination. This application note describes how aluminum, brass, and titanium samples were prepared for EBSD analysis using Allied High Tech mechanical preparation products and Fischione Instruments argon ion mill.*

## Mechanical sectioning

As with ordinary metallographic preparation, sectioning produces deformation in the sample. Thus, it is important to minimize deformation by selecting the right blade for the application. Blade selection depends upon the hardness and ductility of the material, as well as the actual size of the sample to be sectioned; only metallographic blades recommended for that specific material should be used.

Crystal structure has an influence on the amount of damage caused by preparation. Because of their tendency to slip more easily, face-centered cubic metals deform more than body-centered cubic metals, during the same preparation process. Also, high-purity metals demand longer polishing times than alloy systems, due to their more precisely ordered structures.

Secure the samples in the quick-slide vise of the Allied High Tech Products' PowerCut 10™ cut-off saw and section using a rubber-bonded silicon carbide blade. The rubber-bonded blade offers durability and long service life. The silicon carbide abrasive particles cut nonferrous metals cooler with less resistance, while the rubber bond is most effective for sectioning softer metals. This blade composition introduces the least amount of metallurgical deformation to these particular samples.

## Sample mounting

Ultrasonically clean the samples in Allied's GP Cleaning Solution, rinse with ethyl alcohol, and dry with compressed air spray to remove any oil or debris introduced during sectioning or handling. This cleaning process improves the adhesion of the mounting material to the sample, which eliminates abrasive particles and debris from collecting in mounting gaps. Any material collected in these gaps will be a source of scratches on the sample surfaces.

Mount the samples in the Allied TechPress 2™ mounting press using a 1-inch mold assembly. Smaller diameter molds are used because of EBSD high tilt requirements. Encapsulate the samples with copper-based conductive mounting powder to prevent charging in the scanning electron microscope (SEM). The dimensions of each mount should be approximately 1.0 inch diameter x 0.50 inch height. The mounting parameters for this material and mounting press are listed in Table 1.

Table 1. TechPress 2™ mounting press parameters.

| Variable | Specification |
|---|---|
| Preheating time | Not used |
| Preheating temperature | Not used |
| Heating time | 6 minutes |
| Heating temperature | 150 °C (300 °F) |
| Cooling time | 4 minutes |
| Pressure | 4 bar |

## Mechanical grinding and polishing

Because manual polishing will likely fail to produce consistent or accurate results, automatic preparation equipment should be used.

Preparing metallurgical samples for EBSD typically requires longer polishing times and careful selections of consumables. Longer

Table 2. MetPrep 3™/PH-3™ grinding and polishing procedure for aluminum, titanium and brass specimens

| | | 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 | Polyscrystalline diamond | Polycrystalline diamond | Colloidal suspension |
| | Polishing cloth | — | — | — | — | Gold Label | DiaMat | Final A |
| | Coolant | Water | Water | Water | Water | GreenLube | GreenLube | Water* |
| Settings | Platen speed/ direction | 300 rpm/ comp† | 300 rpm/ comp | 300 rpm/ comp | 300 rpm/ comp | 150 rpm/ comp | 150 rpm/ comp | 150 rpm/ contra |
| | Specimen speed | 150 rpm | 150 rpm | 150 rpm | 150 rpm | 150 rpm | 150 rpm | 150 rpm |
| | Force‡ | 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 minute | 1 minute | 1 minute | 1 minute | 10 minutes | 10 minutes | 30 minutes |

* Water was only used to presoak and rinse the cloth after use.

† The platen and sample holder rotate in the same direction (comp).

‡ Per 1-inch mount. If the surface area changes, the force has to be be adjusted accordingly.

§ For one mount at a time. If a different number of mounts are prepared, the time may need to be adjusted.

polishing times are necessary to ensure that all deformation is removed from previous steps.

Using the wrong polishing cloth could lead to smearing or excessive relief in the microstructure, which would prevent access to the true underlying structure. Cloths that are flat and woven are recommended. Cloths of this type are less aggressive and therefore minimize sample deformation and maximize sample flatness, which is ideal for EBSD sample preparation. Examples of suitable cloths include nylon, polyester, silk, or polyurethane.

Polishing suspensions and lubricants should be chemically compatible with the sample materials to prevent inordinate relief or staining.

Grinding consumables are typically selected based on the sample hardness and the amount of material to be removed. Silicon carbide abrasives are most commonly used for grinding; however, diamond grinding discs have proven to be effective for harder materials because they produce excellent material removal rates while maintaining flatness.

Cycle times for grinding with silicon carbide abrasives should be kept below two minutes if multiple samples are prepared at once. If silicon carbide discs are used for longer than two minutes, they typically become dull and generate excessive heat that can cause thermal deformation. It may be necessary to repeat a step if not enough material has been removed to flatten the sample

**ALLIED HIGH TECH MECHANICAL PREPARATION PRODUCTS**

From left to right: Allied PowerCut 10™ abrasive cut-off saw, TechPress 2™ mounting press, and MetPrep 3™ grinder/polisher with PH-3™ Power Head.

or if scratches and deformation remain from a previous step.

For softer materials, less force and a more viscous lubricant should be used for grinding and polishing. Harder materials require greater force and a higher volume of diamond suspension to remove an adequate amount of material in the same timed step. Prepare each sample separately using the MetPrep 3™ grinder/polisher with PH-3™ Power Head in individual force mode; use a new polishing cloth for each sample type to prevent cross contamination. The grinding and polishing procedure is outlined in Table 2.

Use a series of silicon carbide abrasive discs during grinding to remove the deformation from previous processing. After each grinding step, use a microscope to inspect the sample surfaces and ensure that the scratch pattern is uniform.

To remove the deformation caused by grinding, use 3-µm diamond suspension on Gold Label polishing cloth and 1 µm diamond suspension on Kempad polishing cloth with GreenLube. Gold Label is a durable low-nap woven cloth that produces an excellent finish, material removal rate, and flatness; Kempad cloth is a non-woven, very low-nap textile that provides good removal and flatness on a wide variety of materials. GreenLube is a general purpose polishing lubricant used to reduce frictional heat and extend cloth life.

Perform final polishing with 0.05 µm colloidal silica suspension on a Final A polishing cloth, which is a non-woven, low-nap porous polyurethane cloth excellent for final polishing of a wide variety of materials. The alkaline properties of colloidal silica suspension are ideal as a final polishing solution for most materials. The AD-5™ fluid dispenser was used to provide automatic dosing of the polishing suspensions and lubricant, allowing for easy, repeatable and more consistent sample preparation. The dispensing parameters are listed in Table 3.

Table 3. AD-5™ fluid dispenser parameters

| | Steps | |
|---|---|---|
| | 5 and 6 | 7 |
| Type of lubricant | Diamond suspension and GreenLube | Colloidal silica suspension |
| Pulses per minute | 6 | 8 |
| Pulse length | 1.0 | 1.0 |
| Water flush | Off | On |

### Periodic sample cleaning

After each grinding and polishing step, clean the sample and fixture with micro-organic soap to remove debris and abrasive particulates. Dry the sample with compressed air spray; this reduces the likelihood of scratches on the sample surface due to cloth contamination.

## Ion milling after mechanical preparation

Ion milling is an ideal method for producing samples for SEM imaging and analysis. The use of incident argon ions is standard because the gas is easily ionized after reaching an energy threshold of 15 eV.

**FISCHIONE INSTRUMENTS MODEL 1060 SEM MILL**

Premium Edition shown.

The resulting ions are chemically inert, which is critical for the materials scientist. Penetration by argon into the lattice of most engineered materials is only a few nanometers at accelerating voltages less than 6 kV. This depth decreases linearly with decreasing voltage and incident angle; below 1 kV and 10˚, the residual damage layer (amorphization) is less than 1 nm. As argon ions enter and exit an ion-milled surface, surface ions are ejected locally (sputtered). Relaxation of the surrounding crystalline lattice occurs for metals and ceramics with a net change in lattice spacing in the vicinity of the sputtered ion.

For EBSD, the sample is the subject of a diffraction experiment and must become a mirror for electrons. The ideal sample must have a surface that is smooth and planar on the atomic scale with no significant subsurface damage or amorphization. By carefully tailoring the ion milling recipe to the particular material, such quality can be approached.

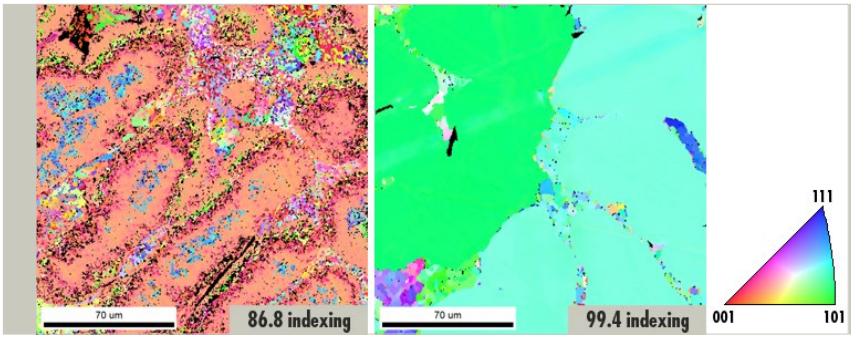
After final cleaning, attach the mounted sample to an aluminum pin-type stub using double-stick carbon tape. Alternatively, create a more permanent bond using an adhesive, such as Crystalbond™ Type 509. Fill the Fischione Model 1060 SEM Mill's built-in dewar (optional) with liquid nitrogen to provide a cleaner, drier environment in the ion mill vacuum chamber.

Insert the stub into the sample stage of the SEM Mill and close the load lock. Selecting the optimal single or multi-step process for ion milling depends on the alloy you are processing. Lowering the ion energy (from 6 to 0.1 kV) and milling angle reduces the risk of a residual amorphous damage layer. Lower angles also promote planarization of alloys that have more than one phase.

Table 4. SEM Mill suggested ion milling parameters

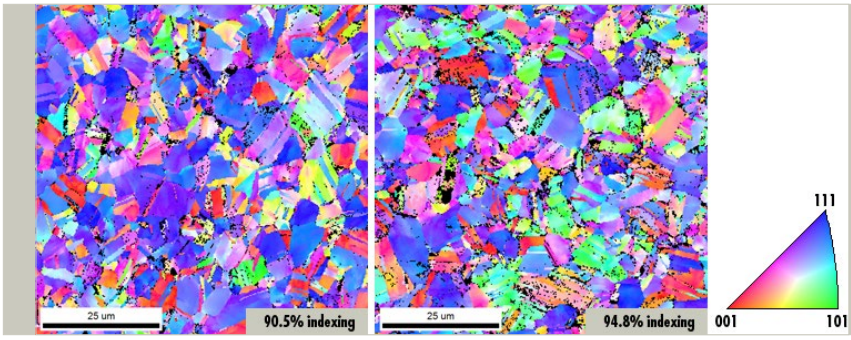
| | |
|---|---|
| Number of ion sources | One |
| Accelerating voltage | 4 kV |
| Ion beam focus | 45% (~2 mm ion beam diameter) |
| Milling angle | 5° |
| Specimen stage motion | 360° rotation at 5 rpm |
| Ion milling time | Aluminum and titanium: 30 minutes<br>Brass: 45 minutes |

The sample height is detected by a laser in the main vacuum chamber, which allows the ion sources to be tilted in an eucentric manner.



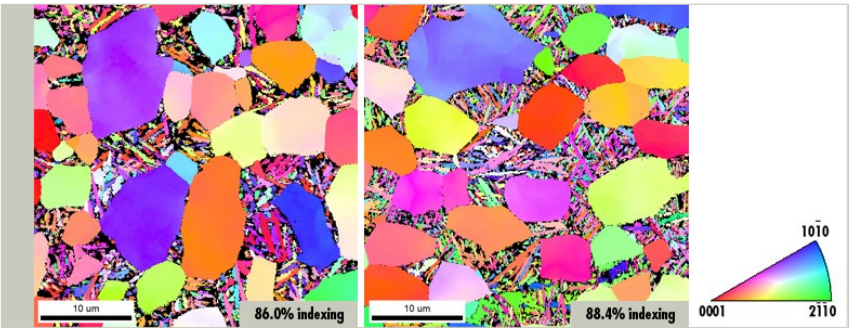

**ALUMINUM ORIENTATION MAPS**

Mechanical preparation only (left) and mechanical preparation followed by ion milling (right).




**BRASS ORIENTATION MAPS**

Mechanical preparation only (left) and mechanical preparation followed by ion milling (right).




**TITANIUM ORIENTATION MAPS**

Mechanical preparation only (left) and mechanical preparation followed by ion milling (right).

Because the EBSD signal will originate from a depth of 10 to 15 nm within the sample, it is critical that the crystallinity of this region be as free from lattice distortion as possible.

Residual oxidation or hydrocarbon contamination is minimal as a byproduct of ion milling. To avoid contamination following ion milling, the sample should be transferred from the SEM Mill load lock to the SEM immediately after ion milling is complete. However, samples may be stored for a short time under dry vacuum or an inert atmosphere.

## EBSD analysis

Following the mechanical and ion beam sample preparation methods described above, orientation maps for aluminum, brass, and titanium were obtained. In each instance, an increase in the indexing rate was observed when the mechanical preparation was followed by ion milling. Both the planarity and quality of the crystalline material analyzed by EBSD is enhanced.

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