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Metallographic Preparation for Electron Backscattered Diffraction

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Electron backscattered diffraction (EBSD) is performed with the scanning electron microscope (SEM) to provide a wide range of analytical data; e.g., crystallographic orientation studies, phase identification and grain size measurements. A diffraction pattern can be obtained in less than a second, but image quality is improved by utilizing a longer scan time. Grain mapping requires development of diffraction patterns at each pixel in the field and is a much slower process. The quality of the diffraction pattern, which influences the confidence of the indexing of the diffraction pattern, depends upon removal of damage in the lattice due to specimen preparation. It has been claimed that removal of this damage can only be obtained using electrolytic polishing. However, the use of modern mechanical preparation methods, equipment and consumables does yield excellent quality diffraction patterns without use of dangerous electrolytes and the problems associated with electropolishing. Basically, if mechanical preparation results in quality polarized light images of non-cubic crystal structure elements and alloys (e.g., Sb, Be, Hf, α-Ti, Zn, Zr), or color tint etching of cubic, or non-cubic crystal structure elements or alloys produces high-quality color images, then the surface is free of harmful residual preparation damage and EBSD patterns with high pattern quality indexes will be obtained.

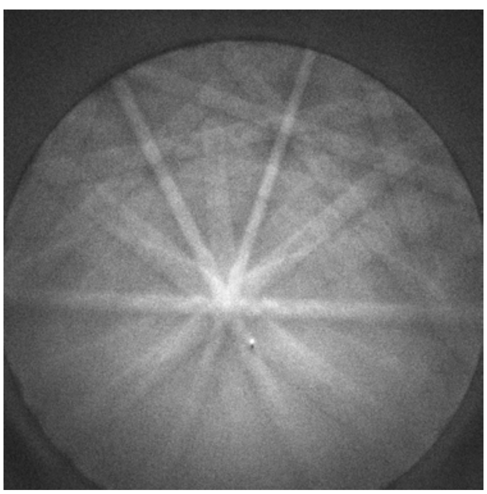
Polarized light image quality is dependent upon the elimination of preparation damage and upon the quality of the microscope optics. Consequently, always check the polarized light response of these metals (mainly hcp), to verify preparation quality before performing EBSD. For cubic metals, etch first with a general-purpose reagent to confirm the nature of the expected microstructure. Then, repeat the final polishing step and use a color tint etch to verify freedom from damage. EBSD is best performed with an as-polished, non-etched specimen due to the steep angle to the electron beam, as surface roughness can degrade the diffraction pattern. In most cases, a well-prepared, un-etched specimen will exhibit good secondary or backscattered image quality.

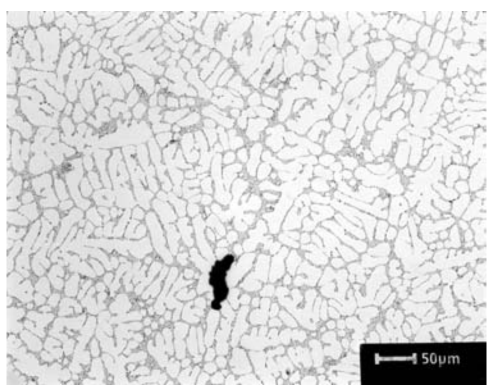
Specimen preparation methods for these metals have been developed that yield excellent results using straightforward methods that generally require less than about twenty-five minutes. Automated preparation equipment is recommended, as the methods will be performed accurately and reproducibly. Manual preparation cannot produce flatness, phase retention and damage removal as easily as automated processing and is less reproducible.

Successful preparation requires that sectioning be performed with equipment and consumables that minimize damage. Sectioning is a violent process and it can introduce massive damage. Crystal structure does influence damage depth. Use only abrasive blades designed for metallography and are recommended for the specific metal/alloy in question. A precision saw yields even less damage as the blades are much thinner and the applied loads are much lower. Cutting with machines and blades/wheels that introduce minimal damage is the most critical step in generating damage-free metallographic surfaces. This fact cannot be over-emphasized. Commence grinding with the finest possible abrasive and surface that will make all of the specimens in the holder co-planar and remove the sectioning damage in reasonable time. This is the second critical rule for obtaining damage-free polished surfaces. The proposed methods utilize flat, woven cloths or pads that minimize relief problems. To minimize damage, use less aggressive surfaces, such as silk, nylon, polyester or polyurethane. The specimen preparation method must remove all scratches. If scratches are present, so to is damage below the scratch. Scratch depths produced in grinding and polishing are not uniform. A deep scratch will have deformation below it. The preparation method must remove the scratches and the underlying damage in order to obtain high quality the EBSD patterns.

The following example shows Al – 7.12% Si prepared mechanically using five steps. The EBSD pattern shown was developed using a Philips (FEI) XL 30 SEM using a tungsten filament at 20 kV, spot size 5 (beam 140 nm diameter), 20 mm working distance and an emission current of 73 µA (~ 0.84 nA on the specimen). The plane-of-polish was oriented 74° from horizontal.

Surface	Abrasive/Size	Load Lb. (N)	Speed rpm/Direction	Time (minutes)
CarbiMet® abrasive discs	220-320 (P240-P400) grit SiC, water cooled	5 (22N)	240-300 Contra	Until Plane
Ultra-Pol® or TriDent® cloths	9-µm MetaDi Supreme® suspension	5 (22N)	120-150 Contra	5
TriDent® or TexMet® 1500 cloths	3-µm MetaDi Supreme suspension	5 (22N)	120-150 Contra	4
TriDent or TexMet® 1500 cloths	1-µm MetaDi paste	5 (22N)	120-150 Contra	3
MicroCloth®, VelTex® or ChemoMet® I cloths	MasterMet® colloidal silica	5 (22N) (7 lb/31N with ChemoMet I)	120-150 Contra	3





Left: EBSD pattern for α-Al in as-cast Al – 7.12% Si – pattern quality index: 87 ± 4.2.
Right: Light micrograph of as-cast Al-7.12% hypoeutectic alloy etched with 0.5% HF in water.

The average atomic weight of an Al – 7.12% Si alloy is quite low and this makes it a difficult subject for EBSD. But, as can be seen, even in the etched condition, an excellent quality diffraction pattern was obtained of the alpha dendrites. This demonstrates that mechanical polishing is capable of producing high quality EBSD patterns when properly performed.

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