Preparing Magnesium Alloys for Electron Backscatter Diffraction

Dr. Tracy Berman

University of Michigan, Ann Arbor, MI 48109, USA

photos encouraged

Overview

Electron Backscatter Diffraction (EBSD) is a powerful characterization tool that is employed to study grain size, grain morphology, crystallographic texture and other important microstructure parameters. Its relatively low atomic number and propensity to corrode can make it difficult to get high quality EBSD patterns from magnesium specimens. This work shares a preparation method that has been used successfully by the author and colleagues for many years. In addition, suggestions for EBSD data collection and analysis are offered.

Grinding and Polishing

- 15 N pressure (light)
- 150 RPM base and 60 RPM head, complementary rotation
- I use Buehler MetaDi II diamond pastes on Buehler Chemomet pads with the Buehler MetaDi® fluid as lubricant
- Flush all polishing pads with ethanol before & after use
- Store polishing pads in a clean environment

Step	Description	Time
1	320 grit SiC w/ water	Until planar, replace paper every minute
2	400 grit SiC w/ water	30 seconds*
3	600 grit SiC w/ water	30 seconds*
4	800 grit SiC w/ water	30 seconds*
5	1200 grit SiC w/water	30 seconds*
6	6 µm paste w/ MetaDi fluid	10 minutes
7	3 µm paste w/ MetaDi fluid	10 minutes
8	1 µm paste w/ MetaDi fluid	10 minutes

*If surface is not uniform, replace SiC paper and repeat step

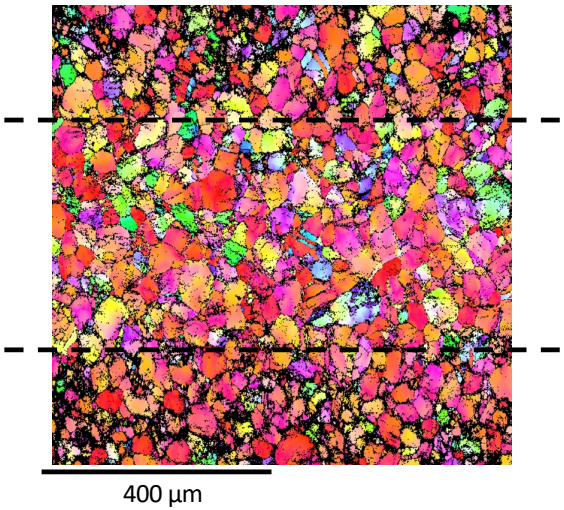
For thin specimen, such as sheet, hand polishing may be preferable. Limited material may force you to accept some scratches.

Etching

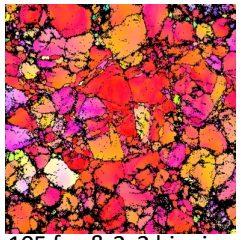
- Etch by submerging for 3-5 seconds in ~5ºC solution of
  - 60 mL ethanol
  - 20 mL water
  - 15 mL glacial acetic acid
  - 3 mL nitric acid
- Rinse with ethanol, ultrasonicate in ethanol, dry well

EBSD Settings and Scan Conditions

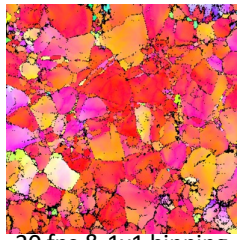
- Dependent on information desired and condition of material – multiple scans may be needed
- 30keV and a high beam intensity/current provide more signal (but limit resolution)
- Samples with deformation may require smaller scan areas and longer camera exposures
- **Run a quick test scan (use large step size) to make sure scan area and settings yield quality information!**



IPF map of deformed Mg-3Zn-0.1Ca alloy. Defocusing results in poor quality data at the top and bottom of the scan.. Specimen is tilted in vertical direction.



105 fps & 2x2 binning

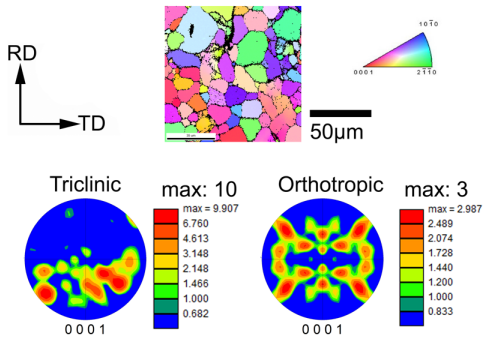


30 fps & 1x1 binning

Slow down for publication ready images. Speed up for quick analysis.

Measuring Texture Using EBSD

- Focus on sampling a large number of grains for a representative texture – okay to use a large step size
- No need to "clean" data
- **Beware forcing a sample symmetry when calculating texture!**



Sampling a small number of grains and forcing sample symmetry results in unrealistic textures.

Acknowledgements

Polishing routine is adapted from one provided by Bob Kubic at General Motors Research and Development

This material is based upon work supported by the Department of Energy, Office of Energy Efficiency and Renewable Energy (EERE), under Cooperative Agreement Award Number DE-EE007756 with the United States Council for Automotive Research's consortium The United States Automotive Materials Partnership (USAMP LLC).