Experiment Brief

PECS II Broad Beam Ion Mill and Velocity Super EBSD Analysis System

Title

Removing the oxide layer from magnesium alloys to allow successful EBSD data collection

Instruments Used

Gatan PECS™ II and EDAX Velocity™ Super EBSD Analysis System with APEX™ 2.0 Software

Background

To obtain valid electron backscatter diffraction (EBSD) orientation measurements, the true structure of a specimen must be undistorted and continuous to the surface. Routinely, mechanical or electropolishing is applied to prepare a specimen surface for analysis. However, on certain materials like magnesium or zinc alloys that are extremely susceptible to oxidation, an oxide layer may quickly form when the sample comes into contact with water. This may happen during polishing or afterward by exposure to water vapor in the air during transport or storage. Such an oxide layer effectively blocks the observation of EBSD patterns and has to be removed before analysis.

Materials and Methods

A thick oxide layer formed on a mechanically polished Mg sample that had been in storage for multiple years, and no EBSD patterns could be obtained. To remove this oxide while avoiding contact with water, the sample was mounted in the PECS II and milled with 5 kV Ar ions at an incident angle of 4˚ for 30 minutes. The duration was selected to ensure that enough material was removed from the surface to expose the native material fully. After milling, the oxide layer was removed over a multiple-mm^2 sample area before it was analyzed using EBSD.

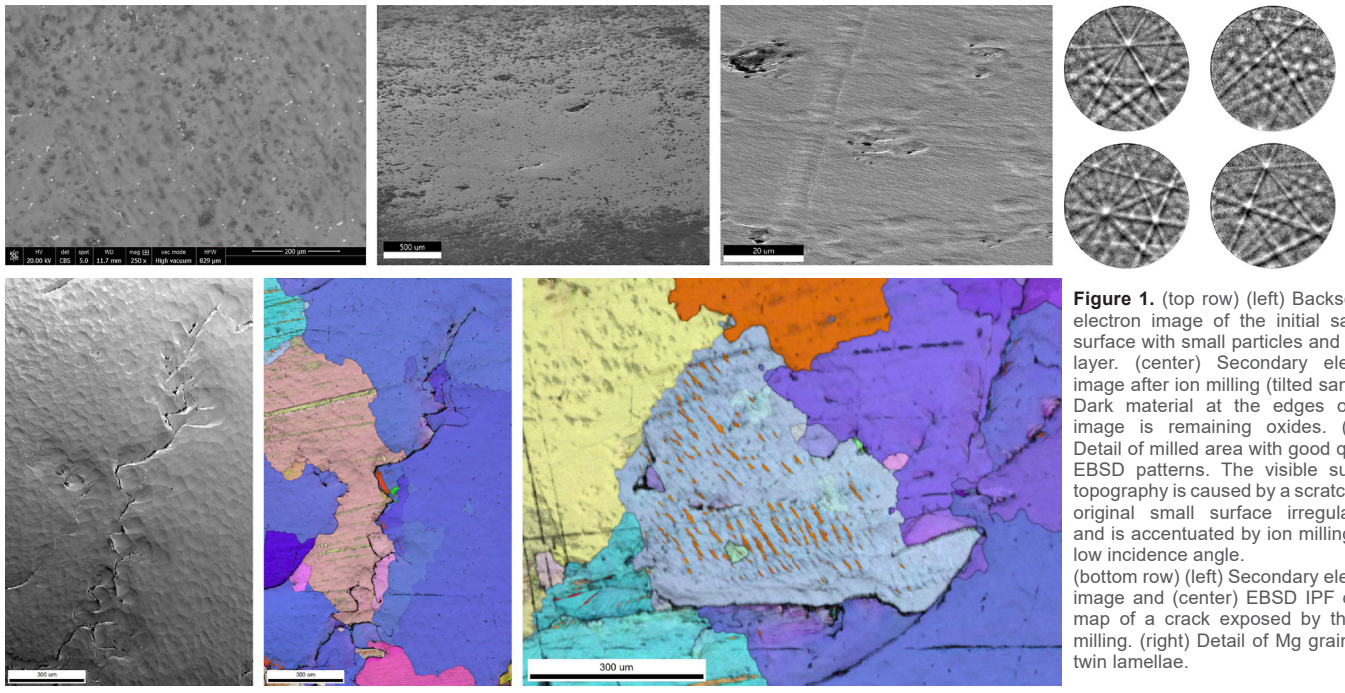

Figure 1. (top row) (left) Backscatter electron image of the initial sample surface with small particles and oxide layer. (center) Secondary electron image after ion milling (tilted sample). Dark material at the edges of the image is remaining oxides. (right) Detail of milled area with good quality EBSD patterns. The visible surface topography is caused by a scratch and original small surface irregularities and is accentuated by ion milling at a low incidence angle.
(bottom row) (left) Secondary electron image and (center) EBSD IPF on IQ map of a crack exposed by the ion milling. (right) Detail of Mg grain with twin lamellae.

Summary

By applying broad beam ion milling, you can remove the oxide layer that quickly forms on Mg alloys without introducing additional mechanical strain. The resulting specimen produces good quality EBSD patterns, and the microstructure can be successfully analyzed.

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