

Experiment Brief

PECS II Broad Beam Ion Mill and Pegasus Analysis System with Octane Elect and Velocity Super

Title

Optimizing the surface of multiphase Al alloys for successful EBSD analysis

Instruments Used

Gatan PECS™ II and EDAX Pegasus Analysis System with Octane Elect, Velocity™ Super, and APEX™ 2.0 Software

Background

To obtain valid EBSD orientation measurements, the true structure of a specimen must be undistorted and continuous to the surface. Any surface contamination and damage introduced during specimen preparation needs to be minimized. On soft materials, such as aluminum, it is very difficult to prepare the sample by mechanical polishing without introducing a thin distorted surface layer that obscures the real microstructure. When secondary phases with different polishing responses are present, simultaneously getting a suitable specimen surface for all phases is even more challenging. Broad beam ion milling provides a way to remove the distorted layer from the entire surface without risking additional damage by mechanical contact.

Materials and Methods

After routine mechanical polishing, a multiphase Al alloy sample with Si, Cu, Fe, and Sn containing phases is analyzed by EBSD mapping. Orientation data could be obtained from most areas. However, some grains show preparation artifacts, while certain intermetallic phases do not produce indexable patterns at all (Figure 1, top row). After analysis, the sample was mounted in the PECS II and milled using 4 kV Ar ions at a low incident angle of 3° for 20 min. The low angle was selected to remove the surface topography that had formed around the secondary phase particles. The resulting surface was smooth, and now EBSD patterns could be collected from all phases for complete surface analysis (Figure 1, bottom row).

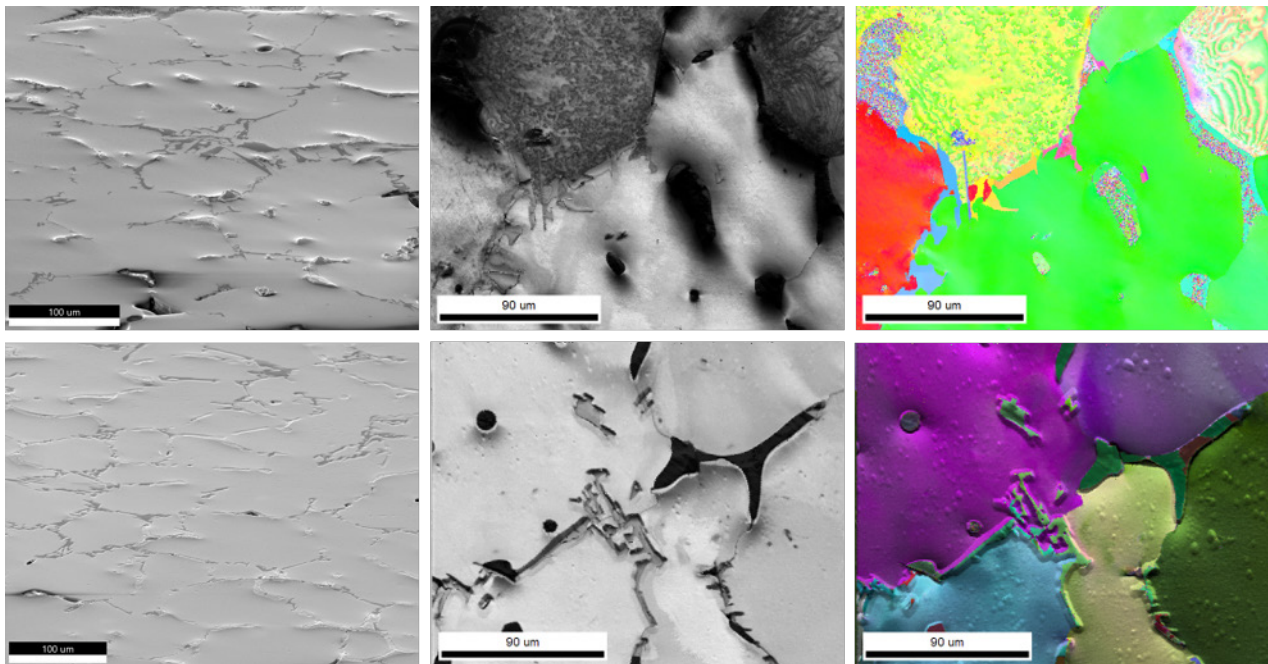


Figure 1. (top row) (left) Secondary electron image of polyphase Al alloy after mechanical polishing (tilted sample). Some phases did not polish well and stand out from the surface. (center) EBSD image quality (IQ) map illustrating that no diffraction patterns could be collected from the protruding grains (black areas). Two grains at the top show an intricate substructure, which is an artifact caused by mechanical polishing. This substructure is also apparent in the inverse pole figure (IPF) map (right). (bottom row) (left) Secondary electron image after ion milling (tilted sample). Most topography has been removed, and all grains now produce indexable EBSD patterns. The artificial substructures have been removed and the true grain structure is displayed in the IQ and IPF maps (center, right).

Summary

By applying broad beam ion milling, Al specimens containing multiple phases with varying polishing responses can be successfully prepared for EBSD analysis. The ion milling ensures a clean and damage-free sample surface and optimizes the electron diffraction signal from all phases, regardless of their polishing resistance.

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