

Microstructural Characterization of Mg-Al-Ca Alloys Using Ion Milling Surface Preparation Technique

Shirin Kaboli, Hendrix Demers, Nicolas Brodusch and Raynald Gauvin

Department of Mining and Materials Engineering, McGill University, Montréal, Canada.

The microstructure of polycrystalline materials changes heterogeneously during deformation. Type and number of activated slip (or twinning) systems, softening processes and phase transformation during deformation control the microstructure evolution [1]. Depending on deformation conditions and type of material, the deformed microstructure may consist of dislocation tangles (or walls), cells, subgrains, deformation bands, kink bands and shear bands [2]. Observation and characterization of these microstructural features are necessary to understand the deformation behavior of materials. Previous investigations mainly involved chemical or electrochemical polishing techniques for metallographic surface preparation. In addition, deformation substructures were observed with polarized light microscopy and transmission electron microscopy [2]. Alternatively, ion milling surface preparation method and electron channeling contrast imaging in a field emission scanning electron microscope (FE-SEM) can reveal microscopic details of a deformed material. In this study, ion milling was applied on Mg-0.3Al-0.2Ca alloy. Dynamic recrystallization and second-phase precipitation occur during hot deformation [3]. Using secondary electron (SE) and backscattered electron (BSE) detectors and different milling parameters, a variety of microstructural features were observed.

Uniaxial hot-compression test was conducted on Mg-0.3Al-0.2Ca alloy using a 100 kN MTS servo-hydraulic testing machine at 400 °C and 0.01 s⁻¹ strain rate. Specimen surface preparation was carried out using a Hitachi IM-3000 flat ion milling system. Figure 1 shows the SE and BSE micrographs of deformed specimens obtained with a Hitachi SU-8000 FE-SEM. Ion milling at low incidence angle revealed grain boundary structures in SE micrographs. Serrations at original grain boundaries, low angle grain boundaries inside original grains and subgrains were observed in a low strain specimen (Fig. 1a). Dynamic recrystallized grains were also observed at original grain boundaries and inside grains in a high strain specimen (Fig. 1b). Ion milling at intermediate incidence angle displayed the deformed microstructure and dynamically formed precipitates in high magnification BSE micrographs. Round and elongated precipitates were detected on transition bands (Fig. 1c). Inside original grains, elongated precipitates were preferably formed on parallel slip planes as shown with arrows (Fig. 1d). Ion milling at high incidence angle showed complex microstructural heterogeneities inside original grains in BSE micrographs. Original large grains were divided with kink banding in high strain specimens. Each kink band was subdivided with several slip bands of alternative bright and dark contrast. In addition, kink bands were separated with long narrow transition bands. Subgrains with low angle grain boundaries were detected inside the transition bands (Fig. 1e). Cell blocks separated with dense dislocation walls were also detected inside a few original grains (Fig. 1f).

Ion milling surface preparation technique was beneficial to study deformed microstructure of Mg alloys. Milling at low incidence angles showed low angle grain boundaries and subgrains with low angle misorientation. Milling at high incidence angle revealed substructures inside grains such as kink bands and dislocation walls. Milling at intermediate incidence angles exhibited precipitates along with deformed microstructure in BSE micrographs.

References:

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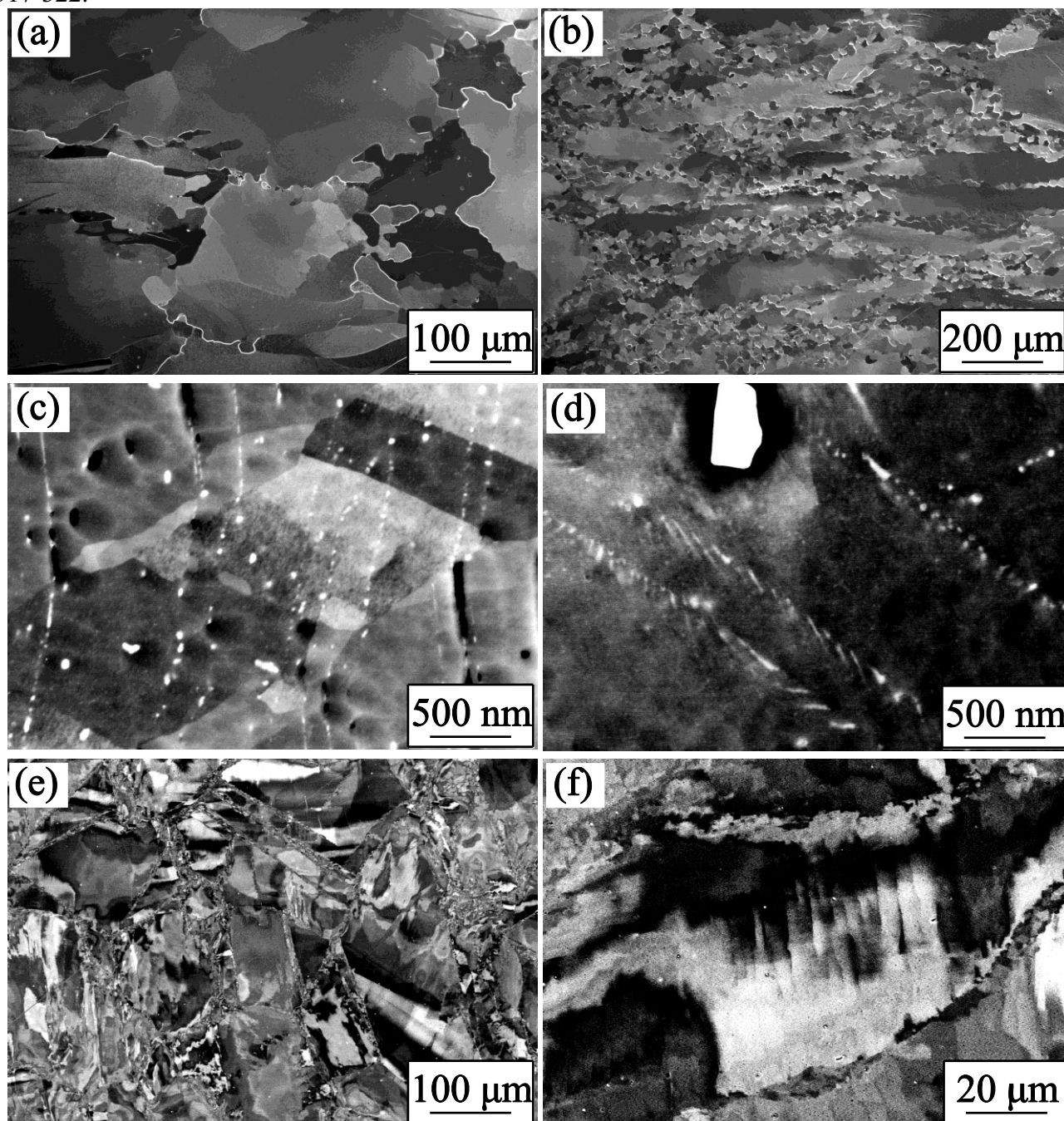


Figure 1. Mg-0.3Al-0.2Ca alloy microstructure after uniaxial hot-compression tests. (a,b) Secondary electron micrograph at low incidence angle milling, (c,d) secondary electron micrograph at intermediate incidence angle milling and (e,f) backscattered micrograph at high incidence angle milling. Subgrains, kink bands and dislocation walls were observed at different milling conditions.