Multiscale, Multimodal Characterization of Recrystallized and Non-recrystallized Grains During Recrystallization in a Hot-Compressed Mg–3.2Zn–0.1Ca wt.% Alloy

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Abstract

High-strength, lightweight magnesium (Mg) alloys have substantial potential for reducing the weight of automobiles and other transportation systems and thus for improving fuel economy and reducing emissions. However, the strong crystallographic texture of rolled Mg sheet leads to poor formability and anisotropy. In specific non-rare earth Mg alloys, annealing can be used to desirably weaken the texture. Here, we present a multiscale in-situ study on the recovery and recrystallization of an 80% hot-compressed Mg–3.2Zn–0.1Ca wt% (ZX30) alloy using high-resolution 3D X-ray diffraction microscopy (HR-3DXRD) and dark field X-ray microscopy (DFXM). We track more than 8000 non-recrystallized grains during annealing. Relative changes in crystallographic orientation and volume of each recrystallized and non-recrystallized grain are measured as a function of annealing time. Finally, local strain and orientation are measured in the interior of the specific grains with a spatial resolution of 77 nm.

Keywords

Magnesium • Characterization • X-ray diffraction

Introduction

This work discusses the use of synchrotron-based X-ray diffraction microscopy techniques to investigate the recovery and recrystallization of an 80% hot-compressed Mg–3.2Zn–0.1Ca wt.% (ZX30) alloy in situ, in 3D, and across several orders of magnitude in length scale. High-strength lightweight magnesium (Mg) alloys have substantial potential for reducing the weight of automobiles and other transportation systems to improve fuel economy and reduce greenhouse gas emissions [1]. A major barrier, however, is the strong crystallographic texture of rolled Mg alloy sheet which can lead to anisotropy, brittleness, and poor machinability. There are several challenges to characterizing the microstructure evolution associated with recovery, recrystallization, and grain growth. Because the microstructure is constantly evolving, it is often necessary to characterize the microstructure evolution in situ. However, the relevant length scale (e.g., grain/subgrain size) and the degree of deformation vary significantly in the as-deformed versus recrystallized states, making it difficult to resolve the microstructure and its key features with a single technique. Temporal resolution is an inherent challenge to diffraction techniques that require a full 360° rotation because of (especially scintillation-based) detector readout times. Finally, the ability to apply these established techniques to highly deformed materials is not quite achievable yet, in part due to spatial resolution limitations and in part due to the necessary peak-finding and peak-fitting procedures.

To address these challenges, here, we use a multiscale, multimodal approach by combining two X-ray diffraction techniques: high-resolution X-ray diffraction (HR-XRD) statistical analysis [2] and dark field X-ray microscopy (DFXM) [3, 4]. For HR-XRD, we used a high-resolution detector to zoom into a subset of Bragg reflections appearing in one particular Debye–Scherrer ring. Using HR-XRD statistical analysis, we track more than 8000 sub-surface non-recrystallized and recrystallized grains during the

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S. Barela et al. (eds.), *Magnesium Technology 2023*, The Minerals, Metals & Materials Series,
https://doi.org/10.1007/978-3-031-22645-8_9

recovery and recrystallization processes during in-situ annealing. At several points during the annealing process, we "zoom in" to individual recrystallized and non-recrystallized grains using DFXM. The results show small intragranular variations in elastic lattice strain and misorientation with a spatial resolution of ~100 nm. This combination of HR-XRD statistical analysis and DFXM enables a multiscale, multimodal diffraction microstructure imaging that is particularly useful for small and/or highly deformed grains that are often difficult to resolve using more standard 3D X-ray diffraction techniques.

Experimental

An 80% hot-compressed ZX30 alloy was used in this study. The deformed sample was machined into $1 \times 1 \times 5$ mm^3 rectangular bars using electron discharge machining. Both HR-XRD and DFXM characterization techniques were performed during a single experiment on ID06-HXM [5] at the European Synchrotron Radiation Facility using an X-ray energy of 17 keV and a beam size of 200 µm × 200 µm^2. We annealed the sample for a total of 514 min, during which time we collected 33 h-XRD measurements. The sample was continuously heated at a rate of 10 °C/min for the first 129 min. Once the sample temperature reached 266 °C at 129 min, the sample temperature was maintained at 266 °C degrees for an additional 385 min (with a total heating time of 514 min). To calibrate the sample temperature, we used the thermal expansion of the {101} Debye–Scherrer ring. The multimodal, multiscale X-ray diffraction microstructure imaging using far-field high-energy diffraction microscopy (ff-HEDM), HR-XRD, and DFXM are shown in Fig. 1.

Results and Discussion

Results are shown in Fig. 2. The results of the HR-XRD statistical analysis are presented in Fig. 2. Figure 2a shows five of the 33 h-XRD statistical analysis measurements (summed over the 6° sample rotation) in order of increasing annealing time and temperature. In the first frame, 1,326 grains exist within our field of view. As annealing proceeds, the number of grains can be seen decreasing, and select grains become very large, as can be observed by the high relative intensity of their Bragg reflections. Toward the end of annealing, there are only 153 grains remain. Two additional sequences are provided in Fig. 2, where each measured Bragg reflection is represented as a circle. The location of each circle corresponds to the location of the Bragg reflection, the size of each circle corresponds to the relative volume of the grain, and the color of each circle corresponds to the relative grain volume in Fig. 2b, and ω (i.e., orientation) position in Fig. 2c. These statistical results show that only seven non-recrystallized grains exist out of 153 total grains at the end of annealing, these seven non-recrystallized grains account for 34% of the total volume [7]. These statistical and other results show the power of these "zoom in" and "zoom out" techniques for tracking microstructure evolution in situ from the as-deformed state during annealing.

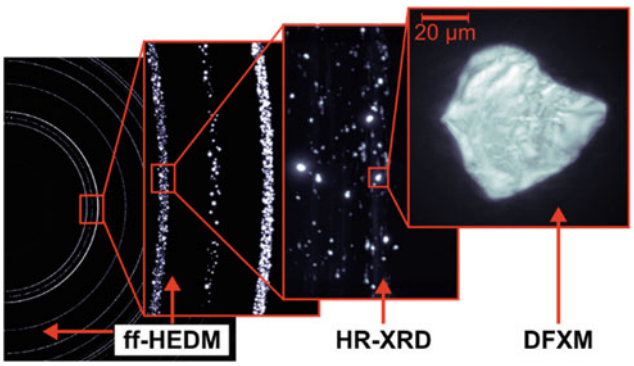

Fig. 1 Multimodal, multiscale X-ray diffraction microstructure imaging using ff-HEDM, HR-XRD, and DFXM. The present work includes only the HR-XRD and DFXM results [7]. The ff-HEDM results are published in [6]

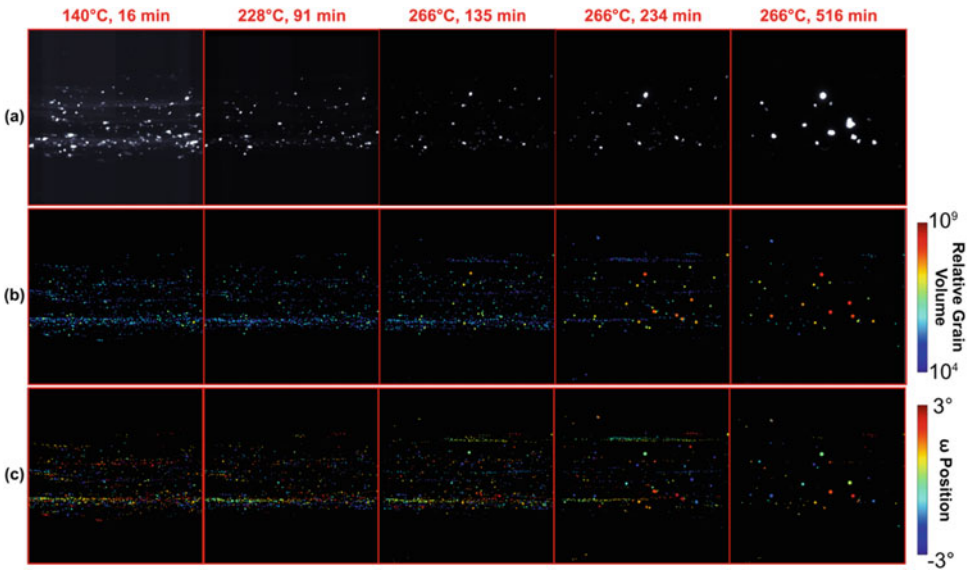

Fig. 2 HR-XRD statistical analysis results **a** raw detector images, **b** relative grain volume, **c** ω position [7]

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