ESRF	**Experiment title:** Studying the Roles of Local Stresses and Solute Concentration on Recrystallization Texture in Magnesium Alloys	**Experiment number**: MA-4480
Beamline: ID06	**Date of experiment**: from: 07 December 2020 to: 14 December 2020	**Date of report**: 01 July 2021
Shifts: 15	**Local contact(s)**: Can Yildirim, Carsten Detlefs	*Received at ESRF:*

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Report:

Motivation: High-strength, lightweight magnesium (Mg) alloys weigh one-third that of aluminum. For this reason, they have substantial potential for reducing the weight and fuel consumption of transportation vehicles, which currently account for ~25% of energy related greenhouse gas emissions worldwide [1]. However, the low formability of Mg alloy sheet is due to its strong basal texture has precluded its widespread use in the automotive and aviation sectors. Recently, research at the University of Michigan and elsewhere has shown that the addition of Ca and rare earth elements can result in a desired weaker texture after a post-process annealing treatment [2,3]. Despite numerous studies, the mechanisms by which this texture reduction occurs remains unknown, and it is likely that several different mechanisms occur simultaneously or sequentially.

Experiment Overview: In this experiment, a Mg-3Zn-0.1Ca alloy was deformed under hot plane-strain compression (to simulate hot rolling) and samples were transported to ID06 at the ESRF, as well as to ID3A at the Cornell High Energy Synchrotron Source (CHESS). In-situ far-field high-energy diffraction microscopy (ff-HEDM) characterization was performed at CHESS, and in-situ intermediate-field HEDM (if-HEDM) and dark-field X-ray microscopy (DFXM) was performed on ID06. By combining the different modalities, we were able to characterize the microstructure evolution during annealing on different length scales, from the subgrain morphology of individual grains (using DFXM) to the aggregate behavior of several thousands of grains (using ff-HEDM). **Fig. 1** shows

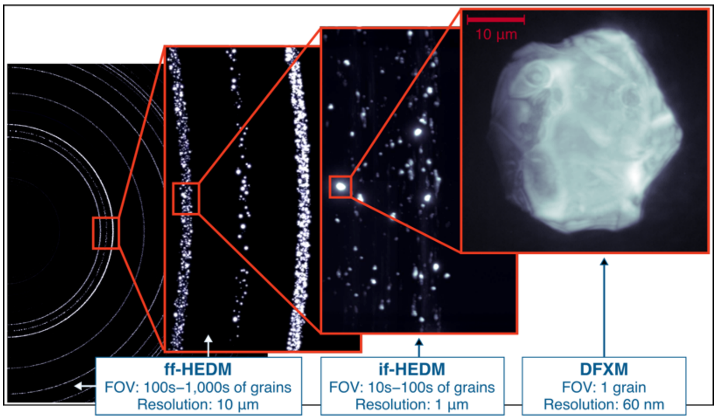

Fig. 1. Multiscale data collected using in-situ ff-HEDM (CHESS), if-HEDM (ESRF), and DFXM (ESRF), illustrating the different fields of view (FOV) and spatial resolutions from combining the different modalities.

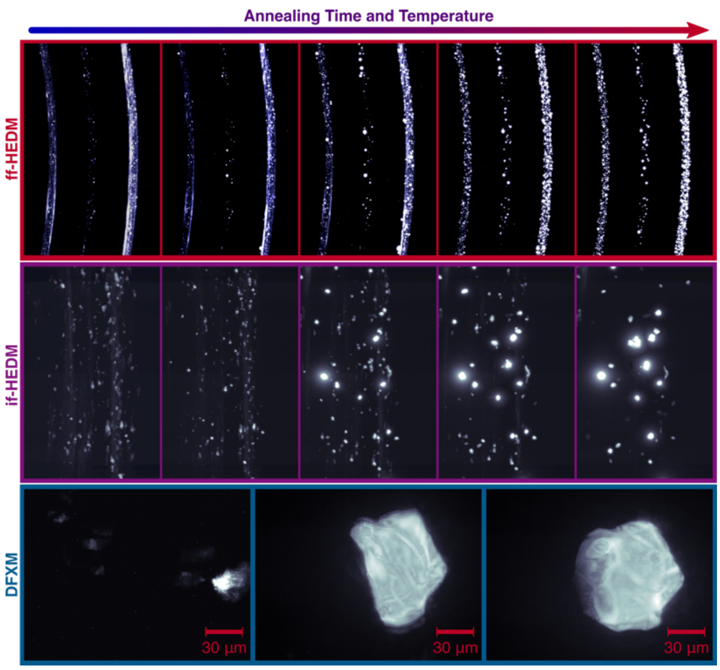

Fig. 2. The evolution of the data from Fig. 1 during the in-situ annealing experiments.

the different length scales represented in the raw data, and **Fig. 2** shows the evolution of this data during annealing.

<u>CHESS Results:</u> By supplementing the ESRF measurements with ff-HEDM measurements at CHESS, we were able to characterize the texture changes during annealing across the entire sample and provide context for the (smaller field of view) ESRF measurements. However, ff-HEDM *cannot* be used to analyze highly deformed microstructures. As a result, the ff-HEDM measurements only included the emergence of low-deformation grains without differentiating whether these are true recrystallized grains or grains that existed in the initial (highly deformed) microstructure that "survived" the annealing process and recovered and grew (**Fig. 3**). For this reason, the higher resolution if-HEDM and DFXM measurements performed on ID06 at the ESRF were critical for correctly interpreting and understanding this data—in particular, the role of the initial as-deformed microstructure, discussed in the next section.

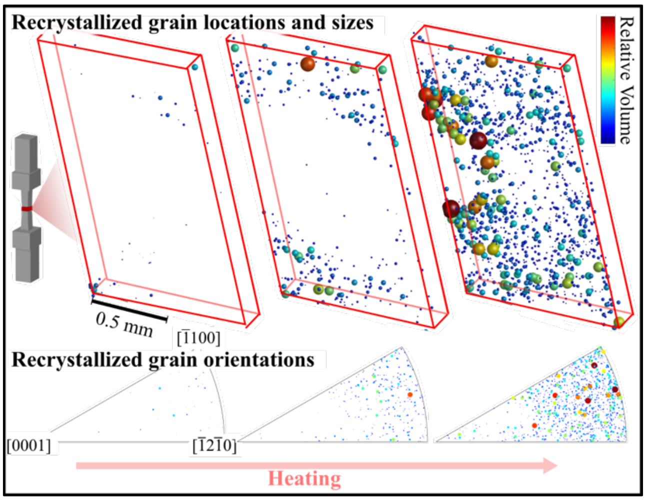

Fig. 3. Some of the reconstructed ff-HEDM data from CHESS showing the emergence of low-deformation, <u>presumably</u> recrystallized grains. However, we were not able to whether these were recrystallized grains and recovered grains from the as-deformed microstructure with this technique.

<u>ESRF Results:</u> The advantage of the if-HEDM technique (the first of the two techniques performed during the ESRF experiment) is the fact that we can use it to observe even highly deformed grains. This capability allowed us to track grains from the initial as-deformed condition all the way to the fully annealed condition in-situ. Some of the time sequence of the if-HEDM data is shown in **Fig. 2** (middle row): Here, one can observe Bragg reflections in the initial microstructure that grow in size and intensity with annealing time and temperature, corresponding to recovery, recrystallization, and grain growth. In order to identify and track grains from the initial as-deformed condition through the annealing process, the following image processing was applied: (1) Any shifting in the image was manually adjusted; (2) A median background image was subtracted; (3) A bilateral filter was applied; (4) Individual Bragg reflections were identified using OpenCV blob detection; (5) The individual Bragg reflections (size, total intensity, location, and angular spread) were measured and tracked from time step to time step. Several of these steps are demonstrated in **Fig. 4**. These measurements allowed us to observe—for the first time—how many grains at each step were newly recrystallized, how many existed from the previous time step, and how many disappeared (**Fig. 5**).

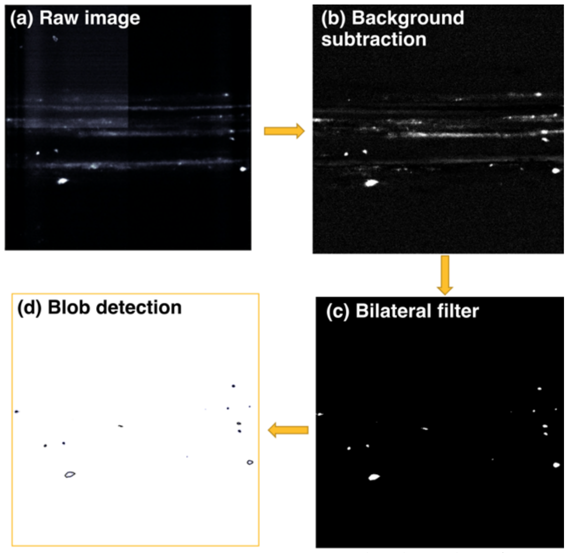

Fig. 4. Image processing applied to the if-HEDM data to track grains from initial condition through the annealing process.

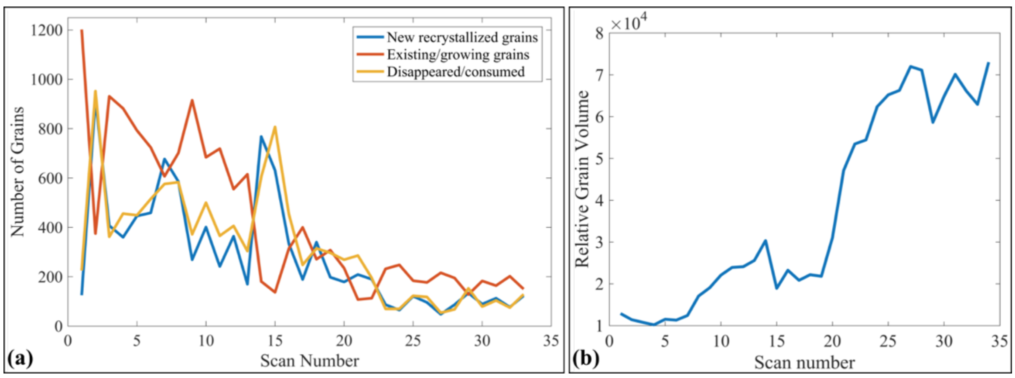

Fig. 5. Number of newly recrystallized, existing, and disappeared grains at each annealing step (a). Average relative grain volume at each annealing step (b).

From this analysis came a very unexpected observation: Within our field of view, roughly 50% of the grains in the fully annealed microstructure also existed in the initial as-deformed condition. This is counter to conventional thought: The traditional understanding is that the microstructure will become what is commonly referred to as "fully recrystallized," wherein all of the grains are *new* grains that have recrystallized at some point during annealing. In fact, this assumption is so engrained in our understanding of these processes that a common measure for in-situ or ex-situ annealing studies is the percent recrystallized and the time and temperature to the fully recrystallized state. In other words, it is almost a "given" that the annealed microstructure is fully recrystallized. The fact that roughly 50% of the grains in the final (what we would normally consider fully recrystallized) state are *not* recrystallized grains is both surprising and important, because these pre-existing grains will presumably carry with them the initial strong crystallographic texture, thus limiting the amount by which the texture can be weakened during annealing (which, recall, is the sole motivation behind this processing step). Preliminary analysis shows that we may be able to predict which grains in the initial as-deformed condition will "survive" the annealing process. **Fig. 6** shows that the orientation spread for the survived grains is low when compared to all grains in the initial microstructure of similar size.

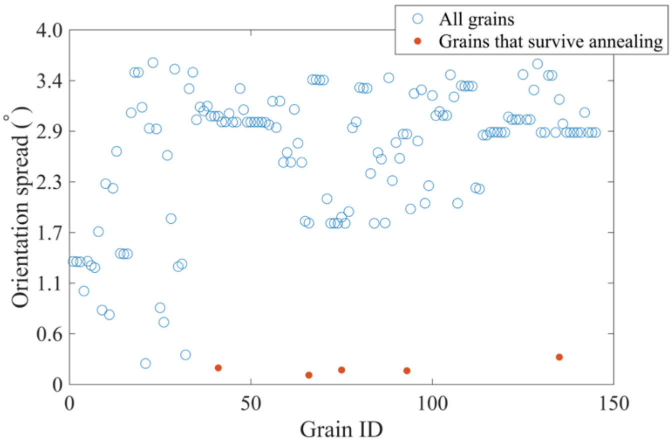

Fig. 6. Orientation spread for grains of similar size in the initial as-deformed state. The red closed markers indicate those of the grains that "survived" annealing and existed in the final microstructure.

The Need for New Experiments: As stated above, the unexpected finding regarding non-recrystallized grains from the initial as-deformed microstructure surviving the annealing process (instead of being consumed by recrystallization) is both surprising and potentially very important to understanding the effects of the initial microstructure on the annealing process and on the weakening of the crystallographic texture. We were able to use the previous experiment to develop a preliminary hypothesis about low orientation spread grains being most likely to survive annealing (**Fig. 6**). However, these results have also introduced new questions, specifically: (1) How does the evolution of low vs. high orientation spread grains differ during annealing? (2) Do non-recrystallized grains differ from recrystallized grains during grain growth or in the final microstructure? (3) Are these phenomena also observed in other components of the sample texture (i.e., those with their basal planes aligned with the rolling vs. transverse vs. normal directions)? In the previous experiment, we developed a procedure for collecting if-HEDM data and then "zooming" into specific non-recrystallized or recrystallized grains using DFXM (**Fig. 7**). We demonstrated this procedure for one non-recrystallized grain at three stages during annealing. In the future, we plan to repeat these measurements for multiple non-recrystallized and recrystallized grains to answer Questions 1 and 2. We also plan to repeat these experiments for different parts of reciprocal space corresponding to different texture components to answer Question 3. (This is necessary because of the relatively small field of view of if-HEDM in reciprocal space.) The potential impact of answering these questions will be a new understanding of the role of the initial microstructure in these critical annealing processes for lightweight Mg alloys, as well as key observations and statistical measurements that will be directly implemented into computational models of annealing within the DOE-funded PRiSMS Center at the University of Michigan.

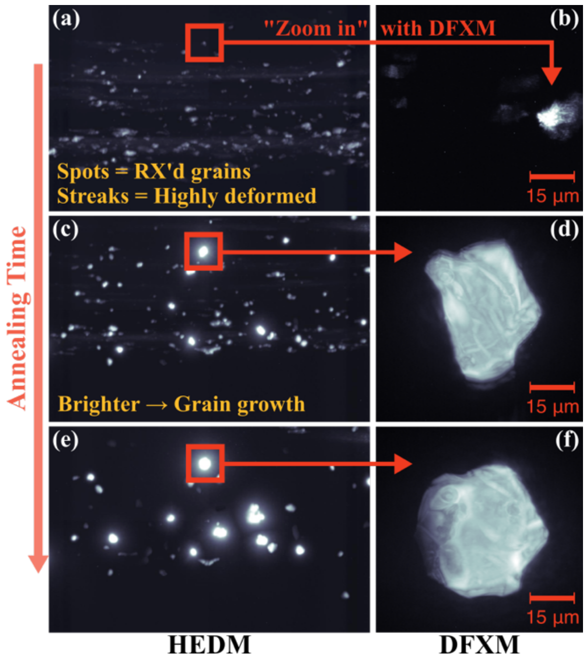

Fig. 7. "Zooming" into a non-recrystallized grain with DFXM.

[1] U.N. Environment, Transport, UNEP - UN Environ. Programme. (2017). http://www.unep.org/explore-topics/energy/what-we-do/transport (accessed August 30, 2021).
[2] T.D. Berman, J.E. Allison, Relating Texture and Thermomechanical Processing Variables in Mg–Zn–Ca Alloys, in: J.B. Jordon, V. Miller, V.V. Joshi, N.R. Neelameggham (Eds.), Magnes. Technol. 2020, Springer International Publishing, Cham, 2020: pp. 175–180.
[3] J. Victoria-Hernández, S. Yi, D. Klaumünzer, D. Letzig, Recrystallization behavior and its relationship with deformation mechanisms of a hot rolled Mg-Zn-Ca-Zr alloy, Mater. Sci. Eng. A. 761 (2019) 138054. https://doi.org/10.1016/j.msea.2019.138054.