ESRF	**Experiment title:** Understanding the role of recrystallized versus non-recrystallized grains during annealing in Mg alloys	**Experiment number**: MA-5401
Beamline: ID06	**Date of experiment**: from: 03 Oct 2022 to: 12 Oct 2022	**Date of report**: 11 Sep 2023
Shifts: 18	**Local contact(s)**: Can Yildirim, Albert Zelenika	*Received at ESRF:*

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Preliminary Report:

Motivation: The aim of this experiment was to study static recrystallization during annealing in magnesium (Mg) alloys using in-situ dark-field X-ray microscopy (DFXM). Lightweight magnesium (Mg) alloys have enormous potential for reducing fuel consumption of transportation vehicles, which account for ~25% of energy related greenhouse gas emissions worldwide. However, the low formability of Mg sheet due to its strong basal texture has precluded its widespread use. Recent studies suggest that post-process annealing treatments can result in desirably weakened split-basal textures and substantial increase in formability. The goal of this study was to better understand these processes, namely, where recrystallized grains nucleated and how recrystallized versus non-recrystallized grains behaved during annealing. Prior to the experiment, our collaborators used electron backscatter diffraction (EBSD) to show that recrystallization occurs near deformation twins that form during the rolling process (prior to annealing). This information led us to focus on to the mechanical deformation stage (as opposed to the annealing stage) for this synchrotron experiment and study how local stresses affected the nucleation and growth of deformation twins under mechanical loading.

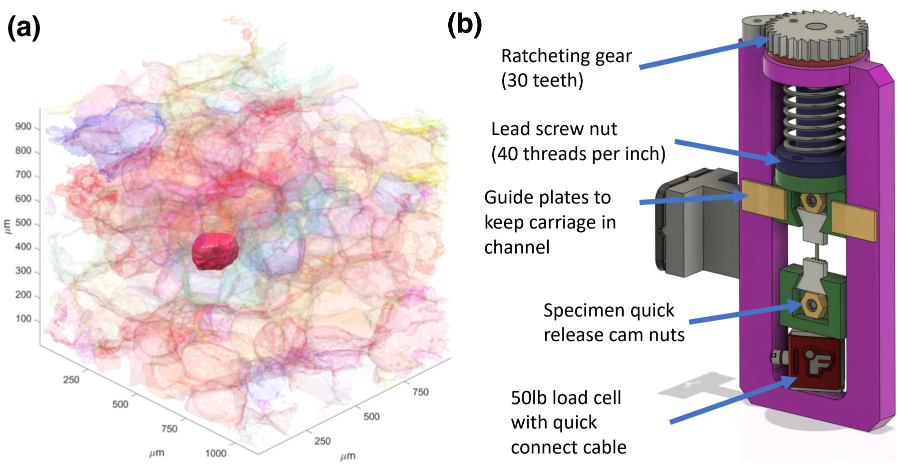

Fig. 1. (a) 3D grain mapping showing the selected grain of interest (red, opaque) inside its grain neighborhood (multicolored, semi-transparent); (b) Miniature tensile stage customized for DFXM on ID06.

Experiment Overview: Prior to the beamtime, coarse-grained Mg-4Al alloys were cut into tensile specimens and pre-characterized using laboratory diffraction contrast tomography (lab-DCT) (**Fig. 1a**). These results were used to identify grains that: (i) were oriented such that extension twinning was likely, and (ii) were oriented so that a high-structure-factor Bragg reflection could be aligned in the DFXM microscope. At the ESRF, the samples were mounted in our custom miniature load frame (**Fig. 1b**) and loaded in tension while the microstructural evolution was measured inside our selected grains using DFXM. We captured the nucleation and growth of multiple {10-12}-type deformation twins inside two different grains inside two different samples, including local elastic strains and intragranular misorientation in the parent grain during these processes.

<u>Results:</u> Preliminary results for one of the two samples are shown in **Fig. 2**. The top row shows the twin morphology and twin variant inside the grain at three different load steps (macroscopic stress values shown at the top of each column in red). Notice how new twins appear and the {01-12} twin coarsens with each new load step. The twin variants were identified and the twin morphologies measured using DFXM (specifically, from the total intensity images), which were taken in two-dimensional "slices" rastered across the grain and then stitched together in post-processing (resulting in three-dimensional reconstructions). The bottom row of **Fig. 2** shows the intragranular misorientation in the parent grain during these processes, i.e., mechanical loading and twinning, also in three dimensions. Notice how more and more red (i.e., high intragranular misorientation) regions appear during with each new load step. These high intragranular misorientation regions are in fact the locations where the twins intersect the grain boundaries. These results may suggest that new twinning events are the results of local stresses near grain boundaries caused by grain-grain interactions (e.g., twin transmission). Also measured by not shown here are two-dimensional "slices" of spatially-resolved elastic strain in the {0001} (basal plane) direction.

<u>Current and Future Plans:</u> The preliminary results shown in **Fig. 2** are one of the two samples that we measured during mechanical loading and twinning. Future work includes analyzing the second sample. Future work also includes using crystal plasticity finite element (CPFE) simulations to model the plasticity (slip and/or deformation twinning), intragranular misorientation, and elastic strain (using the full pre-characterized 3D grain neighborhood shown in **Fig. 1a**). The CPFE simulations will be compared against our experimental findings to identify weaknesses in our modeling capability, and to help elucidate how, when, and where new twins nucleate during mechanical deformation. More broadly, these results will be used to understand the critical deformation twinning processes that precede recrystallization during annealing in an effort to produce lightweight structural Mg alloys with desirably weakened textures.

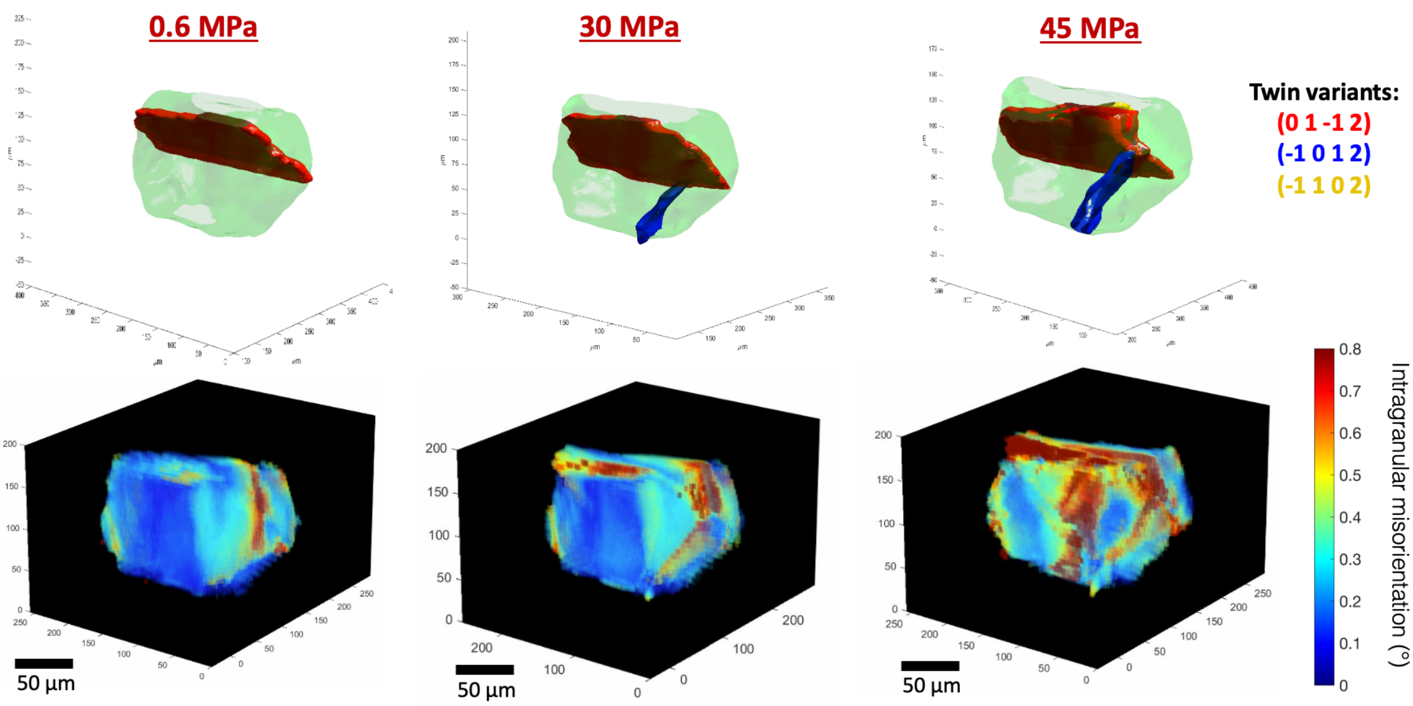

Fig. 2. Preliminary results: (top row) DFXM was used to identify which twin variants formed during loading including their morphology inside the parent grain; (bottom row) DFXM was used to spatially map intragranular misorientation in the parent grain around the grain and twin boundaries.