ESRF	**Experiment title:** Evolution of Dislocation Structures with Time-Resolved Dark-Field X-ray Microscopy	**Experiment number**: ma4481
Beamline: ID06-HRM	**Date of experiment**: from: Oct 27, 2020 to: Nov 3, 2020	**Date of report**: Sept 9, 2021
Shifts: 7 days	**Local contact(s)**: Can Yildirim	*Received at ESRF:*

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Report:

The original proposed work in this experiment intended to quantitatively measure the properties and dynamics of patterns of dislocations deep inside single-crystals by refining time-resolved DFXM. Our goals were to study the statistical enseble of disocations and quantify the dislocation interaction forces that were long-sought by crystal plasticity – over a range of materials to enable generality between material systems. Unfortunately, the restrictions on travel and lab access (in France, Denmark and USA) added significant difficulties to our original plan for the experiment, and we reduced its scope to have a successful experiment. Specifically, we experienced significant delays and stock shortages of materials when ordering samples with shipping delays that prevented us from ordering the Ti samples to complement our Al experiments. Then with limited access to our labs, we were unable to pre-process the samples, preventing us from studying how well-defined sets of initial deformations unravel differently upon recovery. Supply shortages and laboratory access restrictions have been improved and restructured by now; we now understand how future experiments can test materials and initial deformations that we were unable to measure in our original plan for beamtime in Oct 2020.

Under these difficult conditions, this experiment ended up studying the dynamics of dislocations at high temperatures in previously nanoindented single-crystal aluminum, resolving how the statistical collections of dislocations pattern at $T > 0.9\ T_m$. While the generality and associated impact of the work was diminished by its limited scope, our results were still able to map many of these dynamics in this first simplified system. We focused on the high $>0.9T_m$ temperatures for which we could resolve the dislocation structures at the single-dislocation level. With this view, we observed interesting dynamics of dislocations navigating through obstacles, including their passage in and out of single subgrains (Fig. 1). We note that a data-transfer issue between the camera and data acquisition system prevented us from capturing many of these dynamics, and we intend to sort through these challenges ahead of time for our next experiment so that they will be robust for us and other users. Just at the edge of melting, we also resolved how dislocations in aluminum pack into multiscale “lattices” of dislocation boundaries that freely transmit dislocations between neighboring boundaries in a oscillatory fashion. We show a representative snapshot from the dislocation lattices in the weak-beam image in Figure 2. Our ongoing efforts are working to quantify these dynamics and build experimentally-driven models to quantify the local stresses that drive the pattern evolution. At the very highest temperature (T_m), we resolved how dislocation patterns chaotically break down as the material melts, however, the polymer lenses introduced artifacts into our images that prevented us from resolving these dynamics clearly (see below for full description and follow-up experimental needs).

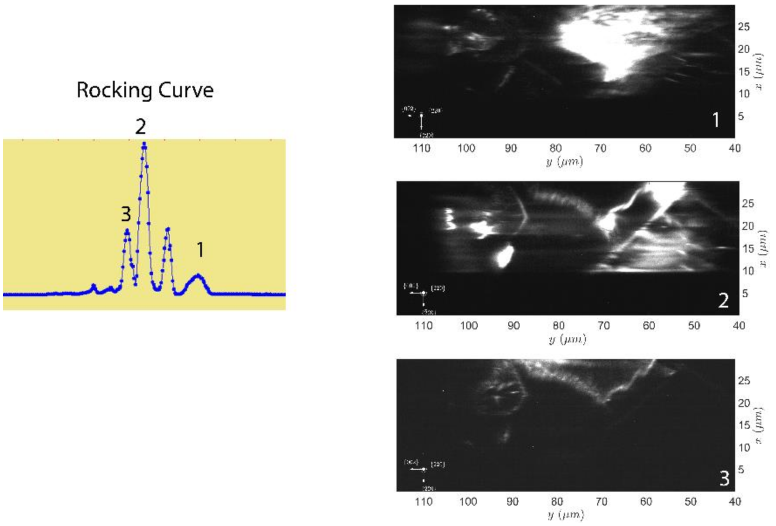

Figure 1. Complex rocking curve of the high-resolution XRD peak, with DFXM images showing each primary component of the peak. These three images show each grain’s dislocation content along the weak-beam condition. Unfortunately, deleterious scatter from the CRLs mitigated resolution and issues in data transfer between the camera and DAQ system prevented us from tracking dislocation as they moved between these grains.

In addition to these exciting material dynamics (work ongoing), our study also did significant technique development for DFXM studies to improve their understanding of dislocations. Our three major findings focused on (1) collecting sufficiently well-defined data to refine the forward model we developed to simulate DFXM images from deformation models[1], (2) finding optical approaches to enhance our ability to classify dislocations we image with DFXM based on their crystallographic character, and (3) resolving the full 3D structures of all dislocation boundaries with DFXM.

For the model refinement, we collected very detailed (x,y,z,χ,ϕ,2θ) scans over fully recovered crystals with significantly separated dislocations. Unfortunately, the numerical aperture of the polymer lenses we used in the experiment are proprietary information, leaving the information required for our model incomplete. We have ongoing research focused on these model refinements and comparison, and plan to repeat these scans with the well-calibrated Be CRLs for conclusive results.

To refine our ability to interpretat the dislocation character with DFXM, we explored how modifications to the contrast mechanism in DFXM experiments enhanced our ability to resolve the dislocations. For well-separated dislocations (recovered at high T), we imaged the dynmaics of dislocations at different points on the rocking curve, corresponding to the “weak-beam” and “strong-beam” conditions used for dislocation tracking in TEM. With a view of *both* the structure and dynamics of the dislocations, we are refining our ability to classify dislocation assignments with DFXM. We demonstrate the contrast mechanisms’ different view of these dislocation boundaries in Fig. 2.

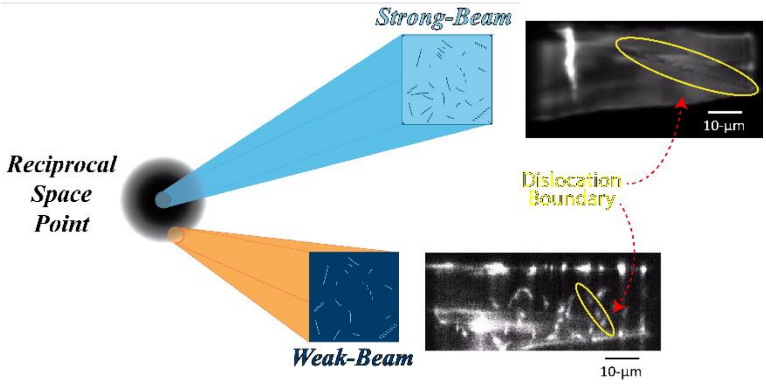

Figure 2. Illustration showing how the strong-beam and weak-beam contrast arise from images of different parts of reciprocal space – showing the same crystal deformations as shadows and bright features, respectively. We show representative images of dislcoations that comprise boundaries on the right (measured in ma4481) under these different contrast mechanisms. We highlight dislocation boundaries with yellow circles for clarity. The significant bright streaks on the left of these images arise from aberrations and ghosting caused by the polymer CRLs. For unambiguous assignment of these features, we need to repeat many of our ma4481 experiments with Be CRLs, whose wider apertures will make the results easier to interpret.

Our study also began to demonstrate the opportunities to study the 3D microstructure of metallic crystals with DFXM, but we did not have time to refine our view of the 3D structure with only one single beamline operator. In a follow-up experiment done on in-house ESRF beamtime, we completed this component of our initial study and saw significant advances in our capabilities with the further refined setup (i.e. Be CRLs, more controled samples, etc.). That work is currently in the final stages of being written up and will be submitted by the end of this month; we include sample results in Fig. 3 to demonstrate the opportunities in these studies. Using these *z*-rastered scans during ma4481, we attempted to map the full extent of the dislocation structures to inform our interpretation of the dislocations we resolved interacting at higher temperatures. With the refined 3D characterization approach, we are now confident that our next experiment will be able to resolve this directly.

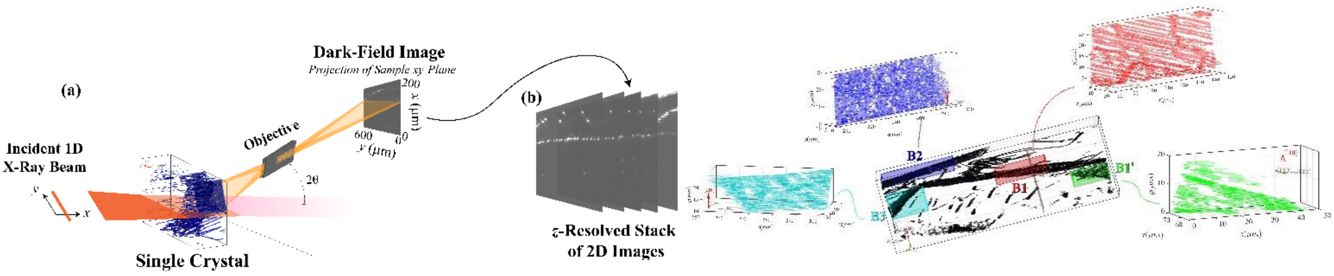

Figure 3. (left) (a) Illustration of the experimental geometry for the 3D dislocation structure measurements, which collect 2D images from the line-beam illumination through a stack of z-positions. (b) We then pack the images into a 3D image array that we input to gradient-based image segmentation methods to identify the dislocations. (right) The resulting arrays show us a clear picture of the dislocation boundaries, which we identified as {110} tilt boundaries that pack into specific boundary planes based on the geometry and dislocation lines.

Unfortunately, due to unforeseen challenges with the polymer lenses (i.e. the numerical aperture is proprietary), we were unable to *fully* calibrate our experimental geometry, though we were able to collect more conclusive data than we had done previously. Our experiment was also limited by the polymer lenses, which created additional artifacts in the signal (likely caused by ghosting effects from other reflection components) that made features difficult to interpret when part of the crystal was in the strong-beam condition. We hoped we could simply crop the images to remove the deleterious noise effects, but ultimately found that they altered something about the detector's signal-to-noise, making the weak signals from the dislocations very difficult to see (as shown in the data from Fig. 4)

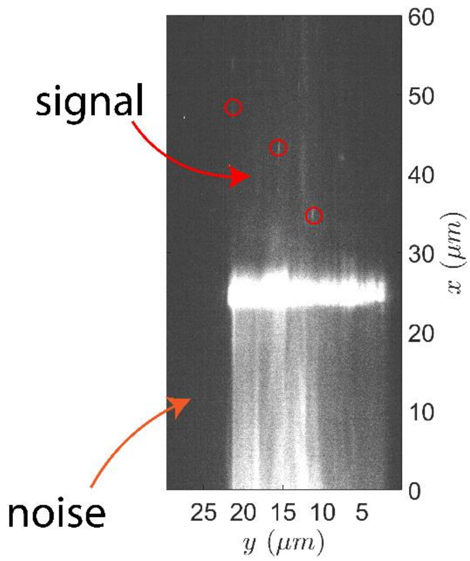

Figure 4. Sample image from the most important T_m dynamics in single-crystal aluminum with DFXM using the polymer lenses. The weak-beam condition requires that most of the sample remain out of the Bragg condition, however, deleterious scatter from other reciprocal-space components through the lenses create artifacts and noise that are significantly brighter than the signal of interest. As such, our dislocation dynamics are very difficult to track at the most important temperature regimes for this work. The apertures on Be CRLs prevent these effects, and require an additional experiment to repeat this high-T data acquisition.

Finally, we note that the original scope of the proposed work was mitigated by our inability to travel to the ESRF, which limited our experimental time because only one person was able to operate the beamline for the entire 7 days. With limited staff, we were unable to study the effects of lattice symmetry on the high-T dynamics of recovery because we ultimately had to reduce scope. We note that this has limited the impact of our study, as it has prevented us from demonstrating how DFXM measurements can generalize material dynamics beyond single-crystal aluminum. In our next experiment, we plan to remedy this by bringing a wider range of samples to afford the breadth of dislocation science necessary to demonstrate the full impact of this technique and research.