Dynamic measurements of the structural evolution of material defects at the mesoscale
NLV-001-19, Year 3 of 3

Marylesa Howard (PI), Arnulfo Gonzalez, Sean Breckling,
Ajanaé Williams, Maggie Lund, Daniel Mortenson

With contributions from Youssef Marzouk (MIT), Michael Brennan (MIT), Leora Dresselhaus-Marais (Stanford)

This work was done by Mission Support and Test Services, LLC, under Contract No. DE-NA0003624 with the U.S. Department of Energy and supported by the Site-Directed Research and Development Program. DOE/NV/03624--1173.

Challenge

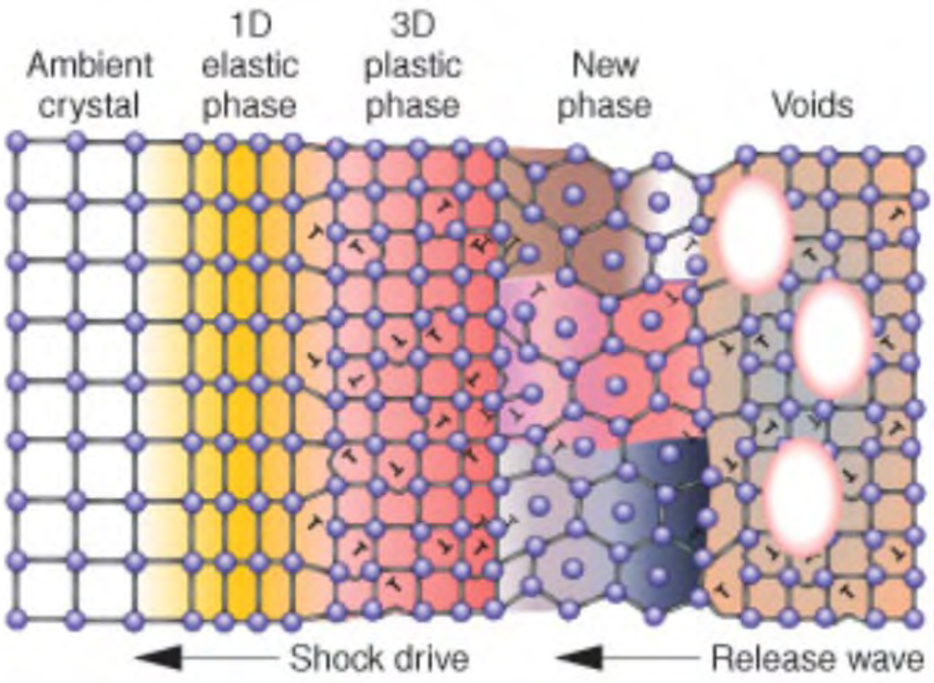
Present in essentially all materials, from circuit components used in electronics to alloys used in armor, from manufactured parts used in automobiles to nuclear weapons components in our mission space, material defects fundamentally change how materials respond to extreme conditions or fatigue over time.

Defect Engineering: If we study properties that defects induce or change in materials, we may engineer products with these defects to produce desired or required material properties

---

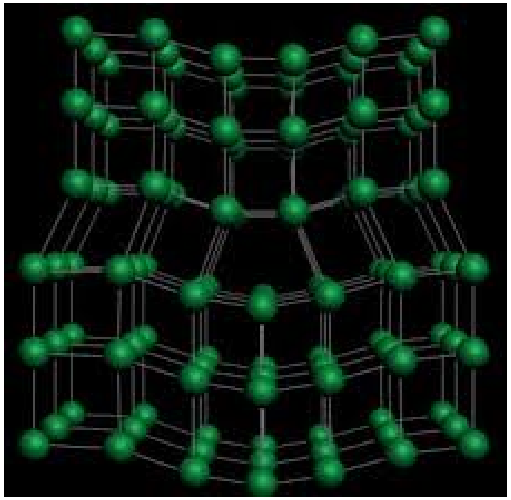
Partnering with LLNL/Stanford, who are developing a time-resolved dark-field x-ray microscopy imaging technique, the NNSS is developing the image processing toolbox that quantifies and statistically characterizes dislocation defects.

Challenge



Spatial progression of defects has never been directly measured during a shock wave at the onset of plasticity

This is the ultimate goal of this collaboration



Edge dislocation in crystal lattice
https://www.ndeed.org/EducationResources/CommunityCollege/Materials/Structure/linear_defects.htm

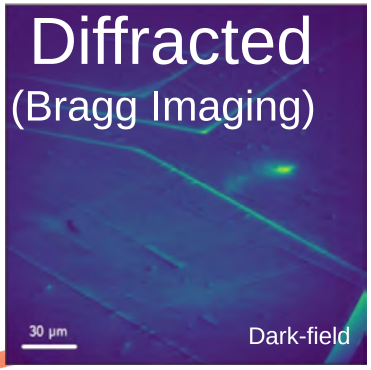
Novel imaging techniques can map the size and morphology of populations of mesoscale defects. Detailed statistical analysis of defect morphology is essential to understand how defects move and interact, irrevocably changing materials.

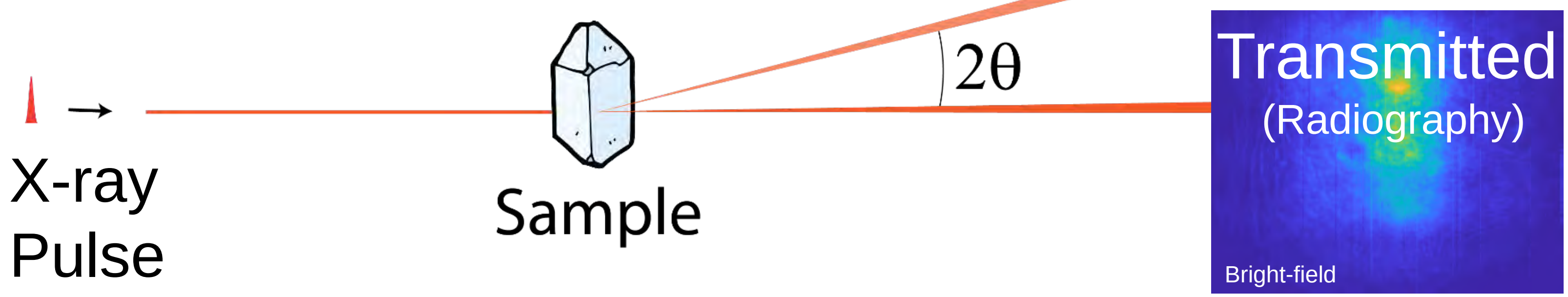
Image from https://str.llnl.gov/str/JulAug06/gifs/LorenzanaBox1b.jpg

Innovation

A new imaging technique

- Led by LLNL/Stanford, and in collaboration with NNSS, DTU, MIT, and many more, we are developing time-resolved dark- and bright-field x-ray microscopy (DFXM/BFXM) to measure bulk deformation mechanisms beneath the material's surface as the material deforms plastically.



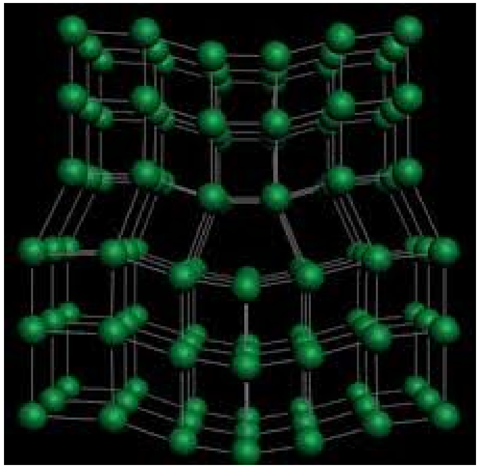


- Bright-field images show bulk material; dark-field images selectively show deformations.

- Collecting both BFXM and DFXM data, we will better understand how defects grow, propagate, interact, and cascade into large-scale material transformations.

- Experiments using XFEL facilities will induce radiation damage in single-crystal samples and enable us to capture rare events.

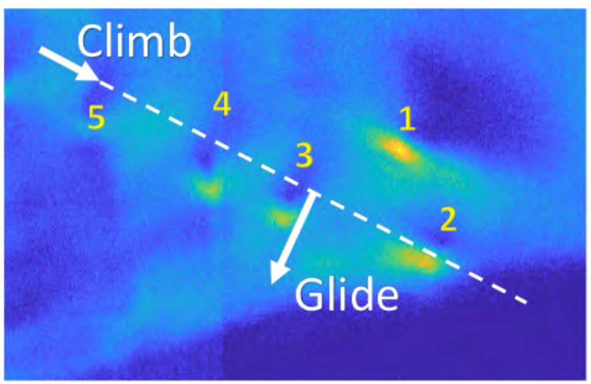
Technical Approach: Dislocations defects



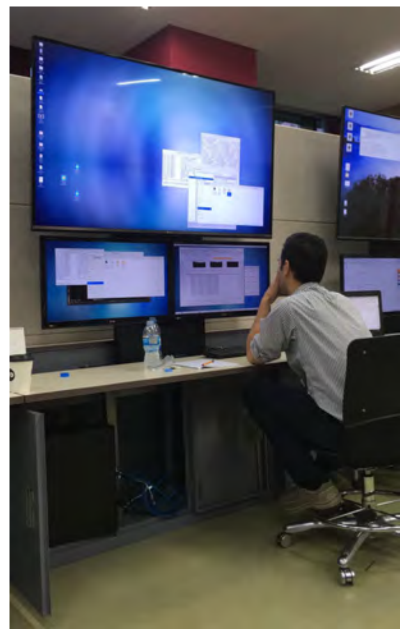
Edge dislocation in crystal lattice
https://www.ndeed.org/EducationResources/CommunityCollege/Materials/Structure/linear_defects.htm

Dislocations are dynamic—they move under shear stress, can multiply, and initiate material cracks.

We studied a mm-sized sample of single-crystal aluminum at the synchrotrons and XFELs to explore dislocations using dark-field x-ray microscopy.



Five dislocations are enumerated in this DFXM image, appearing as bright/dark pairs



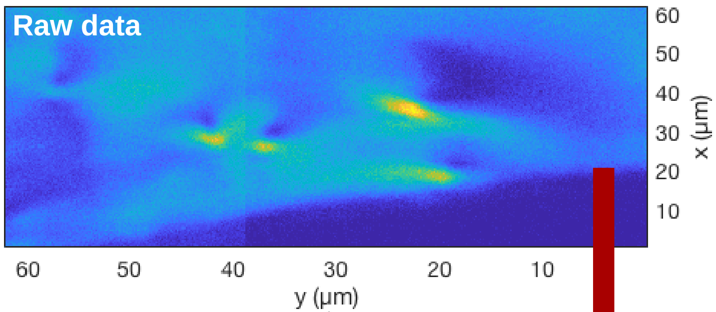
Clockwise from left:
Dr. Goznalez at the PAL-XFEL experiment;
Ms. Williams completing algorithms at APS;
Dr. Breckling inspecting the CRL setup at APS

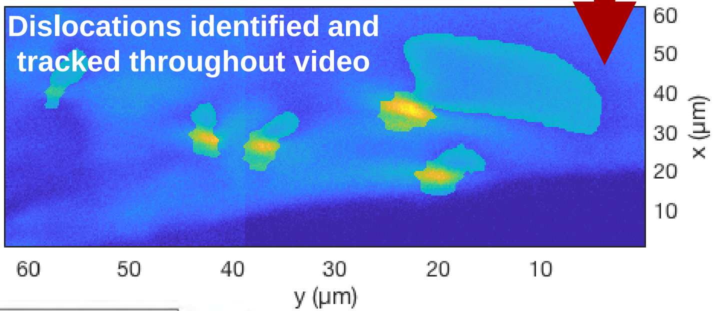
Technical Approach, Results: DFXM image processing

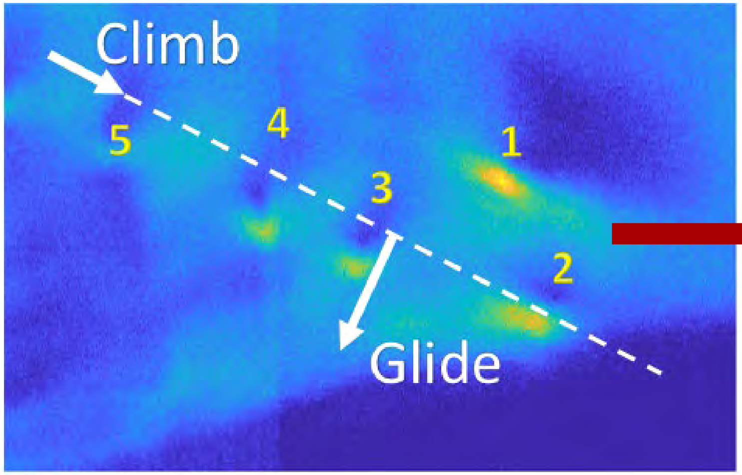
Through the fusion of image and signal processing techniques, including

- Stationary wavelet transforms,
- Binarization,
- Seeded fast marching methods,
- Kalman filtering, and
- Munkres assignment,

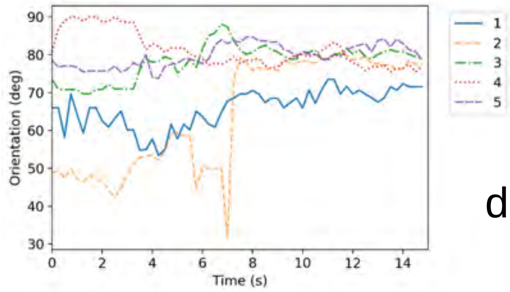
we can locate and track dislocations throughout the video. This allows quantification of behavioral features of interest.



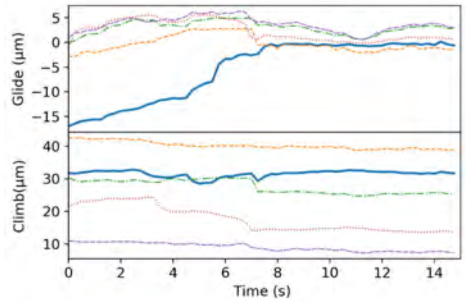




The glide and climb reference axes correspond to the physical mechanism of their motion in each direction



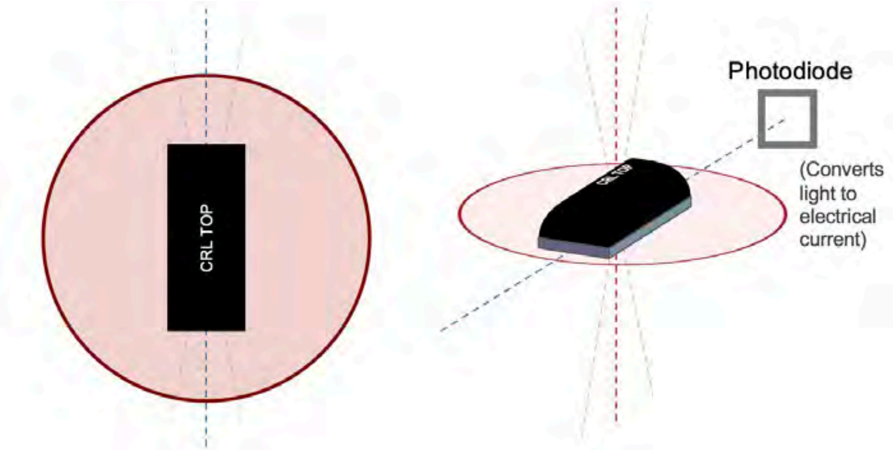
Orientation of dislocations in time

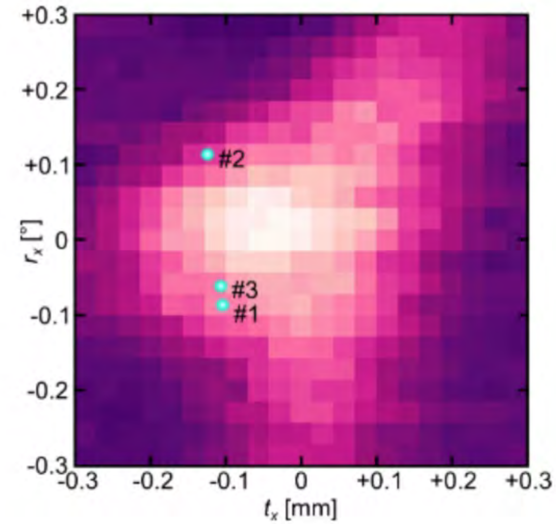


Position of dislocations in time

Technical Approach, Results: CRL alignment

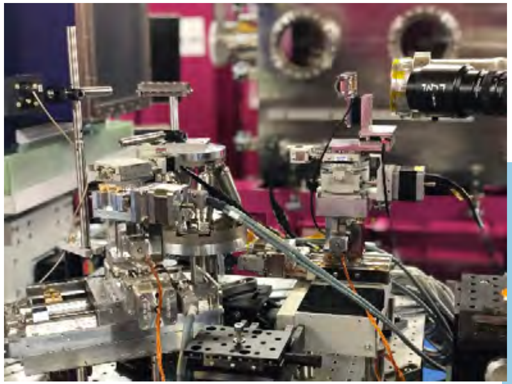
Compound Refractive Lens (CRL)



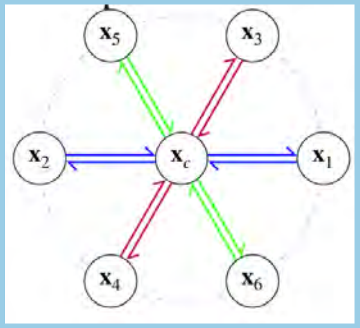


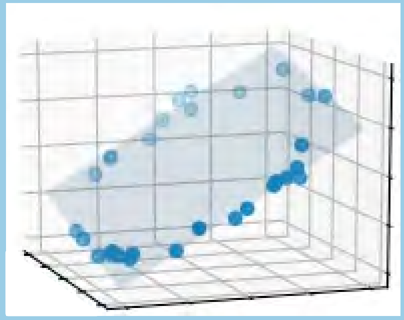
The beam hits a photodiode, which returns a voltage proportional to the intensity of the light hitting the surface. The higher that measured intensity value, the more “aligned” the lenses are.

Current alignment approach at XFELs and synchrotrons is a brute force raster scan that takes 3+ hours each time the CRL is aligned.



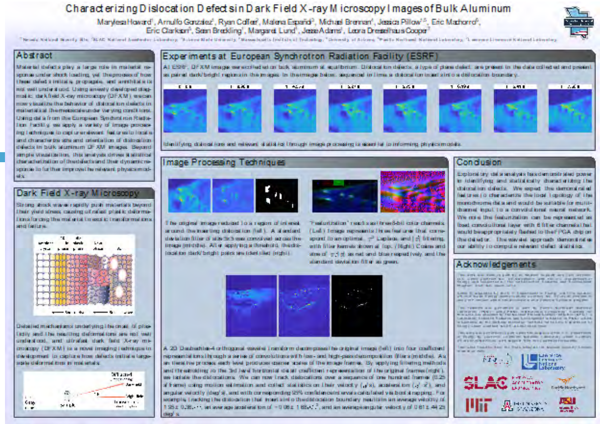
We developed an intensity drift-corrected stochastic gradient descent method with momentum for automated alignment of CRLs.





Impact

- Papers
  - *Science Advances*: In-Situ Visualization of Long-Range Defect Interactions at the Edge of Melting (accepted)
  - *Statistical Analysis and Data Mining*: Methods to Quantify Dislocation Behavior with Dark-field X-ray Microscopy Timescans of Single-Crystal Aluminum (revision submitted)
  - *SIAM Journal of Scientific Computing*: A Dynamic Amplitude-Correcting Gradient Estimation Technique to Align X-ray Focusing Optics (submitted)
  - *Journal of Synchrotron Radiography*: An Automated Approach to the Alignment of Compound Refractive Lenses (submitted)



Poster from Conference on Data Analysis 2020

- Selected Presentations
  - *Pacific Northwest National Laboratory, December 2019*: Materials Defect Study at the Mesoscale: The intersection of diagnostics and analysis
  - *Conference on Data Analysis, February 2020*: Characterizing Dislocation Defects in Dark Field X-ray Microscopy Images of Bulk Aluminum
  - *Science and Technology Work in Progress Seminar*, June 2020: Dynamic Measurements of the Structural Evolution of Material Defects at the Mesoscale

Impact

- Further Impacts
  - Conducted first-ever *Data Analysis for Nuclear Security Sciences* Workshop, held in North Las Vegas, January 2020
    - Participants from LLNL, PNNL, SLAC, MIT, Arizona State University, University of Arizona
    - Presented 3 SDRD project “mini-problems” throughout the week
    - Future events planned for FY22 in partnership with PNNL
  - Developed collaboration with MIT that extends beyond this project

Impact

- Experiment Proposals: Beamtime
  - PAL-XFEL, May 2019
  - European XFEL, October 2019
  - APS, February 2020
  - PAL-XFEL, May 2020 – withdrawn due to travel restrictions
  - ESRF, October 2020 – virtual experiment
- Other Impacts
  - Collaboration with University of Alabama resulted in new hire postdoc in Year 1
  - Partnership with MIT and serving as External Research Advisor to PhD student
  - BYU undergraduate intern joined our team as casual employee
  - Image processing techniques are now being absorbed into NEA; CRL alignment study is leading to work in RAID / SDRD FY22 for accelerator beam tuning

HED Hutch, European XFEL

Bridging the SDRD Valley of Death

Reaction History Analysis (RHA) of legacy Underground Testing (UGT) relies on both image and signal processing techniques.

Film Grain

Oscilloscope film images contain a large degree of noise due to the aggregation of grains. Grain clusters may contribute to the width of signals in the signal extraction process, thereby introducing error into measurement and subsequent analysis.

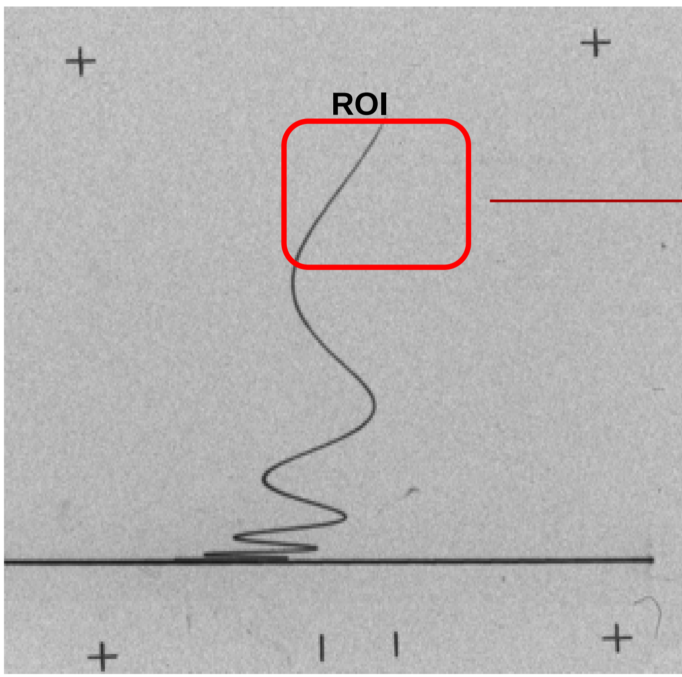
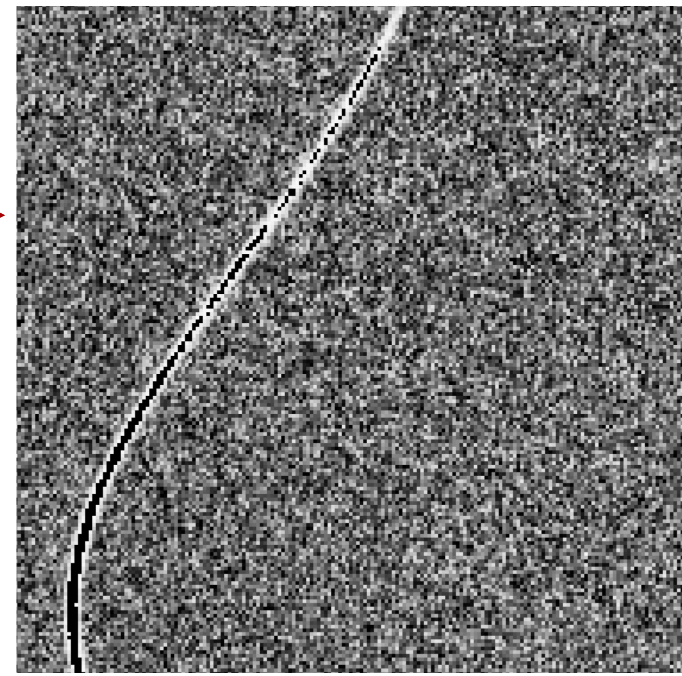


Image processing methods are applied to augment grains

Grains can affect the computer's interpretation of the signal width (*left*), affecting the extracted Rossi signal (*right*)



Bridging the SDRD Valley of Death

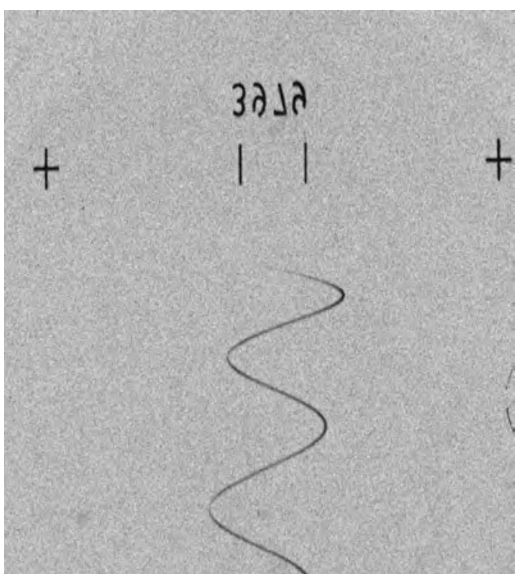
Image processing techniques and skills built in this SDRD project are now being deployed as proof of concept in RHA for future programmatic integration, expected in FY22.

These images demonstrate our ability to produce realistic film grain for synthetic Rossi scope images and extract quantities of interest. This enables us to build robust synthetic film sets

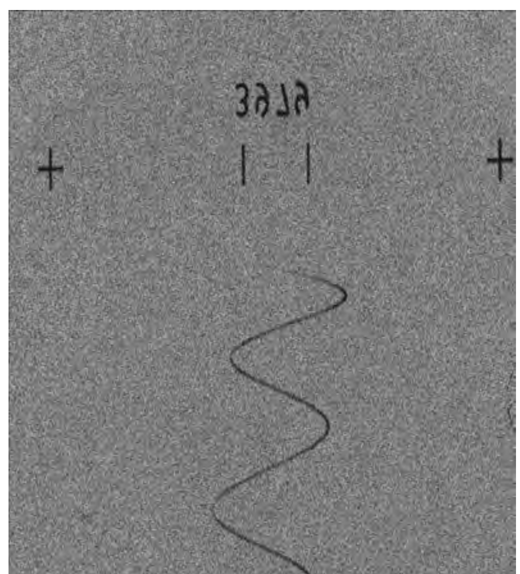
- for training future analysts, and
- to develop deep learning models using a library of synthetic films.

Computational methods are being developed to synthesize realistic film images and replicate their properties, in order to test algorithms.

Synthetic film



Synthetic film with applied grain model



Synthetic film with labels: signal, grain, background