32.0 ALGORITHMIC ANALYSES OF X-RADIOGRAPHY AND COMPUTED TOMOGRAPHY FOR MULTISCALE STRUCTURAL INVESTIGATIONS OF METALS (LEVERAGED)

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This project initiated in Fall 2017 and is supported by Los Alamos National Laboratory (LANL). The research performed during this project will serve as the basis for a Ph.D. thesis program for C. Gus Becker.

32.1 Project Overview and Industrial Relevance

X-ray radiography allows for the imaging of materials in a non-destructive manner by observing x-rays transmitted through the material. This method can be used for post-mortem analysis, as well as in-situ imaging of microstructural development. For example, dynamic imaging of microstructural evolution during processing (e.g. casting or directional solidification) can further our understanding of the mechanisms driving microstructural development, improving predictive capabilities [32.1]. X-ray radiography can also be used to capture multiple images of a sample from different angles, which can be reconstructed and represented as a three-dimensional model. A reconconstruction of a metal sample created through additive manufacturing (AM) can be generated to reveal microstructural characteristics and internal defects. Reconstructions can also be created from four-dimensional microscopy (three spatial and one temporal dimension), in which images are collected from different angles of a time-evolving sample. These reconstructions are created using techniques like the Time-Interlaced Model-Based Iterative Reconstruction (TIMBIR) [32.2], which improves temporal resolution to capture microstructural evolution during processing.

Synchrotron x-ray sources can be used to obtain high-spatial resolution x-ray radiographs (on the order of 1 µm), however these images have a small fields of view and require significant beam time at national user facilities. High-energy micro-focus x-ray radiography has the drawback of lower spatial resolutions, but increases the field of view significantly and can be performed in a laboratory. These laboratory instruments consist of a high-energy micro-focus x-ray source, radiation shielding (typically in the form of a lead-lined cabinet housing the source), a scintillator to convert x-rays to visible light, and a camera/detector to collect the produced light. Tomographic data can be obtained when a rotation stage is included to rotate the sample. Solidifiation experiments are often performed at synchrotron x-ray facilities like the Advanced Photon Source (APS) at Argonne National Laboratory (ANL), as well as in facilities with micro-focus x-ray capabilities, including the non-destructive testing group (E-6, formerly AET-6) at LANL to study the microstructures and properties of materials. Many experiments have been performed by CANFSA faculty and students at Mines to study different aspects of alloy solidification, such as crystal growth and solute segregation [32.1], primary dendrite spacing and size [32.3], dendrite fragmentation [32.4], and the effect of different cooling rates on solidification [32.5].

When x-ray radiography and computed tomography are used for the non-destructive imaging of metals, sample thickness is a contsraint for high density alloys. X-rays at national user facilities (with energies of ~30 keV) can typically penetrate thin foils of material (~100 µm to ~1 mm) to image thin sections or small volumes to produce computed tomographic reconstructions [32.5]. These typically cannot be used to image thicker or larger samples, unless special high-energy beamlines are used with x-ray energies up to ~150 keV. High-energy micro-focus x-ray imaging utilizes x-rays with energies of up to 250 keV and beyond, allowing sample thicknesses on the order of millimeters and larger fields-of-view on the order of centimeters. Proton radiography (pRad), in which 800 MeV energy protons are used as an incident radiation source at LANL's Los Alamos Neutron Science Center (LANSCE), allows for even larger sample sizes and the ability to probe materials with high atomic numbers. This does, however, come at the cost of lower spatial resolution [32.1]. Proof-of-concept experiments of Transmission High-Energy Electron Microscopy (THEEM), in which extremely high-energy electrons (15 GeV) are transmitted through samples at the Stanford Linear Accelerator Center (SLAC) to achieve deeply penetrating electron radiography have also been demonstrated [32.6]. This method has yet to be optimized, but holds potential for dynamic studies of high atomic number (Z) materials. The goal of this project is to establish high-energy (~160 keV), micro-focus x-ray imaging in a laboratory setting at Mines, achieving capabilities between those of synchrotron x-ray facilities (i.e. ~1-2 µm spatial resolution and ~2 x 2 mm^2 fields-of-view) and pRad (~ 25 µm spatial resolution at best for the ×7 magnifier and ~17 x 17 mm^2 field-of-view, although note that the ×1 lens provides a 12 × 12 cm^2 field of view) .

32.2 Previous Work

Prior to the start of this project, *in-situ* solidification of metals was studied using synchrotron x-ray imaging at the APS and using high-energy micro-focus x-ray radiography systems in a laboratory setting at LANL, in collaboration with E-6. An image processing method was created to process data from these experiments. Once processed, this data can be analyzed to capture phenomena like the multiscale solidification dynamics in metallic alloys or defects related to AM processes. This image processing method, developed at the beginning of this project, consists of modular ImageJ macros that each take a directory of images from an experiment and performs a programmed enhancement to experimental images. The processed images are saved as separate files to preserve the original, unedited images. The modular nature of these scripts allows for processing steps to be reperformed in different sequences on the original images, adding to or changing the image processing steps to achieve different results and quantitatively describe dynamic events.

The modular image processing method is demonstrated through the processing of the radiography of the solidification of an Al-Ag alloy (**Figure 32.1a**). This experiment was captured with high-energy micro-focus x-radiography with LANL's E-6 using the same experimental set-up as previous synchrotron x-ray experiments, in which a steel bar with a rectangular portion removed to act as a window for the transmittance of x-rays. Micro-focus x-ray radiography allows for a larger field-of-view than synchrotron x-ray radiography, so the experiment designed for synchrotron radiography takes place in only a fraction of the captured area of the micro-focus system. The first script extracts the lighter portion of the radiographs where the experiment is taking place (**Figure 32.1b**). Another script removes a set amount of pixels from each side of the images, allowing for better contrast (**Figure 32.2a**). Vertical and horizontal banding artifacts from the imaging process can be reduced with a script that performs fast Fourier transform filtering (**Figure 32.2b**).

32.3 Recent Progress

Following the donation of an x-ray cabinet from E-6 at LANL, the retrofitting of this cabinet with a high-energy micro-focus x-ray system is nearing completion. A lab space on the Mines campus has been prepared to hold the system. This system will be used to perform computed tomography of a variety of AM-built lattice structures as a way to inspect the inside of the structures and generate 3D models. These models will be compared to the build models used to create the structures as a way to evaluate any inconsistencies. These inconsistencies, or defects, will be evaluated to determine the types of defects detectable with microfocus x-radiography. Micro computed tomography of these lattice structures is also underway using a Zeiss Xradia 520 Versa Micro-CT located at Mines. Analysis of the computed tomography data captured using this system will be compared to the data that will be collected by the retrofitted system.

To prepare for the 3D data analyses that will be necessary to evaluate the computed tomography of AM-built lattice structures, a 3D dataset has been obtained from LANL depicting the computed tomography of mock high explosives (HE). The development of mock HE allows for the analysis and testing of materials with similar properties in lieu of HE, when the use of HE would represent a safety concern. The mock HE represented in the 3D dataset are crystals of 5-Iodo-2'-deoxyuridine (IDOX), a surrogate for cyclotetramethylenetetranitramine (HMX), embedded in a binder [32.7]. In order to build experience working with 3D data, an attempt has been made to segment the individual IDOX crystals for separation from other crystals as well as the binder.

The mock HE dataset itself has been reconstructed so that each image in the dataset represents a slice of the sample parallel to the axis of rotation, rather than the projection images of the sample orthogonal to that same axis that are collected through the tomography scan. The process of segmenting individiual IDOX crystals in the reconstructed images consists of three major steps, all of which are performed through image processing in Python with the software package *scikit-image* [32.8]. The first step is to perfom some kind of thresholding to convert the reconstructed, 16-bit images from full bit-depth (containing 2^{16} values) to binary images (two values: zero or one). This was performed by converting the images from 16-bit to floating point values (all values are represented in the range of zero to one), then applying a Gaussian filter to smooth out noise. The new values are thresholded at a certain value (**Figure 32.3**), at which point the values above that value are set to one, and all other values are set to zero. The images can then be more clearly represented in 3D by rendering the binary imges as a volume (**Figure 32.4)**. This was perfomed using the open source 3D visualization and analysis program *tomviz* [32.9]. After binary images are created by a thresholding method, a segmentation routine is performed on the binary images using a watershed algorithm. For a given binary image (**Figure 32.5a**), a distance map is created (**Figure 32.5b**), assigning a value at each pixel corresponding to the distance to a zero value of the corresponding pixel in the original binary image. Local maxima are found for that distance map, at which point these maxima are passed to the watershed algorithm as locations to begin "flooding" the binary image. The result is an image of labeled regions that is combined with the reconstructed image from which the binary image was created (**Figure 32.5c**), generating a composite image outlining the segmented regions and their correlation to the IDOX crystals (**Figure 32.5d**). Upon inspection, it is clear that the segmentation does not correlate perfectly with the IDOX crystals, meaning further work is required to refine this process.

32.4 Plans for Next Reporting Period

In the next reporting period, the following tasks will be completed:

- Training on the Zeiss Xradia Versa 520 Micro-CT instrument at Mines to image AM-built lattice structures received from Los Alamos, Sandia, and Lawrence Livermore National Laboratories.
- Reconstruction of lattice structure data and identification of defects and mismatch to build models.
- Finalize delivery and installation of the Mines x-ray cabinet.
- Improve segmentation of IDOX crystals in mock HE data from LANL.
- Continue developing 3D volume processing pipelines using Python and *tomviz*.

32.5 References

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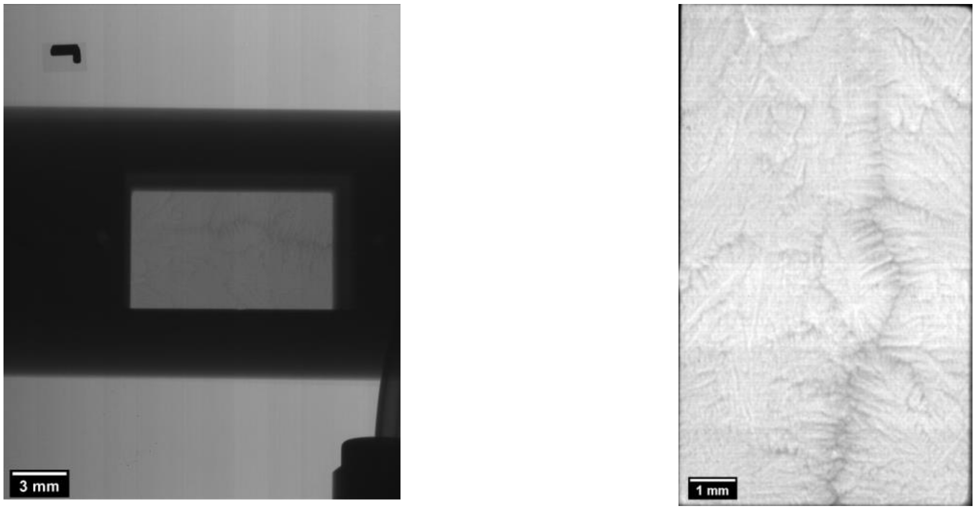
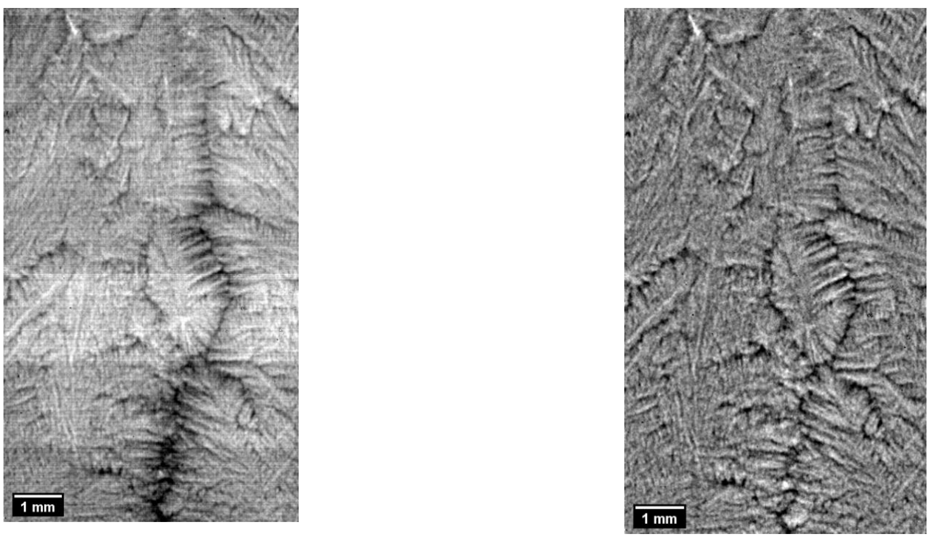
32.6 Figure and Tables



(a) (b)

Figure 32.1: A frame from the solidification process of an Al-Ag alloy obtained using high-energy micro-focus x-ray radiography of a sample undergoing directional solidification. The experiment was performed using the synchrotron x-ray radiography experimental set-up, so the experiment is entirely contained in the small, light gray window in the center of (a) and cropped to that window and rotated in (b). The horizontal feature in (a) is a steel bar. Laboratory x-ray imaging affords new opportunities to study larger fields-of-view.



(a) (b)

Figure 32.2: Further processing of the same frame of Al-Ag solidification shown in **Figure 32.1** by (a) trimming 10 pixels from each edge of the radiograph, allowing for better contrast in the solidification structure, and (b) performing fast Fourier transform filtering to remove horizontal and vertical banding artifacts. Note: rotated 90 degrees for presentability; gravity is to the right of the page.

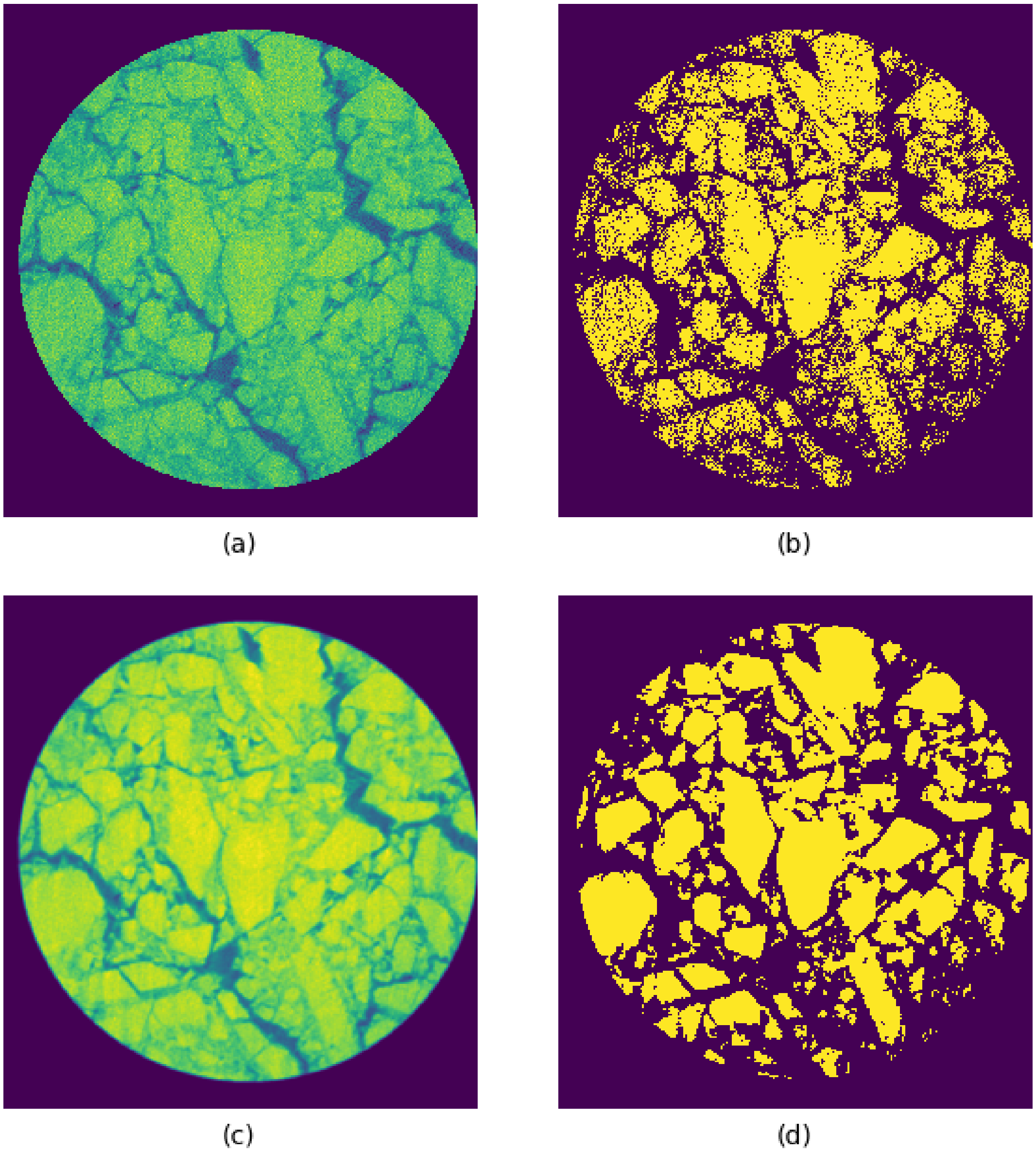
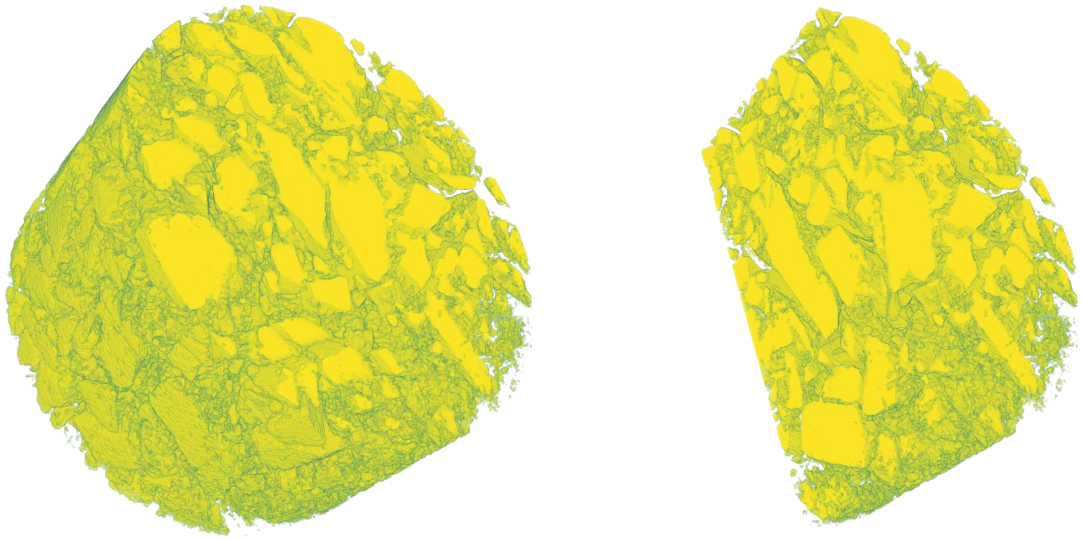


Figure 32.3: (a) Original reconstructed image of cyclotetramethylenetetranitramine (HMX) surrogate material 5-Iodo-2'-deoxyuridine (IDOX) collected through x-radiographic computed tomography. (b) Original reconstructed image converted to a floating point image and thresholded for values greater than 0.65. (c) Reconstructed image from (a) with a Gaussian filter applied. (d) Filtered image thresholded for values greater than 0.65.



(a) (b)

Figure 32.4: The entire dataset of reconstructed images of the IDOX material represented in 3D through volume rendering, (a) clipped in the XY-plane to show the slice repesented in **Figure 32.3a** and (b) clipped in the YZ-plane to show more internal structure not shown in (a).

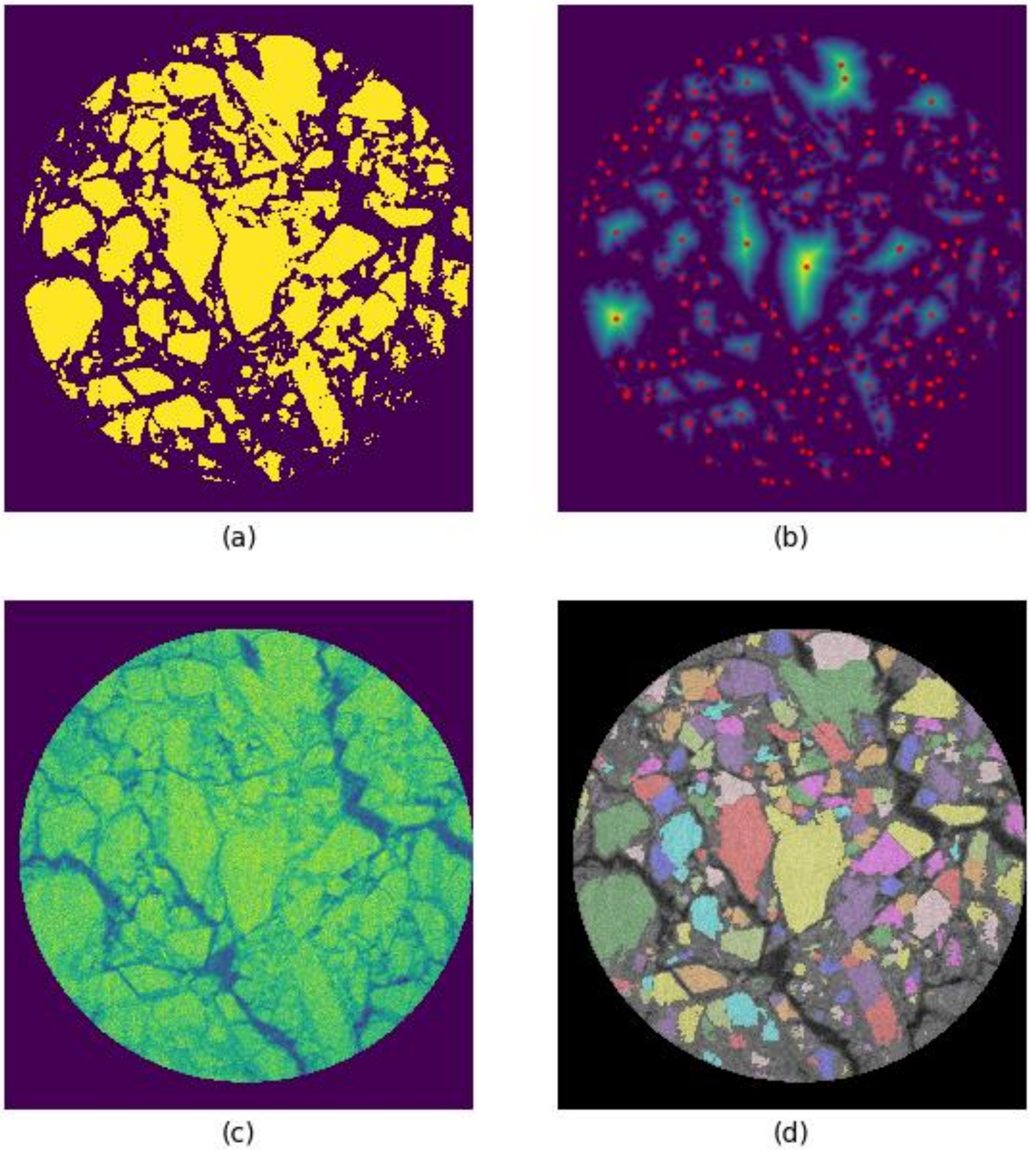


Figure 32.5: The process of segmenting individual IDOX crystals from the binder. The process begins by (a) thresholding an reconstructed image, (b) using the binary image to create a distance map that assigns each pixel a value scaled to the distance from a value of zero of the corresponding pixel in the binary image. Local maxima in (b) are represented by red points. These local maxima are passed to a watershed algorithm where those locations are used as seeds to "flood" the image and create "basins" that are labeled independently. This labeled image can be combined with the (c) original reconstructed image to create (d) a composite image that overlays the labeled regions onto the original reconstructed image.