32.0 DEVELOPMENT OF CABINET BASED COMPUTED TOMOGRAPHY METHODS FOR STUDIES OF MICROSTRUCTURES AND DEFECTS IN METALS (LEVERAGED)

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32.1 Project Overview and Industrial Relevance

X-ray radiography allows for the imaging of materials in a non-destructive manner by sending x-rays at a sample and observing the x-rays transmitted through the material. This method can be used for post-mortem analysis as well as in-situ imaging capable of capturing the development of microstructures, important in the study of solidification. Dynamic imaging of microstructure evolution during materials processes (e.g. casting) can allow for an increase in the understanding of mechanisms driving microstructural develeopment, leading to improved materials processing models [32.1]. X-ray can also be used to capture many images of a sample from different angles that may be reconstructed and combined into a three-dimensional representation of the sample. By imaging an additively manufactured (AM) metal sample, a reconstruction can be created revealing internal defects in the part. Four-dimensional microscopy (three spatial dimensions, one temporal dimension), in which images are collected from different angles of a sample that is evolving in time, can also be performed to create a changing three-dimensional reconstruction. Reconstructions like these are created using techniques like the Time-Interlaced Model-Based Iterative Reconstruction (TIMBIR) [32.2], which improves temporal resolution to capture material dynamics.

Synchrotron x-ray facilities, as well as laboratory micro-focus x-ray tubes, can be used for x-ray radiography. Synchrotron x-ray sources can be used to obtain high-spatial resolution images (on the order of about 1 µm), however these images will have a smaller field of view and require scheduling beam time at an often oversubscribed national user facility. High-energy micro-focus x-ray radiography in the laboratory achieves lower spatial resolutions, however images from larger fields of view are produced with the added benefit that these experiments can be performed in any laboratory with a micro-focus x-ray setup. These setups consist of a high-energy micro-focus x-ray source, radiation shielding for that source (typically in the form of a lead-lined box or cabinet the source is placed inside), a scintillator to convert x-rays to visible light, and a camera to collect that light. If experiments are to utilize tomographic methods of reconstructions that require images taken from different angles, a rotation stage for the sample is also necessary.

Multiple experiments have been performed at synchrotron x-ray facilities like the Advanced Photon Source (APS) at Argonne National Laboratory, as well as in facilities with micro-focus x-rays capabilities like Los Alamos National Laboratory (LANL) to study the properties of materials. Many of these experiments have been performed by our group to study different aspects of alloy solidification, such as crystal growth rates and solute segregation profiles [32.1], primary dendrite spacing and size [32.3], dendrite fragmentation [32.4], and the effect of different cooling rates [32.5].

When x-ray radiography and computed tomography is used for the non-destructive imaging of metals, high densities may constrain experiments by sample thickness. X-rays at national user facilities can typically penetrate thin foils of material (~100 µm to ~1 mm) to image thin sections or small volumes to produce computed tomographic reconstructions utilizing x-rays with energies of around 30 keV [32.5], but typically cannot be used to image 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, for example, but sample thicknesses may be on the order of millimeters. Proton radiography (pRad), in which 800 MeV energy protons are transmitted through samples at LANL's Los Alamos Neutron Science Center (LANSCE), allows for much larger sample sizes and the ability to probe high-Z (atomic number) materials at the cost of lower spatial resolution [32.1]. There have also been 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 [32.6]. This method has yet to be optimized, but hold potential for high-Z materials.

32.2 Previous Work

Prior to the start of this project, in-situ x-ray radiography experiments were performed at national user facilities like APS and in laboratory settings by our group in collaboration with AET-6 (non-destructive testing group at LANL) to capture multi-scale solidification dynamics in metallic alloys. Data from some of these experiments remains unprocessed and is currently being uploaded to a server established at CSM. Image processing of this data has begun, and will continue as more data is uploaded and processing techniques are improved.

32.3 Recent Progress

Image processing is being performed using ImageJ to refine and analyze existing radiography data. Radiography datasets are large, so the built in ImageJ scripting editor has been utilized to automatically process the data. Much of the early high-energy micro-focus experiments at AET-6 were performed with the same set up used during our previous synchrotron x-ray experiments. This set up consists of a steel bar with a rectangular window cut into it to aid in 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 entire captured image, as seen in **Fig. 32.1**. Using ImageJ, intensity differences in the image between the steel bar and the cut out window can be compared to select and extract only the lighter window where the experiment is being performed, as seen in the left image of **Fig. 32.2**. A single radiography experiment lasting only 20 minutes can contain thousands of images, so performing this process by hand would be extremely inefficient. Scripting in ImageJ allows for a written program, like the selection and extraction of an area of an image, to be carried out for an entire dataset automatically. Other image processing methods can be added to the script, such as a process in a solidification experiment that will take the first image in a sequence when the sample is completely liquid and subtract it from the following images, so that contrast in the image is improved by displaying the intensity of the image as the difference from the completely liquid phase. The improved contrast from this process is shown in the right image of **Fig. 32.2**.

32.4 Plans for Next Reporting Period

The CSM server currently has radiography data from previous solidification experiments. Selected datasets are being processed to understand microstructural evolution in metallic alloys during solidification. In the short term, the following tasks will be performed:

- Continued processing of two-dimensional radiography data from previous experiments;
- Improvement upon utilized processing techniques (e.g. adding false color to otherwise gray radiographs to highlight intensity differences, improve feature tracking between images, etc.);
- Begin image analysis by identifying important microstructural characteristics in processed data;
- Obtain more data from different radiography techniques to compare different radiography techniques, as seen for proton, synchrotron x-ray, and micro-focus x-ray radiography in **Fig. 32.3**;
- Train with LANL to perform micro-fcous x-ray radiography experiments and produce computed tomographic representations of samples like that seen in **Fig. 32.4**;
- Work with AET-6 to identify useful experiments involving static imaging of AM parts;
- Begin creating model-informing animations like the proton radiography-informed casting model in **Fig. 32.5**.

32.5 References

[32.1] P. J. Gibbs et al., Multiscale X-ray and proton imaging of bismuth-tin solidification, JOM 66 (2014) 1485–1492.

[32.2] K. A. Mohan et al., TIMBIR: A Method for Time-Space Reconstruction From Interlaced Views, IEEE Trans. Comput. Imaging 1 (2015) 96–111.

[32.3] A. J. Clarke et al., Microstructure selection in thin-sample directional solidification of an Al-Cu alloy: In situ X-ray imaging and phase-field simulations, Acta Mater. 129 (2017) 203–216.

[32.4] J. W. Gibbs et al., In Situ X-Ray Observations of Dendritic Fragmentation During Directional Solidification of a Sn-Bi Alloy, JOM 68 (2016) 170–177.

[32.5] B. M. Patterson, K. C. Henderson, P. J. Gibbs, S. D. Imhoff, and A. J. Clarke, Laboratory micro- and nanoscale X-ray tomographic investigation of Al-7 at.%Cu solidification structures, Mater. Charact. 95 (2014) 18–26.

[32.6] F. Merrill et al., Demonstration of Transmission High Energy Electron Microscopy, (2017).

32.6 Figure and Tables

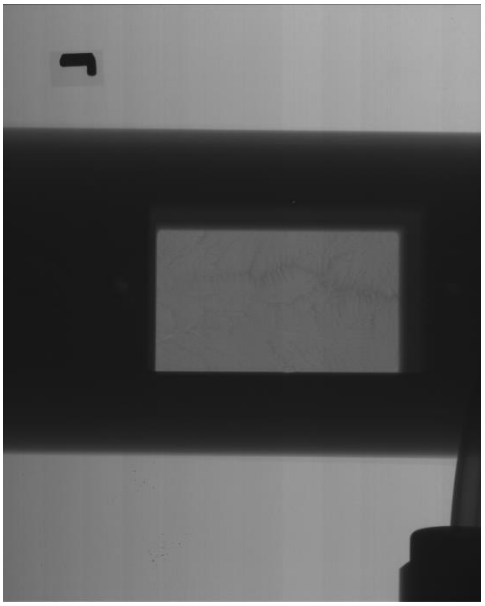

Figure 32.1: An unprocessed radiograph obtained using high-energy micro-focus x-ray radiography of a sample undergoing solidification. The experiment was performed using the synchrotron x-ray radiography setup, so the experiment is entirely contained in the small, light gray window in the center of the image. Laboratory x-ray imaging affords new opportunities to study larger fields-of-view.

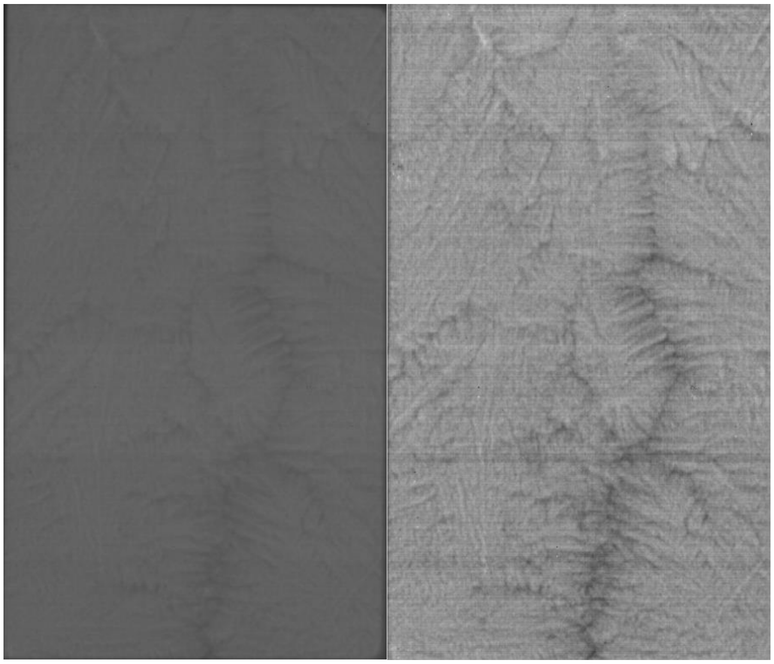

Figure 32.2: The darker radiograph (left) was processed from the image in **Fig. 32.1** by selecting the lower intensity pixels within the window and cropping the image to that selection. The lighter radiograph (right) was cropped using the same process, but was additionally processed by subtracting the first image in the radiography dataset that corresponds to the sample when it is completely liquid.

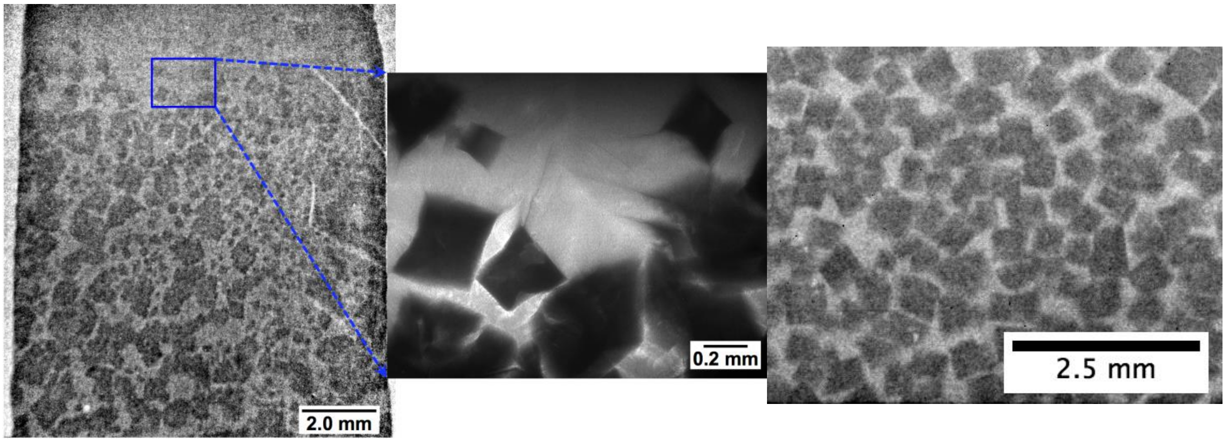

Figure 32.3: Proton (left) and synchrotron x-ray (center) radiographs showing the differences between the methods [32.1]. Synchrotron x-ray radiography exhibits a much higher spatial resolution, but proton radiography portrays a much larger field of view. Micro-focus laboratory x-ray radiograph (right) shows a good middle ground for spatial resolution and field of view [Clarke et al. unpublished].

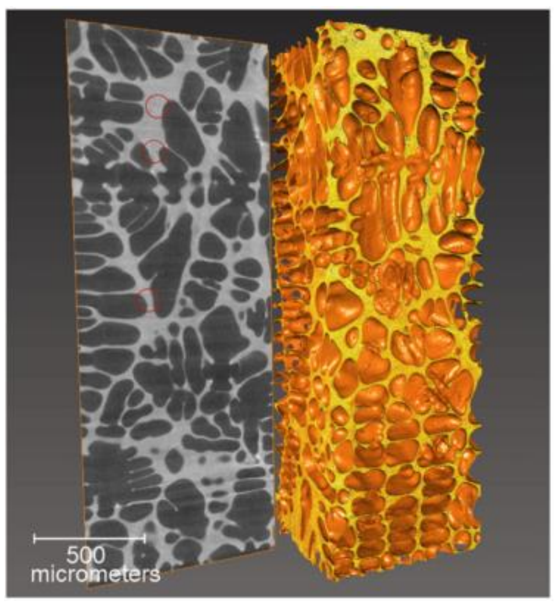

Figure 32.4: Three-dimensional tomographic reconstruction of an Al-Cu alloy from multiple radiographs. Reconstructions allow for manipulation of data unavailable from radiographs alone. The Al-rich primary dendrites in the reconstruction have been rendered translucent to better show the Cu-containing eutectic in orange [32.5].

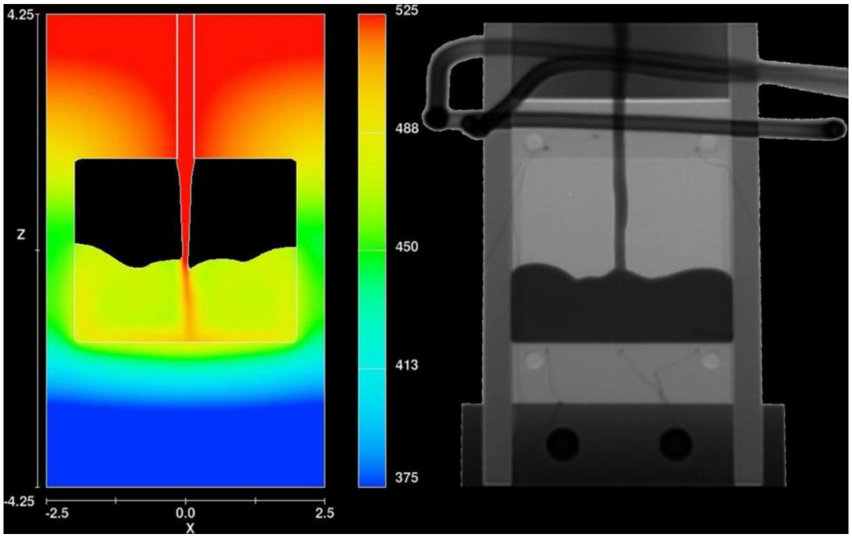

Figure 32.5: Casting model (left) informed by proton radiography of a Sn-Bi alloy (right). Further experiments and observation will bring model closer to reality [Clarke et al. unpublished].