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 observing the x-rays transmitted through the material. It can be used for post-mortem analysis, as well as in-situ imaging capable of capturing the development of microstructure during processing. This is important in the study of solidification. Dynamic imaging of microstructure evolution during processing (e.g. casting or directional solidifcation) can further our understanding of the mechanisms driving microstructural development, leading to improved predictive capabilities [32.1] . X-ray radiography can also be used to capture multiple images of a sample from different angles that can be reconstructed and combined into a three-dimensional representation. For instance, a reconconstruction of an additively manufactured (AM) metal sample can be generated to reveal internal defects in the part. Four-dimensional microscopy (three spatial and 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 an evolving, three-dimensional reconstruction. These reconstructions are created using techniques like Time-Interlaced Model-Based Iterative Reconstruction (TIMBIR) [32.2], which improves temporal resolution to capture material evolution during processing.

Synchrotron x-ray facilities, as well as laboratory micro-focus x-ray instruments, can be used for x-ray radiography. Synchrotron x-ray sources can be used to obtain high-spatial resolution images (on the order of 1 µm), however these images have a smaller field of view and require significant beam time at an often oversubscribed national user facility. High-energy micro-focus x-ray radiography in the laboratory has a 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 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 detector to collect that light. If experiments are to utilize tomographic methods of reconstruction that require images taken from different angles, a rotation stage for the sample is also necessary.

Multiple solidifiation 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 and solute segregation [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 are 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, allowing sample thicknesses on the order of millimeters and significantly larger fields-of-view. Proton radiography (pRad), in which 800 MeV energy protons are transmitted through samples 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 at the cost of somewhat 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 holds potential for high-Z materials.

32.2 Previous Work

Prior to the start of this project, in-situ solidification studies were performed at APS using synchrotron x-ray imaging and in laboratory settings in collaboration with AET-6 (non-destructive testing group at LANL) using high-energy micro-focus x-ray radiography. By analyzing these results, multi-scale solidification dynamics in metallic alloys can be captured. Data from these experiments is being uploaded to a Mines server to be processed and analyzed. One of the goals of this project is to establish a high-energy micro-focus x-ray cabinet at Mines for metallic alloy investigation to allow for further solidification, as well as AM related, experiments to be performed at ease. With the addition of this data, these different x-ray radiography techniques will be able to be further compared in the context of metallic alloys.

32.3 Recent Progress

An image processing method has been created to process data from experiments performed at APS and AET-6, and will be used to process new data once the micro-focus x-ray setup is running at Mines. This method, created using the script editor of ImageJ, consists of modular image processing scripts. Each script takes a directory of images from an experiment and performs a programmed process to enhance the images in some way, saving the results as separate files to preserve the original, unedited images. The modular nature of these scripts allows for the processing steps to be reperformed in different sequences on the original images, adding to or changing the image processing steps to achieve different results. High-energy micro-focus solidification experiments at AET-6 were performed with the same set up used during 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 the directional solidification of an Al-Ag alloy in **Figure 32.1a**. This experiment will be used to demonstrate the current processing abilities of the modular image processing method. The first script extracts the lighter portion of the radiographs from these experiments by thresholding the pixel values in the image corresponding to the steel bar and the cut out window, then selects and extracts only the lighter window where the experiment is being performed, as seen in **Figure 32.1b**. The result of the first script retains a partial border of the darker steel bar, due to the not completely rectangular area selected by the thresholding operation. Another script can remove a set amount of pixels from each side of the images, therefore allowing for better contrast within the desired subject area, the solidification structure. The result of this script can be seen in **Figure 32.2a.** While this image exhibits better contrast between the dendrites and the interdendritic regions, there are still artifacts from the imaging process represented as vertical and horizontal banding. This can be adjusted by means of a script that performs a fast Fourier transform, and filters out repeated structures, such as the banding. The result of this script can be seen in **Figure 32.2b.** A single radiography experiment lasting only 20 minutes can contain thousands of images, so performing these processes through programs is much more efficient than by hand. Other image processing methods can be added to this modular image processing method by the addition of more scripts. Once a sequence of processing scripts is worked out that fulfils the processing needs for analysis of individual experiments, the module can be combined into a single, comprehensive script.

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 refining of modular image processing of two-dimensional radiography data from previous experiments;
- Improvement upon utilized processing techniques (e.g. fine-tune fast Fourier transform filtering, adjust psuedocolor mappings to better reflect microstructural features);
- Quantitatively analyze microstructural features from solidifcation experiments in processed data (e.g. solidification velocity, pixel values corresponding to local solute variations);
- Finalize refurbishment and transfer of micro-focus x-ray cabinet to Mines;

- 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 **Figure 32.3**;
- Train with LANL to perform micro-focus x-ray radiography experiments and produce computed tomographic representations of samples like that seen in **Figure 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 **Figure 32.5**.

32.5 References

[32.1] P.J. Gibbs, S.D. Imhoff, C.L. Morris, F.E. Merrill, C.H. Wilde, P. Nedrow, F.G. Mariam, K. Fezzaa, W.-K. Lee, A.J. Clarke, Multiscale X-ray and proton imaging of bismuth-tin solidification, JOM, 20114, 66(8):1485–1492

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[32.3] A.J. Clarke, D. Tourret, Y. Song, S.D. Imhoff, P.J. Gibbs, J.W. Gibbs, K. Fezzaa, A. Karma, "Microstructure selection in thin-sample directional solidification of an Al-Cu alloy: in situ x-ray imaging and phase-field simulations", Acta Materialia, 2017, 129:203-216

[32.4] J.W. Gibbs, D. Tourret, P.J. Gibbs, S.D. Imhoff, M.J. Gibbs, B.A. Walker, K. Fezzaa, A.J. Clarke, "In situ x-ray observations of dendritic fragmentation during directional solidification of a Sn-Bi alloy", JOM, 2016, 68(1):170-177

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

[32.6] F.E. Merrill, J. Goett, J.W. Gibbs, S.D. Imhoff, F.G. Mariam, C.L. Morris, L.P. Neukirch, J. Perry, D. Poulson, R. Simpson, P.L. Volegov, P.L. Walstrom, C.H. Wilde, C. Hast, K. Jobe, T. Smith, U. Wienands, A.J. Clarke, D. Tourret, "Demonstration of transmission high energy electron microscopy, Applied Physics Letters, 2018, 112:144103

32.6 Figure and Tables

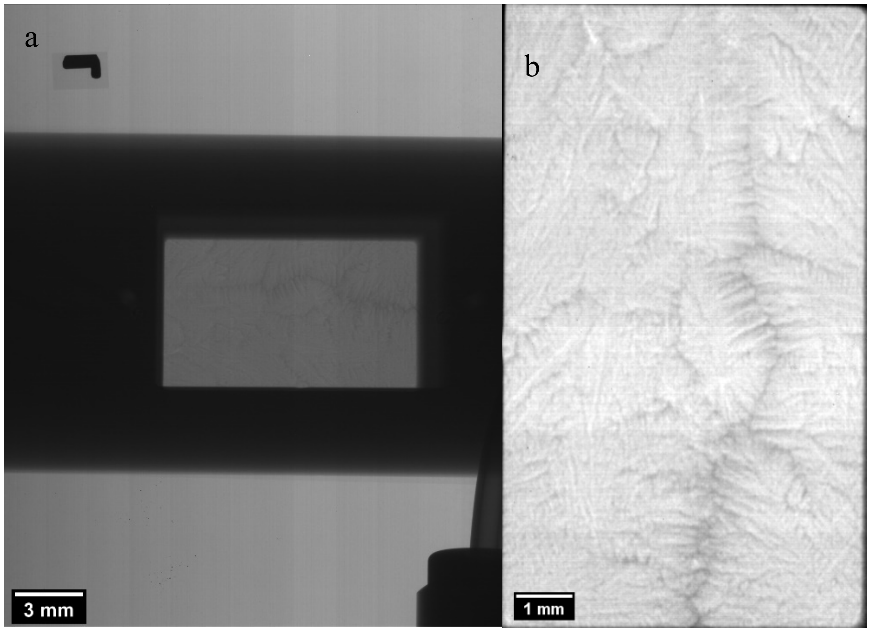

Figure 32.1: A frame from the solidification process of an Al-Ag alloy obtained using high-energy micro-focus x-ray radiography of a sample undergoing directional solidification. The experiment was performed using the synchrotron x-ray radiography setup, so the experiment is entirely contained in the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

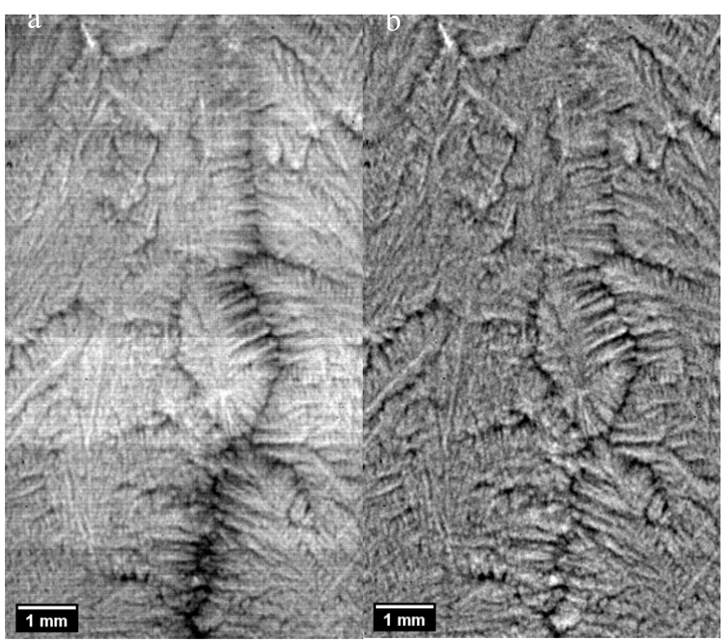

Figure 32.2: Further processing of the same frame of Al-Ag solidification shown in **Figure 32.1** by (a) trimming 10 pixels from each edge of the radiograph, allowing for better contrast in the solidification structure, and (b) performing fast Fourier transform filtering to remove horizontal and vertical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

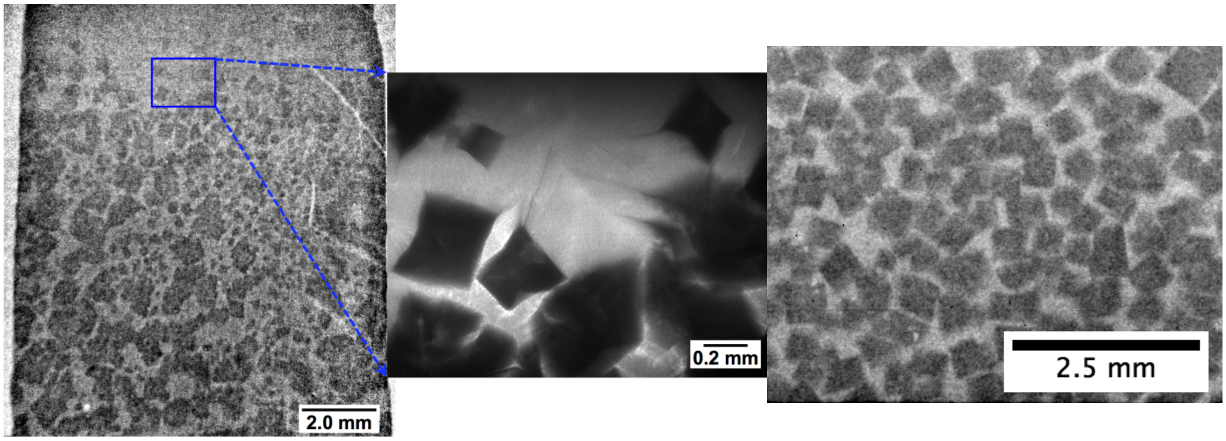

Figure 32.3: Proton (left) and synchrotron x-ray (center) radiographs showing the differences between the methods [32.1]. Synchrotron x-ray radiography exhibits a much higher spatial resolution, but proton radiography portrays a much larger field of view. Micro-focus laboratory x-ray radiograph (right)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

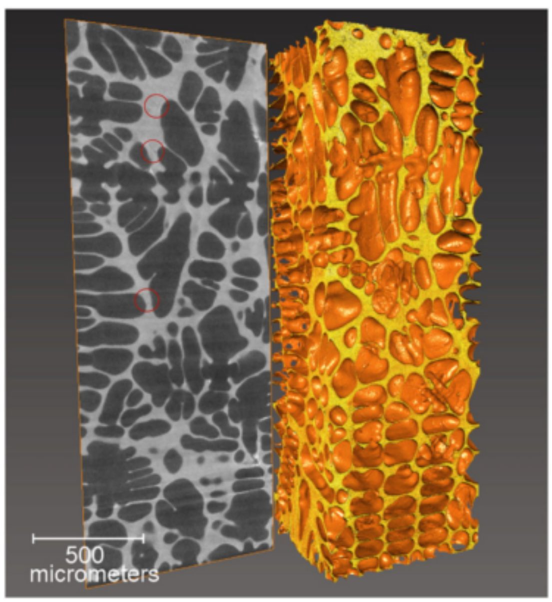

Figure 32.4: Three-dimensional tomographic reconstruction of an Al-Cu alloy from multiple radiographs. Reconstructions allow for manipulation of data unavailable from radiographs alone. The Al-rich primary dendrites in the reconstruction have been rendered translucent to better show the Cu-containing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

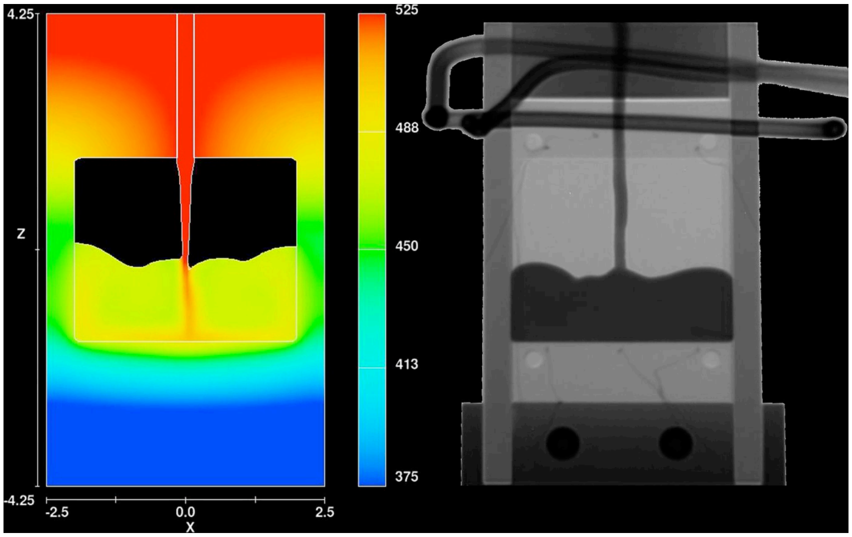

Figure 32.5: Casting model (left) informed by proton radiography of a Sn-Bi alloy (right). Further experiments and observation will bring the modeling closer to reality [A.J. Clarke et al., unpublished research].