ORNL/TM-2023/3016

Report Outlining Computed Tomography Strategy and Microscopy Approach to Qualifying AM 316 Materials

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August 2023

M3CR-22OR0406012

OAK RIDGE National Laboratory

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ORNL/TM-2023/3016

Advanced Materials and Manufacturing Technologies Program

REPORT OUTLINING COMPUTED TOMOGRAPHY STRATEGY AND MICROSCOPY APPROACH TO QUALIFYING AM 316 MATERIALS

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August 2023

M3CR-22OR0406012

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UT-BATTELLE LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

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ABSTRACT

This report is part of work package CR-22OR0406012, *Automated, High-Throughput Materials Characterization Techniques*, under the Advanced Materials and Manufacturing Technologies program (AMMT). The project's primary objective is to leverage our AI-based rapid and high-throughput automated characterization framework to qualify additively manufactured 316 materials comprehensively, focusing on optimizing the additive manufacturing process and evaluating the performance of 3D-printed stainless steel components. This report outlines our strategy for leveraging the automated characterization process for qualifying 316H materials.

1. BACKGROUND

In phase 1 of this project, we procured two state-of-the-art systems, the Zeiss Metrotom 800 x-ray computed tomography (XCT) system and the Gemini 450 scanning electron microscopy (SEM) system. The Metrotom 800 XCT system enables nondestructive characterization of 3D-printed components. This system enables investigation of the effects of various additive manufacturing process parameters (such as power, velocity, and composition) on the quality of printed components. The Gemini 450 SEM system aids in identifying microstructural features that affect performance, including grain size, phase distribution, and porosity.

The XCT and SEM systems were employed in a rapid automated high-throughput characterization framework for characterization of more than 700 coupons printed with 316L and 316H using Concept Laser's M2 and Renishaw AM400 laser powder bed fusion 3D-printing systems at the Manufacturing Demonstration Facility (MDF). Figure 1 summarizes how the automated high-throughput characterization process was used in collaboration with the other work packages under the Advanced Materials and Manufacturing Technologies (AMMT) program to enhance our understanding of process-structure-property-performance relationships, and Figure 2 provides an overview of the automated high-throughput characterization process.

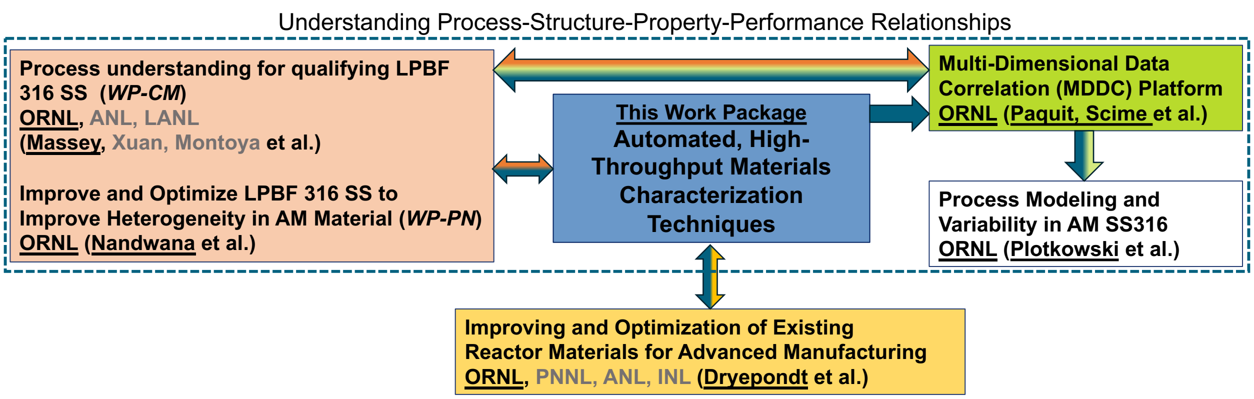

Figure 1. Automated high-throughput characterization and its application in collaboration with work packages under the AMMT program. Abbreviations are Oak Ridge National Laboratory (ORNL), Argonne National Laboratory (ANL), Los Alamos National Laboratory (LANL), additively manufactured (AM), and laser powder bed fusion (LPBF).

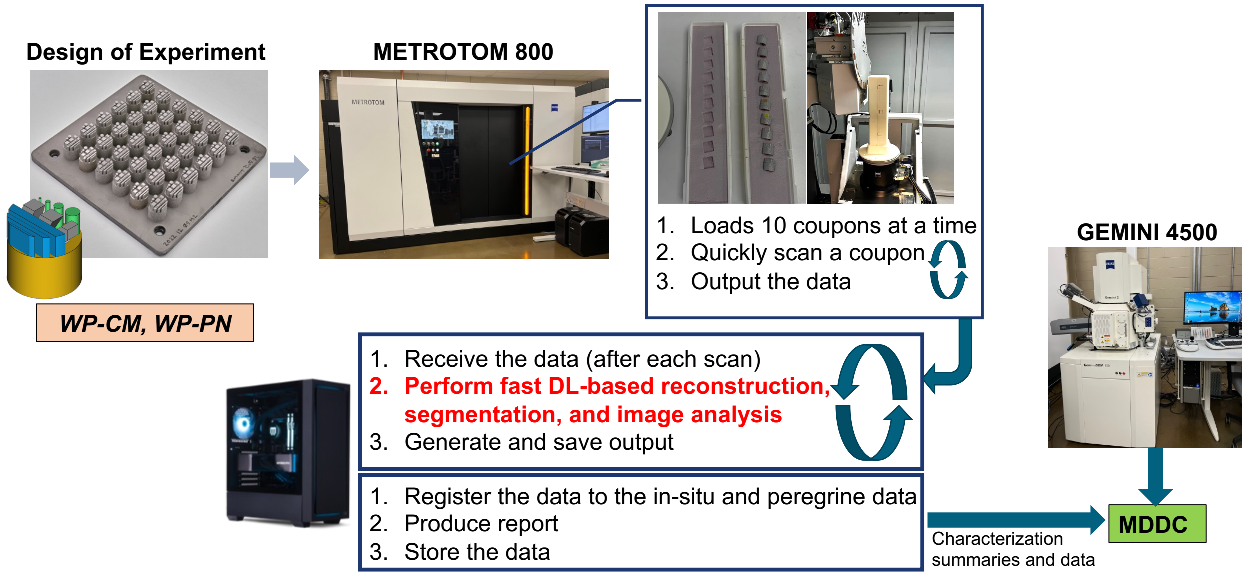

Figure 2. Breakdown of the high-throughput rapid automated characterization process developed in phase 1 of this work package in the AMMT program. Abbreviations are deep learning (DL) and Multi-Dimensional Data Correlation (MDDC).

In phase 1, we developed and fine-tuned our AI-based algorithms (i.e., Simurgh image reconstruction and Kakapo image segmentation) for characterizing hundreds of parts rapidly scanned using the XCT systems at the MDF. In addition, we worked with other work packages, particularly with the digital team, to integrate the outputs from characterization into the Multi-Dimensional Data Correlation (MDDC) framework, which connects the data to other work packages such as the modeling work package to enable a better understanding of the process-structure-property-performance relationships. This enabled the creation of a large pedigree data set for phase 2 of the project.

At a high level, the plan in phase 2 is to leverage the rapid automated characterization, pedigree data, and lessons learned from phase 1 (FY 2022–FY 2023) as well as a meticulous experiment design to find a high confidence window of optimum process parameters for printing fully dense 316 stainless steel (SS). This will include not only identifying the optimum process parameters for different geometric features within the ZEISS ParAM coupons but also finding the window of optimum process parameters that allows us to print a part with varying process parameters while ensuring the transition regions are free of flaws. The expected outcome is a full build plate of printed parts that meet our density and performance expectations and a comprehensive database of XCT and microscopy data.

2. STRATEGY DESCRIPTION

In the project, we will determine the optimal process parameter window for SS 316H on a specific laser powder bed fusion (LPBF) printer, the M2 Concept Laser printer at the MDF. The process parameter windows will be sensitive to local part geometries and the location on the build plate. The quality of each Zeiss ParAM coupon will be determined primarily using XCT.

As part of the AMMT program, on the order of 5,000 coupons will be printed and characterized using our automated characterization framework, and the data and analysis output will be shared on the MDDC framework. The results of high-throughput automated characterization—along with characterization techniques including SEM and electron backscatter diffraction (EBSD), in-situ monitoring data available on the MDDC framework, and machine learning and other thermal simulation tools to be provided by the modeling team—will be used to determine the optimum process parameter window while considering various process parameters. This will encompass various parameters ranging from laser power, speed, and hatch spacing to stripe and contour parameters within each geometrical feature in the parts. Furthermore, we aim to tackle more intricate scenarios where we intend to print fully dense parts with varying process parameters in different regions.

Seven tasks comprise our strategy:

1. Calibrating the M2 Concept Laser printer's spot size using inverse problem-solving
2. Identifying a primary processing window by analyzing a broad range of conditions via XCT scans
3. Refining the processing window by optimizing secondary parameters
4. Determining optimal contour parameters to reduce surface porosity
5. Training and deploying machine-learning models to scrutinize the vast data sets, aiming for accurate interpolations and predictive capabilities in the high-dimensional LPBF processing space
6. Identifying challenges in printing diverse geometries, emphasizing the transition between different sections of the coupons
7. Printing fully dense coupons with varying, geometry-sensitive process parameters on a single build plate

The following sections describe each of these tasks planned for this work package.

2.1 TASK 1: CALIBRATE THE SPOT SIZE FOR THE M2 CONCEPT LASER PRINTER USING AN INVERSE PROBLEM APPROACH

In this task, efforts within the process modeling and variability in AM SS316 work, specifically calibration of Myna's melt pool thermal modeling, will be leveraged to determine the M2 Concept Laser printer's true laser spot size and power measurements. Currently, the M2 Concept Laser 3D printer at the MDF is being used primarily for printing the 316H coupons for the AMMT program. However, no access is provided to calibrate (i.e., to measure the actual laser power and spot size) the laser source. On the other hand, we were able to calibrate the laser source for the EOSM290 system. In collaboration with the modeling team, an experiment has been designed to enable the calibration of the M2 Concept Laser beam.

First, single-bead weld tracks will be printed on top of pads of printed material in the EOSM290 3D printing system using a wide range of processing conditions. These weld tracks will be cross-sectioned, etched, and imaged with an optical microscope so that their dimensions (width and depth) can be extracted. Using these measurements and the true laser power and spot size, the modeling team will calibrate their models for accurate prediction of melt pool shape (depth and width). Then, a new set of single tracks across a predefined range of processing conditions will be performed on the M2 Concept Laser system, for which the true laser power and spot size are not known. These weld tracks will go through the same steps noted above so that their melt pool shapes will be extracted. Finally, the modeling and the melt pool characteristics will be used to estimate the true laser power and spot size for the M2 system.

2.2 TASK 2: IDENTIFY THE VIABLE PROCESSING WINDOW BY PRINTING AND XCT SCANNING COUPONS PRINTED ACROSS A WIDE RANGE OF PRIMARY PROCESSING CONDITIONS

The objective of this task is to identify the viable processing window with primary process parameters—that is, laser power, velocity, spot size, hatch spacing, layer thickness, and location combinations—over which SS 316H can be printed with minimal void-type flaws.

In this task, the workflow developed in Task 1 and the pedigree data from coupons printed in the current fiscal year will be used to preselect a set of 2,000 unique combinations of laser power, velocity, spot size, hatch spacing, layer thickness, and location combinations. Coupons will be printed out of SS 316H powder using each of these process parameter combinations and will then be characterized using our automated characterization process. The XCT results and any microscopy data (on an as-needed basis) will be registered in Peregrine and uploaded to the MDDC framework. Data analysis will be performed to determine the viable processing window over which SS 316H can be printed with minimal void-type flaws; the wider the final processing window, the more freedom can be exercised when tailoring local microstructures. As a result, we will deliver a set of geometry- and location-sensitive process maps describing the viable LPBF processing window for SS 316H. Further, measured porosity fractions for 2,000 Zeiss ParAM coupons printed across LPBF processing space and processing windows delineating regions with less than 2% porosity will be available on the MDDC framework.

2.3 TASK 3: REFINE THE PROCESSING WINDOWS BY OPTIMIZING SECONDARY PARAMETERS

The objective of this task is to determine the stripe width and stripe overlap parameters for each point in the viable LPBF SS 316H processing window such that void-type flaws are minimized. A subset of the original 2,000 process parameter combinations will be used to print new coupons while varying the stripe width and stripe overlap parameters. Again, each Zeiss ParAM coupon will be XCT scanned to measure the porosity content. The XCT results and any microscopy data (on an as-needed basis) will be registered in Peregrine and uploaded to the MDDC framework. This will enable a set of optimal stripe width and stripe overlap values to be determined for each point in the viable LPBF SS 316H processing window. Furthermore, the measured porosity fractions of 500 coupons printed across the viable processing windows using varying stripe parameters and tables of optimal stripe parameters at each point in the processing windows will be available on the MDDC framework.

2.4 TASK 4: DETERMINE THE OPTIMAL CONTOUR PARAMETERS

The objective of this task is to determine the contour process parameters that minimize near-surface porosity and to calculate the CAD offset values required to print dimensionally accurate parts using each viable contour parameter set.

To that end, in this task, 500 coupons will be printed with a small set of optimized raster and stripe parameters. Contours for each coupon will be printed with different combinations of laser power, spot size, velocity, and raster overlap parameters. Each Zeiss ParAM coupon will be XCT scanned to measure the porosity content. Once a subset of optimal contour parameters has been determined, a small number of additional experiments will be performed to calculate the required CAD offset value for each parameter set. The XCT results and any microscopy data (on an as-needed basis) will be registered in Peregrine and uploaded to the MDDC framework.

As a result, a set of optimal contour parameters and corresponding CAD offset values for 316H will be delivered. In addition, the measured porosity fractions for 500 coupons printed with varying contour parameters and the tables of optimal contour parameters and CAD offsets will be available on the MDDC framework.

2.5 TASK 5: TRAIN AND DEPLOY MACHINE-LEARNING MODELS TO FACILITATE EXPLORATION OF MASSIVE DATA SETS CONTAINING PROCESS PARAMETERS

AND XCT-BASED PART QUALITY METRICS AS WELL AS MACROSTRUCTURAL FEATURES

The primary objective of this task is to develop a narrow-focus deep-learning model to facilitate the exploration of the very high-dimensional LPBF processing space for SS 316H.

We will develop a deep-learning approach for exploring the high-dimensional, nonlinear LPBF processing space. We will use these trained models to interpolate between empirical data points and create dense processing maps and to solve the inverse problem—that is, to determine the optimal processing conditions based on desired material properties. Gradient-boosting decision trees, Bayesian optimization techniques, and more advanced machine-learning algorithms will also be used explore process space. In particular, generative adversarial networks could be beneficial for generating new, synthetic instances of data that can resemble the original data and thus for interpolating between empirical data points to create dense processing maps.

We will deliver a trained model and its corresponding dense process map outputs for LPBF-processed SS 316H. To test the algorithm, we will use it to predict the lack-of-fusion porosity content at 100 discrete test points spread across LPBF SS 316H processing space with an average error of less than 10% relative to the experimental XCT flaw segmentation results. Ideally, using these data and algorithms, a separate "optimum" process parameter window for each geometrical feature in the coupons will be found.

2.6 TASK 6: IDENTIFY THE CHALLENGES WHEN PRINTING THE COUPONS WITH VARIOUS PROCESS PARAMETERS DEPENDING ON THE GEOMETRY OF THE PARTS

The objective of this task is to determine the process parameter window for printing dense parts with varying (geometry-based) process parameters both within each geometrical feature and in the transition region between base and the overhangs at the top.

In this task, we will use the optimized process parameters obtained in the previous tasks to print parts with varying process parameters. The bottom cylindrical portion of the parts (an example is shown in Figure 2) will have a separate optimum process parameter window compared with overhangs at the top (rods, thin walls, and inclines). However, one cannot expect to print parts with varying process parameters without optimizing the region of transition between the top and bottom parts. To that end, we will use the information from the machine-learning algorithm in the prior task and the rest of the data to predict the transition region process parameters and print 240 new coupons with varying process parameters between the base cylinders and the top overhangs, which will allow us to understand the process parameter window for printing parts with varying process parameters. This will enable determination of a set of process parameters for printing dense coupons with varying process parameters. Further, the measured porosity fractions of the 240 printed coupons and tables of optimal process parameters will be available on the MDDC framework.

2.7 TASK 7: PRINT BUILD PLATES WITH "FULLY DENSE" COUPONS WITH OUR PREDICATED REGION-SPECIFIC PROCESS PARAMETER WINDOWS

The objective is to print 5 build plates (each 20 coupons) as follows.

1. Full dense base
2. Fully dense inclines
3. Fully dense walls

4. Fully dense rods
5. Fully dense coupons printed with variable process parameters for base an overhang, and for the transition region

It should be noted that “fully dense” does not mean 0% defect on the entire build plate, but less than 0.02% flows are expected on average over the entire build plate.

3. OTHER CONSIDERATIONS

3.1 DATA GENERATED UNDER OTHER WORK PACKAGES

Although we aim to design the experiment step by step starting by calibrating the laser source in the printing system, we will be cognizant of the other efforts and work packages under the AMMT program. To that end, we will coordinate with our colleagues from other work packages so that the design of experiments will include lessons learned and feedback from other teams. This coordination can also provide value to other work packages and avoid duplicates throughout the process.

3.2 MATERIAL PROPERTIES CHARACTERIZATION

To have a holistic understanding of SS 316H’s performance after additive manufacturing, we will incorporate tests to assess mechanical properties like tensile strength and fatigue resistance. This will augment our structural insights with functional performance metrics.

3.3 ADVANCED CHARACTERIZATION TECHNIQUES

Beyond our current characterization tools, we will integrate techniques like EBSD for nuanced crystallographic analysis and perhaps even transmission electron microscopy for an in-depth look into the microstructures.

4. CONCLUSIONS

This report outlined the plan and individual tasks to qualify AM 316 materials comprehensively, focusing on optimizing the additive manufacturing process and evaluating the performance of 3D-printed stainless steel components.

Upon completion of this project, we will have established a comprehensive understanding of the optimal processing parameters for SS 316H using the M2 Concept Laser printer at the MDF. Through rigorous testing and analysis spanning from calibration to machine learning, we have designed a strategy to produce high-quality prints that consider part geometries and build plate location. Using XCT scans and supplementary techniques, we have been able to harness vast data sets to fine-tune our parameters. Our ultimate goal is to be able to print consistently dense coupons, adapting parameters in real time based on geometry. This approach promises to elevate the quality and consistency of LPBF-produced parts and to offer tangible benefits to the broader manufacturing industry.