AMB2025-01 Benchmark Measurements and Challenge Problems

Last updated: 03/05/2025

Modelers are invited to submit challenge problem simulation results before the deadline of 23:59 (ET) on August 29, 2025. There are no restrictions on what challenge problems are attempted. For each set of benchmarks, a downloadable .pdf file is provided that describes all the measurements and challenge problems. Tabulated results using challenge-specific templates are required for most challenges. Because some participants may not be able to share proprietary details of the modeling approaches used, we are not requiring such details. However, whenever possible we strongly encourage participants to include with their submissions a .pdf document describing the modeling approaches, physical parameters, and assumptions used for the submitted simulations.

All evaluations of submitted modeling results will be conducted by the AM Bench 2025 Organizing Committee in conjunction with the relevant AM Bench 2025 measurement teams. Award plaques will be awarded at the discretion of the Organizing Committee.

If you are interested in following or participating in any of the AM Bench 2025 challenge problems, please email us at* ambench@nist.gov *so we can add you to our contact list. This will allow us to inform you if any updates are made.

AMB2025-01: Laser powder bed fusion 3D builds of nickel-based superalloy 625 with variations in feedstock chemistries. Detailed descriptions are found below, and simulation results may be submitted to ambench@nist.gov.

Challenge Problems

- **Benchmark Challenge CHAL-AMB2025-01-SR:** Predict the average solidification cell size, average maximum and minimum segregated mass fractions of Nb and Mo at the cell walls and cell interiors, respectively, and the volume fractions of precipitates, excluding oxides, in the as-built microstructures. Predict the volume fractions of precipitates, excluding oxides, in the microstructures after stress relief heat treatment at 870 °C for 1 h.
- **Benchmark Challenge CHAL-AMB2025-01-H:** Predict the average solidification cell size, average maximum and minimum segregated mass fractions of Nb and Mo at the cell walls and cell interiors, respectively, and the volume fractions of precipitates, excluding oxides, in the as-built microstructures. Predict the volume fractions of precipitates, excluding oxides, in the microstructures after homogenization heat treatment at 1150 °C for 1 h.

Document outline:

1. Overview and Basic Objectives

The AMB2025-01 tests consist of multiple laser powder bed fusion (LPBF) 3D builds of nickel superalloy 625 with variations in powder feedstock. The primary objectives of AMB2025-01 are to investigate the as-built microstructure features and track the microstructural evolution when parts are subjected to stress relief and homogenization heat treatments. The microstructure features of interest are at the sub-grain level and include solidification cell size, elemental segregation, types of precipitates and their volume fractions.

2. Experimental Description

2.1 Substrate material and dimensions

All nickel superalloy 625 builds are deposited on similar 36 mm thick 1045 steel substrates (build plates). The total build area of each build plate is approximately 252 mm x 252 mm, and each substrate was fixed to the build platform by screws.

2.2 Part geometries and build layout

All parts were chosen to have simple geometries consisting of either rectangular or square cross sections with respect to the build height. Five different geometries were built onto each build plate with the following nominal cross-sectional dimensions:

- 75 mm x 25 mm (6 parts)
- 150 mm x 15 mm (3 parts)
- 55 mm x 10 mm (6 parts)
- 25 mm x 25 mm (6 parts)
- 15 mm x 15 mm (15 parts)

Each part was labeled with the format of 'XX.Y', where 'XX' refers to the longest cross-sectional dimension of the part and 'Y' refers to the arbitrary labeling of the part number. A schematic diagram of the build layout is shown in Figure 1, with the part labels overlayed. For the AMB2025-01 challenge problems, only the 15 mm x 15 mm cubes were used.

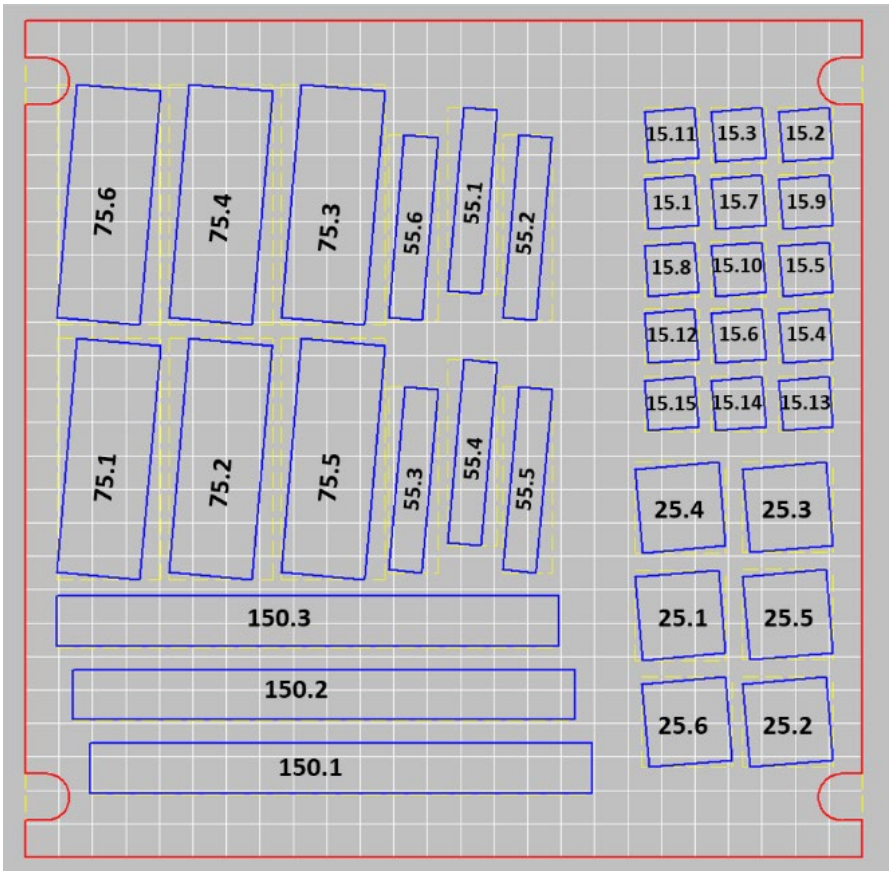

Figure 1. A schematic diagram of the build layout with sample labels overlayed with the sample geometries. The red outline corresponds to the build plate, while the blue outlines correspond to the part boundaries.

2.3 Deposition strategy and parameters

Since the goal of AMB2025-01 is to investigate differences in microstructure owing to variations in powder feedstock composition, all builds were completed with identical processing parameters on an EOS M290[i], hereafter referred to as the commercial build machine (CBM). A list of build parameters is given in Table 1. The parts were deposited onto the build plate using standard alloy 625 parameters provided by the CBM. A stripe deposition strategy was used to help alleviate part distortion during building. The laser was scanned in a bidirectional manner along the stripe width. After the completion of a layer, the scanning direction of the subsequent layer was rotated 67°.

Table 1. A summary of the deposition parameters used for each build plate.

Parameter	Setting or Measurement
Laser power	285 W
Laser scan speed	960 mm/s
Laser spot diameter	(72 +8/-4) µm*
Layer thickness	0.040 mm
Hatch spacing	0.11 mm
Stripe width	10.0 mm
Stripe overlap	0.08 mm
Scan strategy	67° Rotation every layer
Platform heating	80 °C
Maximum oxygen level	0.1 % by volume
Protective gas	Argon
Recoating arm speed	150 mm/s

*Type B uncertainty with a 95 % confidence interval

Material was deposited onto each build plate until the build process was canceled due to insufficient remaining powder. Since different quantities of powder were acquired due to availability, the final build height varied amongst builds. Table 2 lists the number of layers deposited and the corresponding build height for each build. The build process for Build 1 was interrupted due to a machine error after 108 layers, corresponding to a build height of 4.32 mm. The machine operator manually lowered to build platform by the appropriate layer thickness (0.040 mm), spread the next powder layer, and restarted the automatic build process at layer 109. All other builds were completed with no stoppage.

Table 2. The total number of layers and build height for each nickel superalloy 625 build.

Build Number	Number of Layers	Build Height
1	745	29.80 mm
2	612	24.48 mm
3	476	19.04 mm

2.4 Powder feedstock

Gas-atomized powders were acquired from different vendors in various quantities depending on powder availability. Powder samples were collected immediately after opening the powder containers and sealed in glass vials. The samples were analyzed for chemical composition by a certified testing laboratory. Most elements (Al, B, Co, Cr, Cu, Fe, Mn, Mo, Nb, Ni, P, Si, Si, Ta, Ti) were measured using the inductively coupled plasma method with atomic emission spectroscopy (ICP-AES) according to ASTM E1479. Nitrogen and oxygen were measured by inert gas fusion, while carbon and sulfur were measured by

combustion analysis according to ASTM E1019. The reported chemical composition for each powder is listed in Table 3.

Table 3. The measured chemical composition in mass fraction (%) of powder samples. The '<' symbol refers to a mass fraction that was below the detection limit of the technique.

Element	Build 1	Build 2	Build 3
Al	0.20	0.28	< 0.001
B	0.001	0.001	0.003
C	0.060	0.007	0.009
Co	0.020	0.043	0.051
Cr	21.22	20.90	22.00
Cu	0.017	0.017	0.020
Fe	4.01	0.64	0.12
Mn	0.15	0.058	0.33
Mo	9.05	8.76	9.29
N	0.008	0.004	0.072
Nb	3.81	3.89	3.67
Ni	61.13	64.97	63.90
O	0.007	0.012	0.051
P	0.001	< 0.001	0.006
S	0.001	0.002	0.001
Si	0.28	0.096	0.44
Ta	< 0.001	< 0.001	< 0.001
Ti	0.013	0.31	0.004

2.5 Solid samples

Upon build completion, all parts with labels of '15.Y' and '25.Y' were removed from the respective build plate in the as-built condition using electrical discharge machining (EDM). The 15.2 parts from each build plate were selected for chemical composition analysis using identical procedures as the analysis for powder samples. The reported chemical composition for each solid sample is listed in Table 4.

Table 4. The measured chemical composition in mass fraction (%) of solid samples. The '<' symbol refers to a mass fraction that was below the detection limit of the technique.

Element	Build 1	Build 2	Build 3
Al	0.22	0.29	< 0.001
B	0.001	0.001	0.003
C	0.058	0.006	0.008
Co	0.019	0.043	0.050
Cr	21.02	20.72	21.90

Cu	0.020	0.019	0.020
Fe	4.35	0.65	0.11
Mn	0.15	0.058	0.30
Mo	9.12	9.02	9.36
N	0.009	0.004	0.067
Nb	3.83	4.00	3.70
Ni	60.87	64.78	63.96
O	0.006	0.010	0.047
P	< 0.001	< 0.001	0.006
S	0.001	0.001	0.001
Si	0.30	0.086	0.42
Ta	< 0.001	< 0.001	< 0.001
Ti	0.014	0.32	0.003

Figure 2 shows a photograph of a completed build with the '15.Y' and '25.Y' parts removed. A small portion measuring approximately 1.2 mm to 1.4 mm of each part remained on the build plate after EDM removal. Table 5 compares the part height of each build before and after removal from the substrate.

Table 5. A comparison of the measured part heights before and after removal from the build plates by EDM.

Build Number	Height before removal	Height after removal
1	29.80 mm	28.58 mm
2	24.48 mm	23.10 mm
3	19.04 mm	17.73 mm

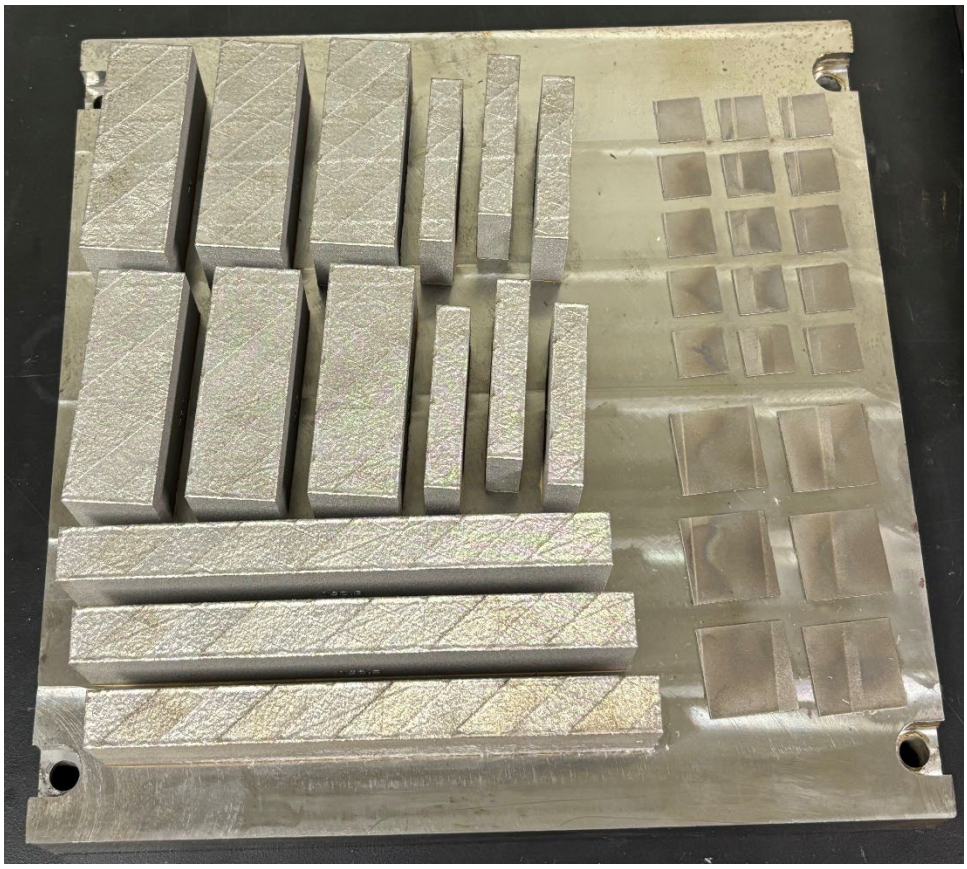

Figure 2. A photograph of a completed build plate with the '15.Y' and '25.Y' parts removed by EDM.

3. Measurement Descriptions

3.1 Heat Treatment and Sample Sectioning

The 15.4 parts were subjected to a stress relief heat treatment at 870 °C for 1 h, and the 15.5 parts were subjected to homogenization heat treatment at 1150 °C for 1 h. These parts from each build plate were vacuum encapsulated in a quartz tube to protect from atmospheric contamination during heat treatment. Each quartz tube was then placed into the hot zone of a tube furnace with a temperature deviation of approximately ± 1 °C, confirmed with a type K thermocouple. Upon placement of the quartz tube, the temperature of the hot zone decreased by about 3 °C to 5 °C. The furnace temperature gradually increased to the temperature setting after approximately 270 s to 300 s, at which point the 1 h timer began. After 1 h, the encapsulated samples were removed from the furnace and placed under flowing water. Samples could be handled safely after approximately 10 min, at which point the quartz tubes were broken and the samples were removed.

Parts 15.13 (as-built), 15.4 (stress-relieved) and 15.5 (homogenized) from each build plate were sectioned for characterization using a cubic boron nitride metal-bonded wafering blade operated at 1500 rpm. The samples were first sectioned parallel to the build direction at various build heights as described in Figure 3. Approximately 4.5 mm measuring from the EDM surfaces were first removed to avoid material affected by the stoppage that occurred when building Build 1. These sections are labeled as Section A for all investigated parts. Section B was then removed by making a parallel cut approximately 10 mm from the previous cut for Build 1 and Build 2, and approximately 7.5 mm for Build 3 due to the limited build height. Section thicknesses with respect to the build height were measured using a micrometer and the results are shown in Table 6. All microstructural characterization was performed on material from Section B and Section C, and the remaining Section A and Section D were preserved for future studies.

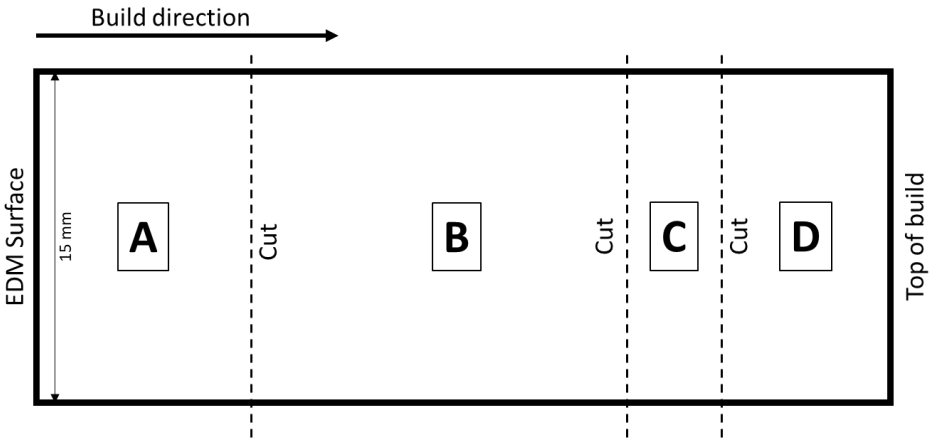

Figure 3. A schematic diagram for the initial sectioning of 15.Y parts along the build height. Note the sketch is not drawn to scale.

Table 6. A summary of the measured section thicknesses in mm for parts 15.13, 15.4, and 15.5 for each build and material condition corresponding to Figure 3.

Build number	Condition	Part Number	Section A	Section B	Section C	Section D
1	As-built	15.13	4.488	10.285	2.008	10.591
2	As-built	15.13	4.585	10.260	1.990	5.135
3	As-built	15.13	4.589	7.541	2.669	1.746
1	870 °C/ 1 h	15.4	4.272	10.153	1.852	10.973
2	870 °C/ 1 h	15.4	4.583	10.060	1.754	5.544
3	870 °C/ 1 h	15.4	4.273	7.075	1.810	3.355
1	1150 °C/ 1 h	15.5	4.311	10.214	1.873	10.994
2	1150 °C/ 1 h	15.5	4.248	10.116	1.831	5.857
3	1150 °C/ 1 h	15.5	4.100	6.787	1.926	3.865

Section C required no further preparation and served as an approximately 15 mm x 15 mm XY cross section (where Z refers to the build direction) for microstructure examination. Section B of each part was further downsized by sectioning additional planes parallel to the build direction as shown in Figure 4. The first cut was made perpendicular to the chosen y-axis of the sample to reveal the XZ sample plane labeled as Section 1, followed by a second cut perpendicular to the x-axis to reveal the YZ plane labeled as Section 2.

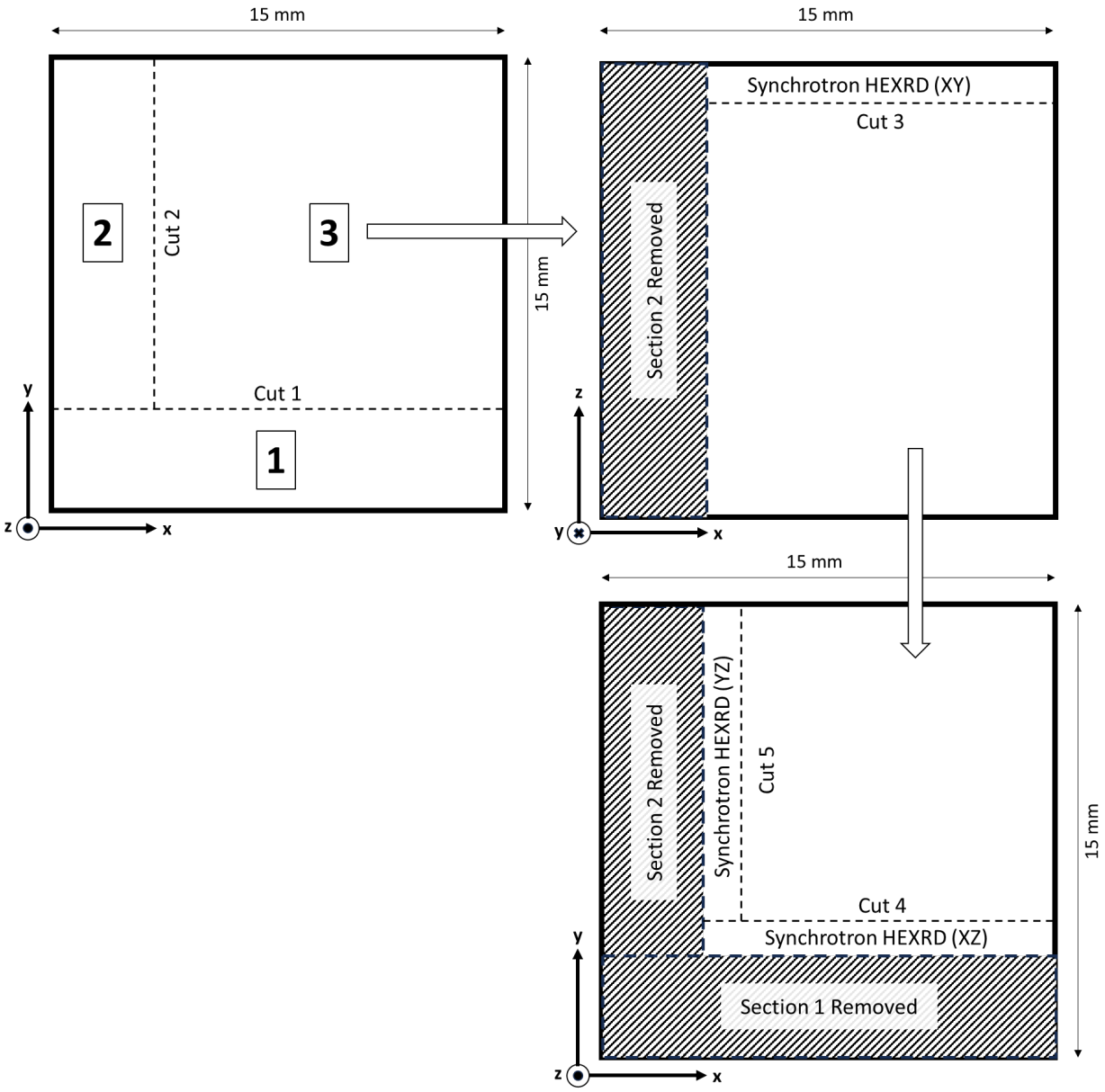

Figure 4. A schematic diagram for the sectioning of 15.Y parts to reveal different orientation planes for microstructural characterization. Note the sketch is not drawn to scale.

3.2 Scanning Electron Microscopy

Samples in the XY, XZ and YZ orientations for scanning electron microscopy (SEM) were mounted in graphite-filled conductive epoxy using hot pressing at a temperature of 180 °C and a pressure of (2.9×10^7) MPa (290 bar) into 32 mm diameter pucks. Mounted samples were subjected to standard metallographic preparation by first grinding with a series of silicon carbide papers starting at 400 grit (P800) and ending with 1200 grit (P4000). Polishing was successively performed on cloths with 6 µm, 3 µm and 1 µm diamond suspensions to achieve a mirror finish, followed by final polishing with 0.05 µm colloidal silica. Microstructural features were revealed using electrolytic etching with 10 % by volume aqueous chromic acid at 3.5 V and current densities ranging from approximately 0.07 A/cm^2 to 0.12 A/cm^2 depending on the cross-sectional area of the sample. The etching response was monitored by visual inspection and typically completed after 5 s to 10 s.

The samples were placed in a vacuum desiccator for at least 24 h prior to loading into the SEM. The large interaction volume with respect to the size of precipitates and scale of elemental segregation meant that only secondary electron imaging could be used for analysis in the SEM. The SEM was operated at an accelerating voltage of 5 kV, a beam current of 1 nA, and a working distance of approximately 4 mm. At least five locations were inspected for each sample orientation, and images were acquired at different magnifications at each location.

The electrolytic etching revealed solidification cell boundaries in the as-deposited microstructures and images were acquired where cellular growth was either parallel or perpendicular to the plane of view. The average cell size was quantified using the linear intercept method commonly used for grain size determination. Multiple lines of equal length were overlayed on the solidification microstructure and the intercepts of the lines with cell wall boundaries were counted. The total number of intercepts, where the intersection with a single boundary was recorded as a value of 1 and an intersection with a triple point was recorded as a value of 1.5, were summed. The length of the line was then divided by the total number of intercepts for each line, and the average cell size and standard deviation were calculated.

3.3 Transmission Electron Microscopy

A thin wafer of the XY plane measuring approximately 250 µm was sectioned from each as-built material using a precision saw. The wafer was mechanically ground with 600 grit (P1200) silicon carbide paper to an approximate thickness of 100 µm. A hole punch was then used to create 3 mm discs for twin jet electropolishing. The 3 mm discs were placed in the electropolishing apparatus and subjected to an electrolyte consisting of 300 mL of

methanol, 175 mL of butanol and 30 mL of perchloric acid. Electropolishing was conducted in the temperature range of -18 °C to -10 °C at voltages ranging from 25 V to 34 V and currents ranging from 34 mA to 45 mA. The amount of time to achieve electron transparency varied from approximately 50 s to 105 s depending on the sample.

The as-built samples were prepared for transmission electron microscopy (TEM) with the primary objective of visualizing any precipitates and quantifying the elemental segregation in the solidification microstructure. The samples were loaded into the TEM equipped with energy dispersive spectroscopy (EDS) for characterization and the TEM was operated at an accelerating voltage of 200 kV. Quantitative EDS maps were acquired using spectral imaging with an image size of 512 pixel x 512 pixel, a dwell time of 40 µs at each pixel and 100 frames acquired per map. The EDS data was calibrated using the known chemical composition of NIST SRM 2063a. The elemental segregation was quantified by extracting approximately 500 nm line scans centered over cell wall boundaries directed towards the center of the cell. Line scan locations were chosen to avoid denuded zones caused by precipitate formation during solidification. The mass fractions of all elements were averaged at the cell walls and interiors from multiple line scans.

3.4 Synchrotron High Energy X-Ray Diffraction

Phases present in each material were identified and quantified using high energy synchrotron X-ray diffraction (HEXRD) performed using beam line 1-ID at the Advanced Photon Source (APS) of Argonne National Laboratory (Lemont, IL, USA). The beam energy was 61.332 keV and the corresponding wavelength was 0.20215 Å. The diffraction data was acquired in transmission mode using a Pilatus 3 2M CdTe detector with a pixel size of 0.172 mm x 0.172 mm. The sample was positioned 685.92 mm from the detector, which was calibrated using NIST SRM 674 (CeO_2). A total of 36 individual locations were probed using a 6 x 6 measurement square sampling grid positioned around the sample center, with a 1 mm spacing between each location. The beam size was 0.25 mm x 0.25 mm, and the sample was exposed to the beam for 0.1 s per exposure for a total of 10 exposures at each location. The data at all 36 locations were then averaged into a single, 2D diffraction datafile to increase counting statistics and sampling volume.

The intensities of the 2D diffraction data were integrated along the azimuthal angle for each diffraction angle, 2θ, step to generate a 1D diffraction pattern of intensity as a function of 2θ. The 2θ peak positions were extracted from a Pseudo-Voigt fit to the data, and individual peaks were identified based on the powder diffraction files from the NIST Inorganic Crystal Structure Database. Volume fractions were calculated using the integrated intensities of fitted peaks and the corresponding phase-dependent structure factors using the direct comparison method.

4. Benchmark Challenge Problems

4.1 Benchmark Challenge CHAL-AMB2025-01-SR

This challenge problem consists of predicting the as-built microstructure in parts 15.13 and the microstructure evolution in parts 15.4 from each build after stress relief heat treatment at 870 °C for 1 h. The submission template for both AMB2025-01 challenge problems is found here, and the corresponding measurements are described in Section 3 of this document. Modelers are to enter the average solidification cell size in nm, the maximum and minimum mass fractions of Nb and Mo, corresponding to the cell walls and cell interiors, respectively. Additionally, modelers are asked to enter the volume fraction of each precipitate in the as-built and heat-treated microstructures. The phase corresponding to each calculated volume fraction should be indicated and more rows for additional precipitates may be entered as necessary. Oxide volume fractions may be entered, although these will not be judged as part of the challenge problem due to the difficulty of experimentally quantifying volume fractions of phases that may be partially or non-crystalline in nature.

4.2 Benchmark Challenge CHAL-AMB2025-01-H

This challenge problem consists of predicting the as-built microstructure in parts 15.13 and the microstructure evolution in parts 15.5 from each build after stress relief heat treatment at 1150 °C for 1 h. The submission template for both AMB2025-01 challenge problems is found here, and the corresponding measurements are described in Section 3 of this document. Modelers are to enter the average solidification cell size in nm, the maximum and minimum mass fractions of Nb and Mo, corresponding to the cell walls and cell interiors, respectively. Additionally, modelers are asked to enter the volume fraction of each precipitate in the as-built and heat-treated microstructures. The phase corresponding to each calculated volume fraction should be indicated and more rows for additional precipitates may be entered as necessary. Oxide volume fractions may be entered, although these will not be judged as part of the challenge problem due to the difficulty of experimentally quantifying volume fractions of phases that may be partially or non-crystalline in nature.

5. Data to be Provided

No additional data is currently provided to support this challenge problem, although new data files, updates, and/or changes to URLs may be made periodically.

6. References

[i] Certain commercial equipment, instruments, software, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the Department of Commerce or the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.