AMB2022-05 Benchmark Measurements and Challenge Problems

Last updated on 4/15/2022

Modelers are invited to submit simulation results for any number of challenges they like before the deadline of 23:59 (ET) on July 15, 2022. Tabulated results using the challenge-specific templates are required for most challenge problems and simulation results may be submitted <u>here</u>. An informational webinar for AMB2022-05 will be held on May 5, 2022, from 14:30 – 15:30 Eastern Time. The webinar registration link is <u>here</u>. After the webinar is completed, links to the recorded presentations and to a FAQ page will be added to the AMB2022-05 description page. Additional information may become available later so updated versions of this document may be posted. Please check back occasionally.

All evaluations of submitted modeling results will be conducted by the AM-Bench 2022 organizing committee. Award plaques will be awarded at the discretion of the organizing committee. 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.

Please note that the challenge problems reflect only a small part of the validation measurement data provided by AM Bench for each set of benchmarks. The Measurement Descriptions section, below, describes the full range of measurements conducted.

<u>AMB2022-05</u>: Microstructure measurement extension to AMB2018-01: laser powder bed fusion (LPBF) 3D builds of nickel-based superalloy IN625 test objects. Detailed descriptions are found below, and simulation results may be submitted <u>here</u>.

Challenge

- Microstructure (CHAL-AMB2022-05-MS): Histograms of direction-specific grain sizes from specified regions within an as-built specimen.
- 1. Overview and Basic Objectives
- 2. Build Process and Part Design
- 3. Measurement Descriptions
- 4. Benchmark Challenge Problems
- 5. Data to be Provided⁺
- 6. References

1. Overview and Basic Objectives

AMB2022-05 is a direct extension to the measurement data provided by AMB2018-01. For AMB2018-01, data were provided for laser powder bed fusion (LPBF) builds of IN625, including powder characterization, detailed information about the build process, in situ measurements during the build,

ex situ measurements of the residual stresses, part distortion following partial cutting off the build plate, location-specific microstructure characterization, and microstructure evolution during a post build heat treatment. Through interactions with the AM modeling community, it became clear that additional microstructure characterization data would prove useful. Here, we extend the previous microstructure data to include three additional data sets: 1) large-area SEM characterization of the mid-plane of the bridge specimen, including part of the baseplate, 2) multiple SEM cross sections of a complete test artifact parallel to the baseplate, and 3) a 3D microstructure measurement obtained through serial sectioning with optical and SEM imaging.

When the original AMB2018-01 specimens were produced, a full build plate of four IN625 bridge specimens was reserved for future use and two of these were used to provide samples for AMB2022-05. Because several microstructure-related challenge problems were conducted for these builds in 2018, we are adding just a single challenge for these new 2022 data. Note that these builds performed on the commercial build machine (CBM) utilize a different coordinate system than those performed on the NIST additive manufacturing metrology testbed (AMMT) in other AMB2022 challenge sets.

2. Build Process and Part Design

Full details of the build plate, part design, IN625 powder, build process, in situ monitoring, and ex situ measurements for the AMB2018-01 build plates are available on the AMB2018-01 website <u>here</u>, or the various associated AMB2018-01 publications listed <u>here</u>. The reserved build plate used for AMB2022-05 is designated AMB2018-625-CBM-B3.

2.1 Part Layout and Specimen Naming Convention

Figure 1 shows the design of the AMB2018-625-CBM-B3 build plate along with the part naming convention. The two parts used for these benchmark measurements are AMB2018-625-CBM-B3-P3 and AMB2018-625-CBM-B3-P4. Figure 2 shows the leg numbering convention used.



Figure 1: Part numbering for reserved AM Bench 2018 build plate



Figure 2: Leg numbering for reserved AM Bench 2018 bridge specimens

- 2.1.1 XZ Plane Cross Section: The large-area SEM measurements of the XZ midplane used a single multiple-leg segment from part 3 (P3) including legs L1, L2, and L3, with a sample designation of AMB2018-625-CBM-B3-P3-L1-L2-L3-O1, where the O1 specifies the cross section taken from the front part of the bridge section as shown in Fig. 2.
- **2.1.2 XY Plane Cross Sections:** For the measurements on AMB2018-625-CBM-B3-P4, the specimen was sectioned along 4 planes parallel to the build plate (XY plane). The planes are at heights of 1 mm, 4 mm, 7 mm, and 11 mm from the top surface, neglecting the small 0.5 mm nubs. The heights at which the samples are sectioned are shown in Fig. 3 and the arrows on the right specify the side that was imaged for each cross-sectional measurement. The samples are then grouped into 5 verticals (A, B, C, D, and E) as shown in Figure 4. The 4 verticals (A, B, C and D) share the repeatable geometry (thick, thin, and medium leg). The geometry of vertical E is non-repeating.

The measured parts are designated using a combination of the part ID, vertical section, horizontal section, and plane number. Thus, AMB2018-625-CBM-B3-P4-AK3 specifies the Plane 3 surface of section k as defined in Fig. 3, for vertical section A as defined in Fig. 4. This area includes cross sections of L1, L2, and L3. Thus, for SEM measurements of L3, the complete unique sample ID would be AMB2018-625-CBM-B3-P4-AK3-L3.



Linear dimension units are in millimeters

Figure 3: Diagram showing locations of planes used for the SEM measurements parallel to the build plate. The arrows specify the specific surfaces used for the measurements.



Figure 4: Vertical sections used to define SEM sample groupings

3. Measurement Descriptions

3.1 XZ Plane Cross Section

After cross sectioning using electrical discharge machining, the specimen was mounted in epoxy resin and progressively polished using 1200, 2000, 4000 grit SiC papers, followed by 1 µm diamond and colloidal silica. Figure 5 shows a photograph of the mounted specimen.

Electron backscatter diffraction (EBSD) measurements were conducted on an FEI Apreo SEM⁺ with the settings listed in Table 1. The individual mapped regions are shown and numbered on Fig. 5. Additional measurements from these same regions may be added later. A montage of the measurement areas was produced by a self-adaptive montage stitching algorithm that maximizes the cross-correlation within the overlapping regions between the tiles. It is assumed that each of the EBSD scans have the same scanning

distortion, thus the optimization routine finds the coefficients of a single polynomial spatial warping function that maximizes the summed maximal values of the cross-correlation functions of each overlapped region. After this optimal warping function is found, the tiles are stitched together by the translations that again maximize the cross-correlation between each tile (note here that unlike the spatial warping function, these translations can be different for each set of tiles). The data are combined into a single dataset file that then can be analyzed as a whole, or with different sub-regions.



Figure 5: Photograph of the mounted and polished XZ-plane specimen, AMB2018-625-CBM-B3-P3-L1-L2-L3-O1. The approximate EBSD measurement locations are outlined and numbered.

Tuble 1. Lood mup measurement and display settings	
EBSD Map Measurement and Display Settings	
Accelerating voltage	20 kV
Binning	4 x 4
Step size	1 μm
Minimum points/grain	2

3.2 XY Plane Cross Sections

The samples were cut using wire electrical discharge machining (EDM) and mounted in conductive bakelite, polished in steps from 600, 800, 1200, 2000 grit SiC followed by polishing in 3 μ m and 1 μ m diamond suspensions which is then followed by colloidal silica for 6 hours. SEM measurements were conducted on a Tescan Mira 3⁺ with a 20 KV accelerating voltage with varying step size (2-5 μ m).

3.3 3D Microstructure

This set of automated serial sectioning measurements (Uchic et al. 2012, Chapman et al. 2021) was designed to produce a micrometer-scale, 3D characterization of the microstructure within a region of interest (ROI) of a 2.5 mm leg and extending into the baseplate. Figure 6 shows the 3D ROI within the base of the 2.5 mm leg AMB2018-625-CBM-B3-P3-L12-O1. The ROI measures approximately 500 μ m × 500 μ m × 750 μ m along the build X, Y, Z directions respectively, and is contained in a larger needle shaped region excised from the leg using wire EDM. All EDM cuts were separated from the ROI by at least 250 μ m. Additionally, the Z dimension of the ROI includes approximately 250 μ m of the baseplate material. Fiducial marks were laser engraved onto all X and Y faces of the specimens using a Micromac microPREP PROTM⁺ system, with all fiducial marks separated from the ROI by at least 50 μ m. The specimens were then affixed in a specially designed holder which was incorporated into a conductive metallographic mount. The build X and Y directions lie in the plane of the mount, with the build Z direction oriented inward and normal to the mount surface.



Figure 6: Location on Leg 12 where sample is extracted for serial sectioning measurements

Each serial sectioning step proceeds by first removing approximately 2 µm of material, including a final polish with 40 nm colloidal silica in a Robomet.3DTM⁺ system. The height of the serial section surface is determined for each section using a differential imaging measurement. The specimen is then transferred to a Zeiss AxioImager.Z2m⁺ optical microscope to collect several optical bright-field images, including a montage of the full cross section. Next, the specimen is transferred to a Tescan Vega 3 scanning

electron microscope (SEM) where a backscatter electron (BSE) image covering the full specimen cross section is collected. This is followed by collection of a higher resolution BSE image covering a more limited field of view centered on the ROI. Finally, an electron backscatter diffraction (EBSD) image is collected with the specimen at a 70° inclination using a Bruker Quantax e-Flash 1000⁺ EBSD detector.

The individual datasets described above are post-processed to produce a 3D reconstruction of the ROI using a range of tools including ImageJ/Fiji, DREAM.3D, and custom scripts. The individual optical montages are first reconstructed into a 3D stack by aligning features visible in the metallographic mount. Next, to minimize distortions introduced in SEM data collection, the lower resolution BSE image is spatially registered to the optical montage from the corresponding slice. Next, the higher resolution BSE image covering just the ROI is registered to the larger area BSE image, and finally the EBSD data are registered to the higher resolution BSE image. The ROI contains two orthogonal as-printed exterior surfaces as well as the top-surface of the substrate plate. These features are sufficient to locate the ROI with respect to the original build coordinate system. Application of this series of transformations therefore allows placement of ROI data into the original build coordinate system. The final ROI spans from unaffected baseplate material, through remelted baseplate material, and into additive deposited material.

4. Benchmark Challenge Problems

4.1 CHAL-AMB2022-05-MS

There are many possible metrics that can be used to evaluate the degree of similarity between measured and simulated microstructures. For this challenge problem, we needed a metric that could be easily implemented by the modelers and that would readily distinguish between different directions in the microstructure. We tried several different metrics using published microstructure data from AM Bench 2018 IN625 builds and the algorithm we selected is a histogram of chord lengths obtained from multiple parallel lines intersecting grain boundaries along the orthogonal directions. As an example, Fig. 7 shows a Z-direction inverse pole figure (IPF) map obtained using EBSD from a YZ surface within an AM Bench 2018 IN625 build (refer to Figure 9 in 2018 measurement description). Here, adjacent pixels with a 2° misorientation angle are delineated by a thin line, and those with an angle of 10° or higher are delineated by heavier lines. Looking only at the 10° or higher boundaries, Figure 8 shows a set of lines drawn parallel to the Z axis for a sub-region of the full measurement. Histograms of the intercept lengths are shown in both linear and log scale plots. Figure 9 shows this same analysis applied along the Z direction. Note the differences in the histograms for the two orientations.



250 µm

Figure 7: Z-direction inverse pole figure plot from a YZ plane within an AM Bench 2018 IN625 build



Figure 8: Z-direction intercept length example for a sub-region using the EBSD map shown in Figure 7.



Build direction intercept length (log scale)

Figure 9: Y-direction intercept length example for a sub-region using the EBSD map shown in Figure 7.

The measurement data used for this challenge problem are the extended XZ-plane large area EBSD maps acquired from AMB2018-625-CBM-B3-P3-L1-L2-L3-O1 (as built) as described in section 3.1. Two 0.6 mm x 1.0 mm square regions and one 1.0 mm x 1.0 mm region from the as built sample will be used for the challenge problem. As shown in Figure 10, these include a region at the base of a 5.0 mm leg near the edge, a second region toward the top of the same 5.0 mm leg near the same edge, and a third region within the bridge section. The locations and sizes of the specified regions are given in Figure 10.



Figure 10: Zoomed-in region from Fig. 5 showing the sizes and locations of the three modeling challenge regions for CHAL-AMB2022-05-MS

For this challenge problem, adjacent pixels with at least a 10° misorientation angle define a grain boundary. The line spacing between the parallel lines should be 5 μ m and the width of the bins for the linear intercept length histograms should be 100 μ m. Thus, for the X lines for a 0.6 mm x 1.0 mm box, the number of intercept lengths within the range (0,100] will be summed in the first bin, the number of intercept lengths within the range (100,200] will be summed in the second bin, etc. up to the final bin where the range is (500,600]. *It is important to note that these regions reside in the XZ plane, which is different from the AMB2018 example shown above in Figs. 7 - 9.* The submission template for this challenge problem may be found in the Challenge Description Dataset <u>here</u>, with the filename "CHAL-AMB2022-05-MS submission template.csv". Please also submit an IPF-Z map for each sample region for which a histogram is submitted.

5. Description and Links to Associated Data

The microstructure evolution during the build process has some dependence upon the microstructure of the baseplate. We are therefore providing an EBSD dataset for an XY plane within the build plate that was obtained during the 3D serial sectioning measurements described in section 3.3. The provided data is from a region deep enough into the baseplate to be unaffected by the build process.

The provided data may be found in the "AM Bench 2022 Microstructure of IN626 3D Builds - Modeling Challenge Description Data (AMB2022-05)" dataset available here[†]: <u>https://doi.org/10.18434/mds2-</u>2618. Please refer first to the "2618_README.txt" file for details on the available files.

6. References

Citations are provided throughout this document as hyperlinked URLs to the associated digital object identifier (DOI). Clicking these hyperlinked text should open the associated publication or cited source.

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