

AMB2022-07 Benchmark Measurements and Challenge Problems

Modelers are invited to submit simulation results for any 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 [here](#). An informational webinar for AMB2022-07 will be held on May 6, 2022, from 12:15 – 13:15 Eastern Time. The webinar registration link is [here](#). After the webinar is completed, links to the recorded presentations and to a FAQ page will be added to the AMB2022-07 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 Description section, below, describes the full range of measurements conducted.

AMB2022-07: Vat Photopolymerization Measurements of Cure Depth and Print Fidelity Vs. Varied Exposure Duration, Photopattern Dimensions, and Resin Characteristics

Detailed descriptions are found below, and simulation results may be submitted [here](#).

- Optical profile at print plane (CHAL-AMB2022-07-OP): Light intensity profile at the print plane.
- Cure depth dependence on photomask dimensions (CHAL-AMB2022-07-CD): Cure depth as a function of photomask linewidth and resin characteristics.
- Print profile (CHAL-AMB2022-07-PP): Solid cross section profile of the printed patterns as a function of photomask linewidth and resin characteristics.

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1. Overview and Basic Objectives

This Photopolymer AM-Bench 2022 Challenge is to accurately model the relationship between photopattern exposure duration and resultant single-layer part dimension (i.e. fidelity and cure depth) for four resins, which serve to orthogonally probe the relationship between resin reactivity k and

viscosity ν . The primary objectives are to determine if the dimensions of a photomask and resin viscosity and reactivity affect cure depth and print fidelity. Ultimately, the improved understanding and prediction of these relations will foster enhanced print resolution and part performance.

Experimental data for model calibration and challenge comparison is provided through cure profile measurements using laser confocal scanning microscopy in conjunction with resin characterization (i.e. Fourier transform infrared spectroscopy, oscillatory rheometry) and system calibration (e.g., photomask dimensions, beam profilometry, radiometry). These experiments were carried out at the National Institute of Standards and Technology and the University of Colorado. Released calibration measurements were performed on four, open-source resins to serve as representative examples for resins in the field. Calibration data are available for download [here](#).

2. Printing and Post- Processing Description

2.1. Materials and Sample Preparation

2.1.1. Resin Formulation

Acrylates and methacrylates are known to have different polymerization rates. In order to develop two resins of equal viscosity, with different polymerization rates, methacrylate based and acrylate based resins with viscosities of ≈ 150 mPa*s and ≈ 85 mPa*s were formulated.

The following materials were used in the formulations of the four resins:

Acrylate monomers: Bisphenol A glycerolate (1 glycerol/phenol) diacrylate (BisGA, Sigma Aldrich, CAS# 4687-94-9, $M_w = 484.54$ g/mol), isobornyl acrylate (IBA, Sigma Aldrich, CAS# 5888-33-5 607-133-00-9, $M_w = 208.30$ g/mol), and trimethylolpropane triacrylate (TMPTA), Sigma Aldrich, CAS# 15625-89-5, $M_w = 296.32$ g/mol)

Methacrylate monomers: Bisphenol A glycerolate dimethacrylate (glycerol/phenol 1) (BisGMA, Sigma Aldrich, CAS# 1565-94-2, $M_w = 512.59$ g/mol), isobornyl methacrylate (IBMA, Sigma Aldrich, CAS# 7534-94-3, $M_w = 222.32$ g/mol), trimethylolpropane trimethacrylate (TMPTMA, Sigma Aldrich, CAS# 3290-92-4, $M_w = 338.40$ g/mol).

Photoinitiator: Diphenyl(2,4,6-trimethylbenzoyl)-phosphine Oxide (TPO, TCI Chemicals CAS# 75980-60-8, $M_w = 348.38$ g/mol)

The bisphenol based di-functionalized monomers have very high viscosities, so lower viscosity comonomers were employed to bring viscosities down to our targeted range appropriate for 3D printing. Isobornyl acrylate/methacrylate and trimethylolpropane triacrylate/trimethacrylate were used as the lower viscosity comonomers and the tri-functionalized monomers allowed for high reactivity, respective to the other monomers, while the acrylates exhibited the higher reactivity relative to the methacrylates. Many common 3D printers use a 405 nm light to initiate photopolymerization so TPO at 1 wt% was selected as the photoinitiator for these formulations. Resins were mixed by selecting a 10 gram basis and initially weighing out the high viscosity bisphenol based monomers, followed by the addition of the lower viscosity isobornyl and trimethylolpropane based monomers (by mass). The resin formulations were mixed in a 45 °C water bath until the formulations were homogeneous. Finally, the TPO photoinitiator was added and mixed at room temperature until fully dissolved.

For the high reactivity, acrylate-based resins, the BisGA was diluted with IBA and TMPTA to bring the viscosity down to the targeted range of ≈ 150 mPa*s. For the lower viscosity, ≈ 85 mPa*s, acrylate-based resin, the BisGA content was reduced and supplemented with IBA to reduce the viscosity to the target value. For the relatively lower reactivity, methacrylate-based resins, BisGMA was diluted with IBMA and TMPTMA at different weight ratios to bring the viscosities down to the targeted range of ≈ 150 mPa*s and ≈ 85 mPa*s.

Table 1: Resin composition broken down into respective reactivities, viscosities, and components

| | | | BisGMA (wt%) | IBMA (wt%) | TMPTMA (wt%) |
|----------|------------|---------|--------------|------------|--------------|
| Low k | High ν | Resin 1 | 40.25 | 17.25 | 42.5 |
| | Low ν | Resin 2 | 31.5 | 13.5 | 55 |
| | | | BisGA (wt%) | IBA (wt%) | TMPTA (wt%) |
| High k | High ν | Resin 3 | 28 | 12 | 60 |
| | Low ν | Resin 4 | 23 | 17 | 60 |

2.1.2. Sample substrate preparation

All samples were fabricated using methacrylate-functionalized 140 μm thick coverglass (Sigma Aldrich) as a substrate/window to ensure the pattern polymerized to the desired substrate. The functionalization procedure is as follows:

- Mix ethanol (95 % by volume) with deionized water (5 % by volume)
- Add acetic acid (Sigma Aldrich) to lower pH to between 4.5-5.5
- Remove desired amount from stock solution and place into coating beaker
- Add 3-(trimethoxysilyl)propyl methacrylate (Sigma Aldrich) (2 % by volume)
- Mix using magnetic stir bar at 200 RPM for 30 min
- Clean coverglass by rinsing in pure ethanol for 10 minutes
- Place cleaned coverglass immediately into vial of methacrylate solution for 20 min
- Remove coverglass from solution, rinse with approximately 3 mL ethanol to remove excess silane, and dry using air gun
- Thermally cure coverglass for 10 min at 110 °C for 10 min to cure methacrylate functionalization
- Mechanically clean coverglass to remove remaining, untethered silane
 - o Deposit ethanol onto coverglass, rub front and back surface of coverglass between index and forefinger in circular patterns using lens paper for 10 seconds
 - o Repeat rubbing procedure for 10 seconds using new, dry lens paper

2.2. Light Engine

The light engine is comprised of a high-power, collimated 405 nm LED (Thorlabs, SOLIS-405C), a graduated iris aperture (Thorlabs, SM1D12C), and a chrome photomask (Edmund Optics, high precision Ronchi ruling), illustrated in **Figure 1**.

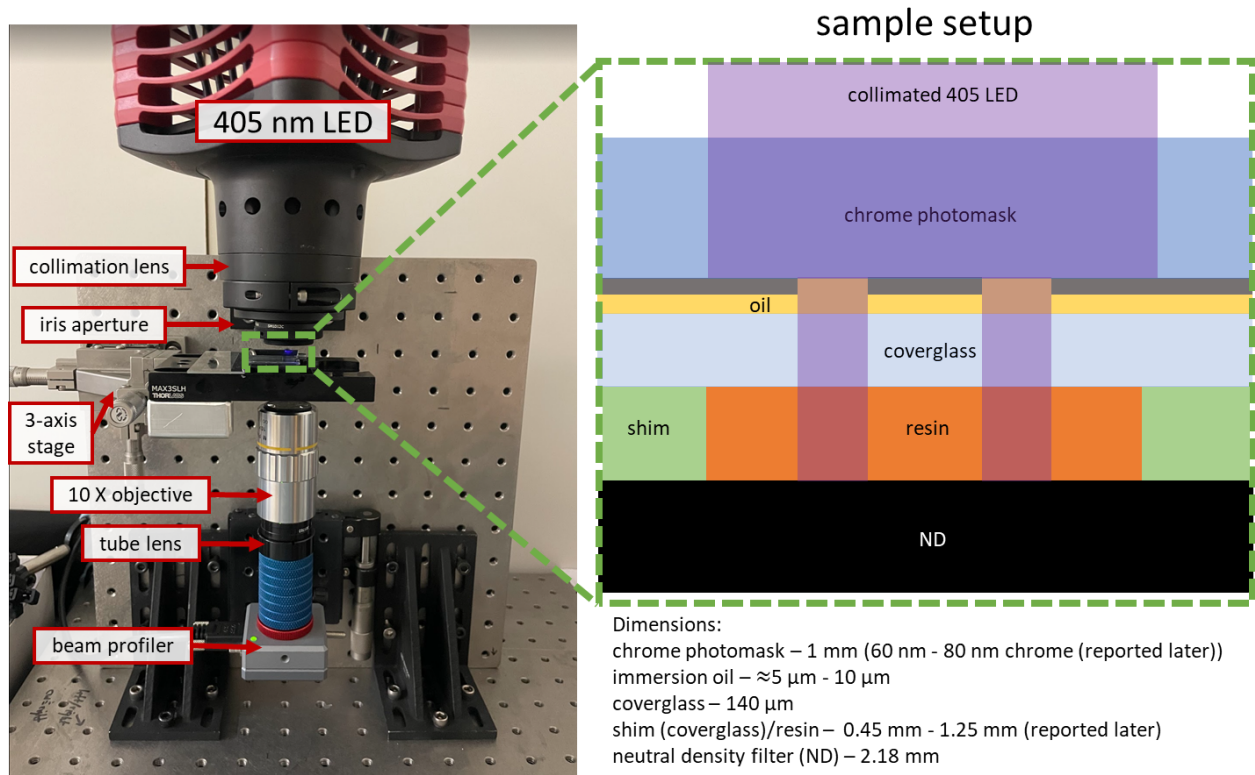
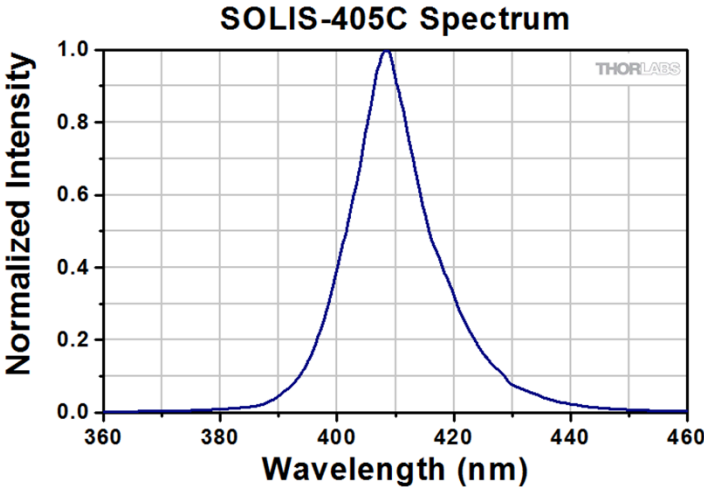


Figure 1: Projection light engine used for all photopatterned exposures with an expanded schematic of the sample setup.

2.2.1. Light source

A high-power, collimated 405 nm LED was used for all photopatterning (Thorlabs, SOLIS-405C). The light engine uniformity and intensity were measured using beam profilometry and radiometry, respectively. Additional manufacturer specifications for the light source are listed in **Table 2**.

Table 2: Specification provided by Thorlabs for SOLIS-405C LED
 (https://www.thorlabs.com/newgrouppage9.cfm?objectgroup_id=8986&pn=SOLIS-405C).

| Specified property | Value |
|---------------------------------|---|
| Maximum Collimated Output Power | 5.0 W |
| Typical Collimated Output Power | 3.9 W |
| Bandwidth (FWHM) | 12 nm |
| LED Spectrum |  <p>The graph, titled "SOLIS-405C Spectrum", plots Normalized Intensity on the y-axis (ranging from 0.0 to 1.0) against Wavelength (nm) on the x-axis (ranging from 360 to 460). A single, sharp peak is centered at approximately 405 nm, reaching a normalized intensity of 1.0. The full width at half maximum (FWHM) of the peak is approximately 12 nm. The Thorlabs logo is visible in the top right corner of the plot area.</p> |
| Collimating optic | N-BK7 Bi-Convex Lens, $\varnothing 2''$, $f = 60.0$ mm, ARC: 350 nm -700 nm |

2.2.2. Aperture

A ring actuated, graduated iris was used to define the edge boundaries of the aperture, which can be locked to a set aperture dimension. The aperture functions as a hard, zero-transmission boundary for photopatterning. A 3 mm aperture and 2 mm aperture were used for the cure depth calibration dataset and solution dataset, respectively.

2.2.3. Photomask

Ronchi ruling photomasks (Edmund Optics) were used for all experiments. The patterns are comprised of vacuum-deposited chrome (nominal optical density (OD) $\cong 3.0$) on float glass (nominal thickness $\cong 1.5$ mm). The masks are 25.4 mm x 25.4 mm overall lateral dimension, with the pattern repeated periodically across the surface. The pattern dimensions are defined by line-pairs-per-inch (LPPI), which can be converted to nominal line width by assuming a 50 % duty cycle of mask/un-mask. Nominal linewidth dimensions are listed in **Table 3**.

True optical density can be calculated for each specific pattern using the chrome thickness measured by atomic force microscopy for each photopattern. Modelers may also incorporate the float glass thickness data measured with ± 10 μm accuracy, measured by digital calipers. Measured lateral dimensions of photomask features were measured by Laser Scanning Confocal Microscopy.

Table 3: Manufacturer-specified photomask linewidths with designated identifier, line-pairs-per-inch (LPPI), and the subsequent conversion from LPPI to nominal micrometers per line (assuming 50 % duty cycle).

| Pattern Identifier | LPPI | $\mu\text{m}/\text{line}$ |
|--------------------|------|---------------------------|
| P1 | 25 | 508 |
| P2 | 50 | 254 |
| P3 | 100 | 127 |
| P4 | 150 | 84.7 |
| P5 | 200 | 63.5 |
| P6 | 250 | 50.8 |
| P7 | 300 | 42.33 |

2.3. Printing process

All samples were printed using the following procedure:

- Deposit index matching immersion oil (Sigma Aldrich, index of refraction = 1.52) onto back surface of clean coverglass
- Place photomask directly on top of oil with chrome surface contacting the oil
- Lay lens paper onto to surface of photopattern and then place 1 kg aluminum weight onto pattern and wait 1 minute to ensure uniform oil thickness layer
- Deposit resin solution onto neutral density filter (ThorLabs, Optical Density = 3.0) between two shims (0.45 mm for the solution dataset and 1.25 mm for the calibration dataset) until resin fills entire volume between shims and coverglass substrate (approximately 350 μL (solution dataset) and 1 mL (calibration dataset))
- Place coverglass-photomask system directly onto open resin surface with clean coverglass contacting the open resin, ensuring no air bubbles formed in resin during lamination
- Place entire sample system into the light engine photopatterning apparatus described in Section 2.2
- Translate sample vertically until at photopatterning location (where focus calibration measurements were taken)
- Begin photopatterning for pre-defined exposure durations, translating the x and y actuators to polymerize new locations within the single slide
- Once photopatterning complete, remove sample from system

2.4 Post-processing

All samples were post-processed using the following procedure:

- Remove Ronchi ruling photomask from back surface of coverglass by sliding the mask along the narrow dimension of the coverglass until removed, holding tension at the shim locations to ensure the coverglass does not shift on the ND filter + shim system
- Gently slide photo-patterned coverglass from ND filter + shim system to obtain resin-encased prints
- Remove immersion oil from the back surface of the coverglass by applying IPA with lens paper

- Place sample vertically into tissue-lined, light-tight sample holder container to allow excess resin to drip and wick from substrate for between 2 h and 5 h
- Remove monomer-soaked tissue and place entire sample holder into volume of IPA and mix system at 100 rpm for 45 minutes using a magnetic stir bar (ensuring stir bar does not directly contact/agitate sample holder)
- Remove sample holder from IPA bath and gently rinse each sample using IPA and placing a tissue at the edge of the slide to wick away remaining IPA
- Place sample into UV post cure oven for 30 minutes (now ready for characterization)

2.5 Specimen Naming Convention

All specimens were named using the following convention:

AMB2022-07_{experiment date}_{resin used}-{v}{k}_{Ronchi ruling used}_{exposure time}_{specimen slide number}_{row number}_{column number}

Here, AMB2022-07 is for Additive Manufacturing Benchmarks 2022 Challenge Problem 7 and the bracketed regions indicate a specific, unique identifier for the specimen. For example, a specimen made on February 18, 2022 using Resin 1 (which has high viscosity and low reactivity ranking), the 300 LPPI, and a 1 s (1000 ms) exposure with the slide number 3 would go as follows:

AMB2022-07_2022-02-18_Re1-HL_1000ms_300LPPI_3_r1_c2

An example specimen is shown in **Figure 2** to demonstrate the definition of rows and columns.

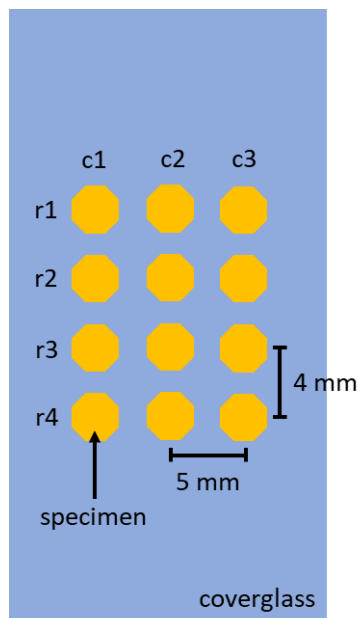


Figure 2: Illustration of an exemplary photopatterned exposure dataset with the associated rows and columns defined and the corresponding center-to-center photopattern separation distances. Columns are exposure replicates and rows are variations in exposure duration.

3. Measurement Descriptions

The AMB2022-07 benchmark elucidates fundamentals of vat photopolymerization additive manufacturing by measuring the shape of patterned features subject to varying exposure duration, photomask dimensions and resin characteristics. The calibration data seek to provide the most essential properties to predict the profile of the photopattern and the dominant properties of the resin. The data also provide phenomenological characteristics such as the working curve and associated critical exposure dose and depth of polymerization. These working curve raw data obtained on ≈ 500 μm wide masked features also provide insight into the real, non-rectangular profiles of the progressing cure front. The measurements include:

Resin Characterization

- Fourier transform infrared spectroscopy measurements of polymer conversion vs exposure duration for a constant exposure intensity
- Oscillatory rheometry measurements of resin viscosity prior to exposure

Light Engine Characterization

- Dimensional measurements of the chrome photomasks using laser scanning confocal microscopy and atomic force microscopy
- Radiometric measurements of the LED light source to determine optical power
- Beam profile measurements at the print plane to determine uniformity of the illumination

Printed Feature Characterization

- Laser scanning confocal microscopy measurements of printed patterns as calibration data using ≈ 500 μm photomasks and as solution data using the other photomask sizes

3.1. Resin Characterization

3.1.1. Resin Reactivity: Fourier Transform Infrared Spectroscopy (FTIR)

Fourier-Transform Infrared Spectroscopy was conducted using a Thermo Fisher Nicolet 6700 FT-IR. The acrylate/methacrylate peak at 6167 cm^{-1} was monitored for double bond conversion to determine the kinetics of curing. Experimental parameters are as shown in **Table 4**.

Spacers of $800\text{ }\mu\text{L}$ thickness were punched with a 12 mm diameter hole-punch. The resins were then filled into the spacers and sandwiched between glass slides to eliminate the potential for oxygen inhibition. Resins were then placed in the spectrometer and the kinetic runs were started, monitoring the 6167 cm^{-1} peak with a sampling interval of 0.52 s and a resolution of 4 cm^{-1} . An EXFO Acticure 4000 spot curing lamp with a 405 nm filter at 1 mW cm^{-2} was employed for photocuring and at the 30 s time point, the lamp was switched on and reduction in peak area, corresponding to double bond acrylate/methacrylate conversion was monitored.

Table 4: FTIR experimental parameters used for all resin characterization.

| Parameter | Value |
|--------------------|-----------------------|
| Sample compartment | Main |
| Detector | InGaAs |
| Beamsplitter | XT-KBr |
| Source | Transmission E.S.P. |
| Window | KBr |
| Max range limit | 7000 cm^{-1} |
| Min range limit | 4000 cm^{-1} |
| Gain | Autogain = 8 |
| Optical velocity | 3.7974 |
| Aperture | 5 |

3.1.2. Resin Viscosity: Rheometry

All resin viscosities were determined with a Brookfield CAP 2000+ Viscometer with a 30.25 mm diameter spindle rotating at 250 RPM. 25.10 μL of resin was dispensed on a frictionless mirror and the spindle was lowered onto the resin and held for 10 s to equilibrate. The spindle was then rotated for 10 s at a shear rate of 3,333 s^{-1} and viscosities were determined ($n = 7$). All rheology measurements were done in accordance with the following industry standards: ASTM D4287, ISO 2884, and BS 3900.

3.2. Light engine characterization

3.2.1. Photomask Optical Density: Atomic Force Microscopy

Atomic force microscopy (AFM, Asylum Research MFP3D) was used to determine the chrome thickness for all photomasks by using contact mode AFM to image the photopattern topography. A standard force modulation AFM probe (FM, Nanosensors) was used for all measurements. Photomasks were imaged in 3 arbitrary locations. Image analysis was performed in open source software (Gwyddion). Images were line flattened along the chrome, then 3-point plane fit to the glass substrate. Resultant height histograms were then used to determine the mean thickness between glass and chrome (**Figure 3**). These measurements were averaged for the 3 locations. From the chrome thickness, modelers can calculate the optical density of the photomask. Summary of chrome thickness measurements is shown in **Table 5**.

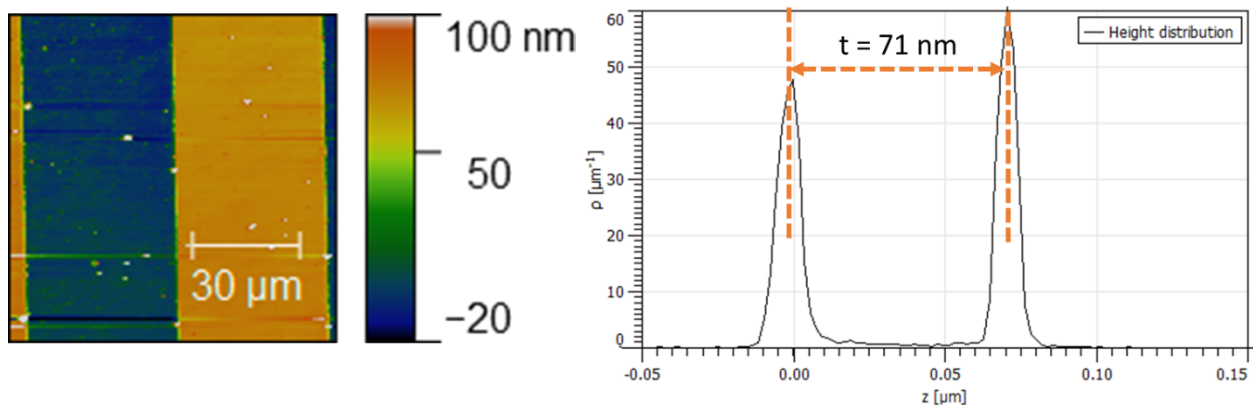


Figure 3: Contact mode atomic force microscopy topography scan of 300 LPPI Ronchi ruling. Height of chrome is determined from mean distance between substrate and chrome, determined by histogram.

Table 5: Average thickness of chrome layer in Ronchi ruling photomasks.

| Nominal LPPI | Average Chrome Thickness (nm) | Standard Deviation (nm) |
|--------------|-------------------------------|-------------------------|
| 300 | 75.4 | 1.6 |
| 250 | 68.2 | 0.8 |
| 200 | 78.9 | 2.6 |
| 150 | 71.5 | 1.5 |
| 100 | 78.4 | 2.0 |
| 50 | 77.0 | 3.0 |
| 25 | 69.1 | 6.4 |

3.2.2. Photomask lateral dimensions: Laser Scanning Confocal Microscopy

Laser Scanning Confocal Microscopy (LSCM, Keyence VK2000) was used to determine the lateral dimensions of the Ronchi rulings. Scans were performed with 20X objective (50X for 500 LPPI features), auto-brightness adjustment and automatic dual-scan selection. The dual scan function performs a 2nd LSCM scan at different brightness to enhance sidewall measurement accuracy. Image analysis was performed in open source software (Gwyddion). Measured line-pairs-per-inch were based on an average of at least 4 line pairs. Lateral dimensions of masked d_{mask} and un-masked $d_{\text{un-mask}}$ regions were measured from the midpoint of the height step between glass and chrome (Figure 4). Summary of LSCM Ronchi ruling measurements is shown in Table 6.

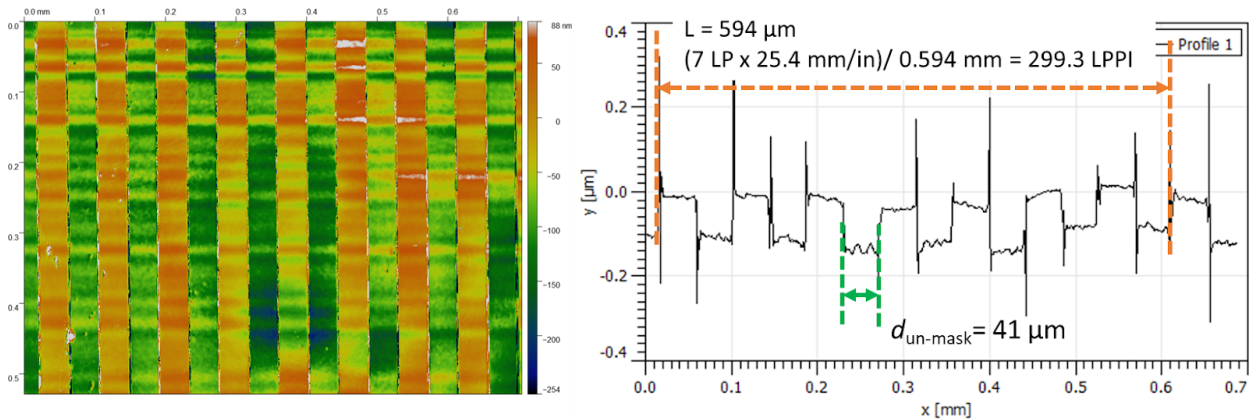


Figure 4: Example LSCM measurement on 300 LPPI Ronchi ruling. LPPI was measured across 4 or more line pairs (7 here). Un-masked feature size $d_{\text{un-mask}}$ is measured as indicated and averaged for at least 3 features.

Table 6: Summary of measured Ronchi ruling lateral dimensions

| Nominal LPPI | Measured LPPI | Nominal Un-masked width (μm) | Measured un-masked width $d_{\text{un-mask}}$ (μm) |
|--------------|---------------|---|---|
| 300 | 298.82 | 42.33 | 41.2 |
| 250 | 249.43 | 50.80 | 49.2 |
| 200 | 197.82 | 63.50 | 60.2 |
| 150 | 150.36 | 84.67 | 80.2 |
| 100 | 99.87 | 127.00 | 122.6 |
| 50 | 50.00 | 254.00 | 252.4 |
| 25 | 25.04 | 508.00 | 513.5 |

3.2.3. Light engine intensity calibration: Radiometry

Radiometry (Thorlabs, PM100D console, S170C photodiode) was used to determine the optical power of the 405 LED at the photopatterning plane. The average optical power at the sample plane was $39.3 \text{ mW cm}^{-2} \pm 0.1 \text{ mW cm}^{-2}$ ($n = 200$). As the photopattern exposure time was implemented by turning off and on the LED source, the LED optical power was also taken as a function of time to allow modelers to calibrate for any fluctuations in output power (**Figure 5**). Note: the LED response time was faster than the radiometer (10 Hz), thus power at the sample plane throughout exposure can be assumed constant. See Table 7 for all manufacturer-supplied power meter specifications.

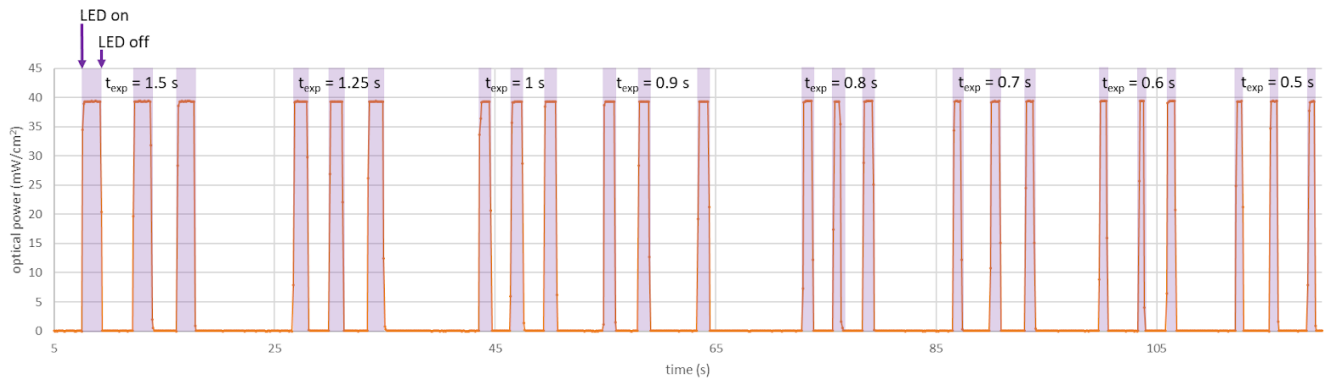


Figure 5: Optical power at the photopatterning plane, with triplicate exposures for each exposure time probed ($t_{\text{exp}} = \{1.5, 1.25, 1, 0.9, 0.8, 0.7, 0.6, 0.5\}$ s) to mimic experiment conditions. Violet shaded regions indicate the duration the LED was on.

Table 7: Optical power meter parameters used for photopatterning plane characterization.

| Parameter | Value |
|-------------------------|-------------------------------|
| Sample interval | 0.1 s (maximum sampling rate) |
| Sensor | S170C |
| Type | PM100D |
| Wavelength responsivity | 405 nm |

3.2.4. Light engine uniformity: Beam Profilometry

The light engine uniformity was calibrated using an optical beam profiler (DataRay, MODEL X). To ensure sufficient resolution to calibrate the intensity across the photopatterning plane at better resolution than the smallest photomasks features, a 10X objective (Mitutoyo, M Plano Apo, infinity corrected, $f = 200$ mm, $NA = 0.28$) was used with a tube lens (Optem, $f = 100$ mm) to magnify the projection onto the beam profilometer sensor (see system design in **Figure 1**). The beam profilometer + magnification optics system was then aligned with the photopattern exposure plane and focused using a mechanical linear micrometer. With the beam profiler aligned to the photopatterning plane, the open-aperture profile of the light engine was obtained (see **Figure 6**). For the solution dataset, all beam profilometry images were taken with each subsequent chrome photomask in the system aligned to the same focal plane.

Note that because the photopattern region was confined using an inset ring-actuated iris, the chrome photomask could only be placed at a distance approximately 2.5 mm from the aperture, resulting in defocused edges at the boundaries of the exposure (see **Figure 6**). Due to this region of defocus at the pattern edges, a 500 μm by 700 μm subregion at the center, more-uniform region of the photomask was taken, where the intensity distribution across the subregion and line profile across the entire aperture are shown in **Figure 6(d,b)**.

To obtain the beam profile measurement, the LED was turned on and the profilometer imaged the projected pattern, being deliberate to utilize the software absolute-value setting to ensure no normalization or auto-scaling of the intensity distribution was done. Each intensity distribution was collected and subsequently exported as a 16-bit tiff file.

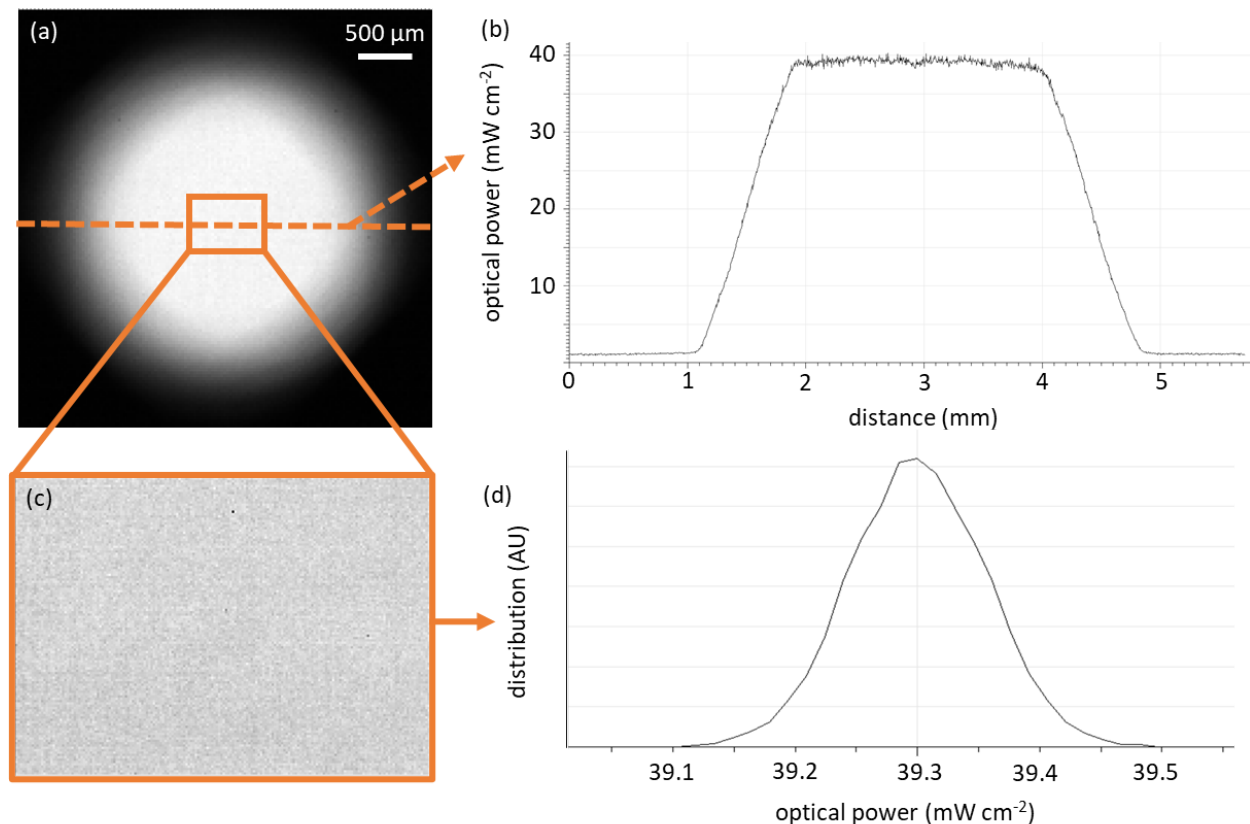


Figure 6: (a) Example beam profilometry measurement of the aperture at the photopatterning focal plane and (b) a representative line profile across the 3 mm aperture. The (c) expanded subsection is the

location where all solution data measurements are taken with (d) the corresponding intensity distribution.

3.2.5. Light engine spectrum uniformity: Optical Spectroscopy

A spectrum of the nominally 405 nm LED light source (Thorlabs, SOLIS-405C) was collected using a fiber coupled CCD spectrometer (Avantes, AvaSpec-UL4096CL-EVO). The spectrometer was configured with a 25 μm slit and a 600 lines/mm grating that yields a 0.6 nm spectral resolution. Wavelength accuracy was better than 0.15 nm as confirmed via a Hg/Ar calibration lamp (Avantes, AvaLight-CAL). The instrument response function was calculated via a NIST traceable halogen light source (Avantes, AvaLight-HAL-CAL-MINI). The Fresnel reflection from a glass slide was used to direct a portion of the 405 nm LED light source onto a 600 μm diameter broadband fiber cable that was connected to the spectrometer. The data were plotted as “relative irradiance” (i.e. proportional to irradiance – power/area/bandwidth). The stray light specification given by the manufacturer is 1 % and so features in the spectrum that are <1 % of the peak relative irradiance are not meaningfully interpretable.

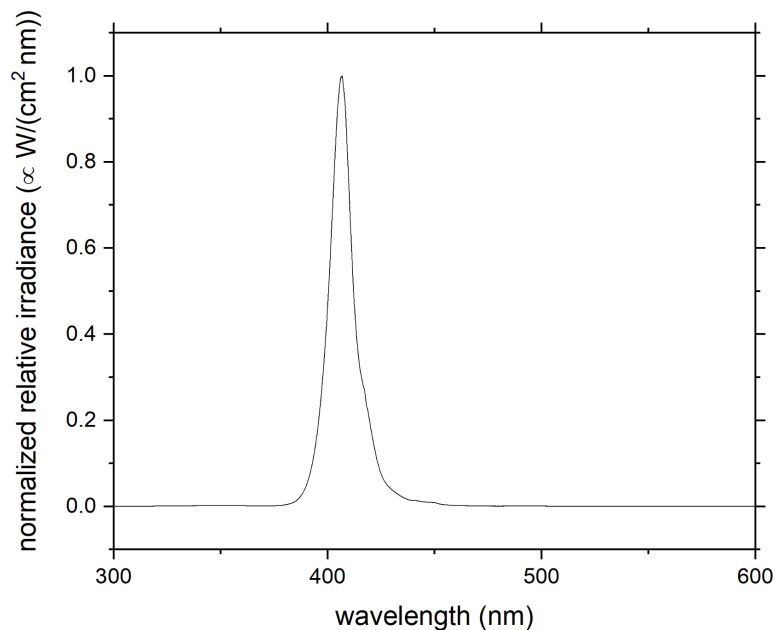


Figure 7: Spectrum of 405 nm LED light source collected from fiber coupled CCD spectrometer.

3.3. Printed feature characterization: Laser Scanning Confocal Microscopy

Laser Scanning Confocal Microscopy (LSCM, Keyence VK2000) was used to determine the lateral dimensions of the Ronchi rulings. Scans were performed with 20X objective, auto-brightness adjustment. For basic thickness measurements, single-scan was used, whereas automatic dual-scan was used for profiles. The dual scan function performs a 2nd LSCM scan at different brightness to enhance sidewall measurement accuracy. LSCM provides a surface topographic map of the printed line features with excellent X, Y and Z resolution.

3.3.1. Cure depth measurement, cure profiles, working curve

The cure depths were measured by 3-point plane fitting the LSCM data to the coverglass substrate, then measuring the maximum height on 20 separate profiles along the feature's length (**Figure 8**). The 20 maximum height values were averaged to obtain a mean maximum height. These mean maximum height measurements were repeated for 3 replicate prints. Working curve measurements utilized the P1 pattern prints (25 LPPI) with an expanded range of exposure duration (from 0.5 s to 1.5 s) (**Figure 9**). With the 500 μm photomask, the resultant prints are considered large enough to be bulk-like in their cure.

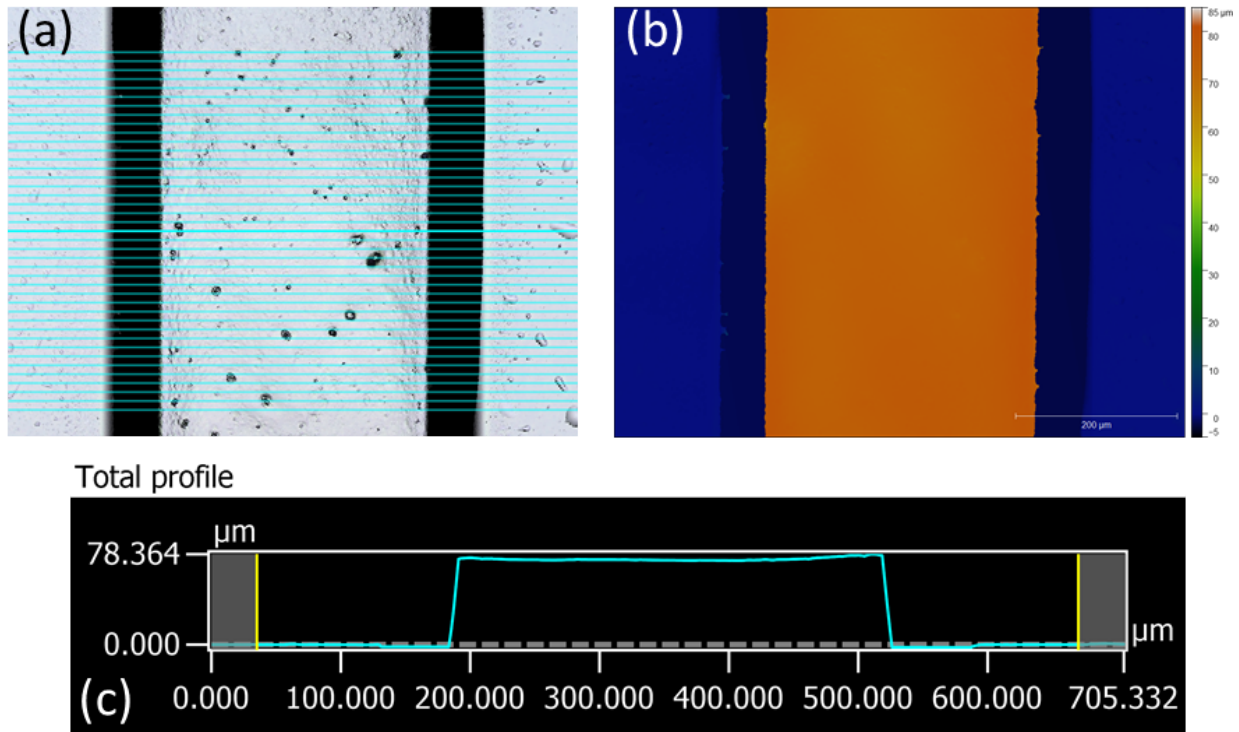


Figure 8: (a) Laser scanning confocal microscopy of an exemplary 25 LPPI feature (resin 4, 0.7 s exposure) indicating 20 line profiles over which average cure depth was measured. Dark regions are feature sidewalls, which were not captured in this scan. (b) Height map of exemplary feature. (c) Also shown is a profile of the feature, which should be predicted for the smaller features in (CHAL-AMB2022-07-03). The solution profiles will be averaged over at least 3 prints to establish mean value and uncertainty.

The measured working curve is provided here for convenience. Data are plotted as cure depth vs exposure on Linear-Log scale.

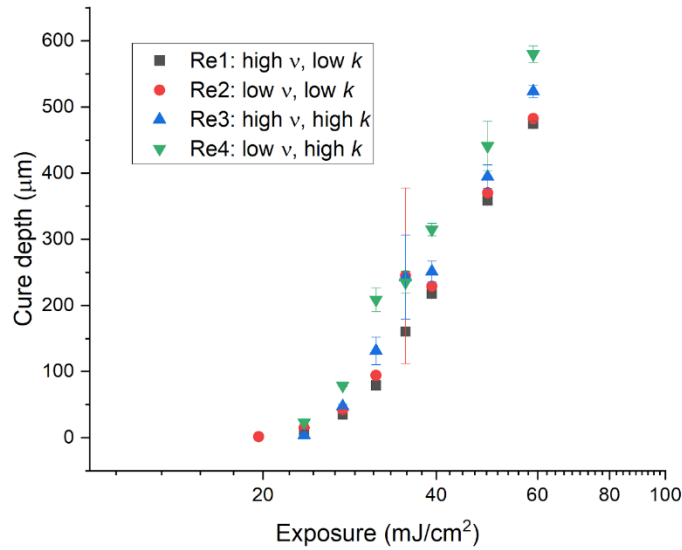


Figure 9: Measured working curve (cure depth vs exposure) for the 4 resins with 25 LPPI photomask, obtained from LSCM.

Table 8: Working curve raw data.

| Exposure duration (s) | Exposure (mJ/cm ²) | Re1 cure depth (μm) | St Dev (μm) | Re2 cure depth (μm) | St Dev (μm) | Re3 cure depth (μm) | St Dev (μm) | Re4 cure depth (μm) | St Dev (μm) |
|-----------------------|--------------------------------|---------------------|-------------|---------------------|-------------|---------------------|-------------|---------------------|-------------|
| 0.5 | 19.65 | No print | | 1.6 | 0.5 | No print | | No print | |
| 0.6 | 23.58 | 10.7 | 0.6 | 14.8 | 0.4 | 3.8 | 0.7 | 22.9 | 0.5 |
| 0.7 | 27.51 | 34.8 | 1.1 | 41.8 | 0.7 | 47.5 | 2.5 | 78.7 | 1.7 |
| 0.8 | 31.44 | 79.0 | 3.7 | 94.3 | 4.7 | 131.3 | 20.8 | 208.6 | 17.9 |
| 0.9 | 35.37 | 160.2 | 3.7 | 244.8 | 132.8 | 242.6 | 63.5 | 234.9 | 16.7 |
| 1 | 39.3 | 217.4 | 2.3 | 229.1 | 2.7 | 251.2 | 15.9 | 314.7 | 9.6 |
| 1.25 | 49.125 | 358.4 | 2.0 | 370.3 | 1.0 | 394.7 | 18.0 | 441.0 | 37.3 |
| 1.5 | 58.95 | 474.3 | 4.3 | 482.5 | 1.5 | 523.5 | 9.2 | 580.0 | 12.8 |

4. Description of Benchmark Challenge Problems

4.1. CHAL-AMB2022-07-OP

The optical profile challenge asks the modelers to predict, at the print plane, the intensity profile (intensity in mW cm^{-2} versus lateral position) of the photopattern for each of the seven photomasks. The modelers may choose to use this best prediction in their cure predictions, or they may simplify the prediction as they see fit. The measured optical profiles cannot accurately capture the expected high contrast of the chrome photomask, thus verification will be restricted to the maximum intensity and full width half max (FWHM). The solution score for this challenge problem will be based on total RMS error between predicted and measured maximum intensity and FWHM.

4.2. CHAL-AMB2022-07-CD

The cure depth challenge asks the modelers to predict the cure depth, as measured by LSCM at the center of the feature, as a function of photomask linewidth, exposure duration, and resin type. The resins exhibit varying degrees of reactivity and viscosity, and thus the interplay between reaction and species diffusion can be expected to differ in the different resins, which may affect cure depth.

Solutions shall be judged on both a relative and absolute basis.

The resin dependent relative solution will account for 30 % of the score and will be determined by the minimum RMS error in determining the cure depth ratios between the 4 resins at 0.5 s exposure duration for the 200 LPPI mask.

The pattern size dependent relative solution will account for 30 % of the score and will be determined by the minimum RMS error in determining the cure depth ratios for resin 2 (low viscosity, low reactivity) resins at 0.5 s exposure duration for each of the masks.

The absolute solution, worth the additional 40 % of the score, will consider the total RMS error between modeled cure depth and measured cure depth for all 4 resins, all 4 exposure durations and 6 photomasks (from 50 LPPI to 300 LPPI).

4.3. CHAL-AMB2022-07-PP

The print profile challenge asks the modelers to predict the cross-sectional profile (i.e. thickness versus lateral position) of the printed features for each of the 4 resins, 4 exposure durations and 6 photomasks (from 50 LPPI to 300 LPPI).

The solutions will be evaluated on a per resin basis, with each resin worth 25 % of the total score, and each evaluated as the total RMS error between the modeled and measured profiles.

5. Description and Links to Associated Data

All data available to support the AMB2022-07 challenges are contained in the “Resin properties, light engine calibration, and calibration print dimensions (AMB2022-07)” dataset available here:

<https://doi.org/10.18434/mds2-2597> .

New data files, updates, and/or changes to download URLs may be made periodically. Users should refer to the README text file which will record all updates. Additionally, the NIST Public Data Repository (PDR) undergoes frequent updates. If file downloads fail or are unavailable, users should wait several hours before contacting the technical support listed on the AMB2022-07 dataset webpage.

5.1 File Naming Convention

All measurement data files followed a similar naming convention to the specimen naming convention. For the master compressed file names found in the data repository, the names have been consolidated for clarity. A description of the naming conventions is found below, with specific examples pertaining to each dataset listed in subsection format. We note that for all measurements where a portion of the designated filename structure is not used (e.g., not all experiments required resin), 'none' has been used in its place (as will be demonstrated below).

Master compressed file naming convention:

AMB2022-07_{measurement}_Challenge.zip

AMB2022-07_{measurement}_Solution.zip

Measurement file naming convention:

AMB2022-07_{measurement}_{experiment date}_{resin used}-{v}{k}_{Ronchi ruling used}_{exposure time}_{specimen slide number}_{row number}_{column number}_{replicate}

5.1.1. Fourier Transform Infrared Spectroscopy

Measurement name: FTIRVP

Master compressed file names:

AMB2022-07_FTIRVP_Challenge.zip

AMB2022-07_FTIRVP_Solution.zip

Measurement file name example for an FTIR experiment done on April 18th, 2022 using Resin 1 (high viscosity, low reactivity) and is the first replicate:

AMB2022-07_FTIRVP_2022-04-18_Re1-HL_none_none_none_none_none_n1

5.1.2. Rheometry

Measurement name: RheoVP

Master compressed file names:

AMB2022-07_RheoVP_Challenge.zip

AMB2022-07_RheoVP_Solution.zip

Measurement file name example for a rheometry experiment done on April 18th, 2022 using Resin 1 (high viscosity, low reactivity) and is the first replicate:

AMB2022-07_RheoVP_2022-04-18_Re1-HL_none_none_none_none_none_n1

5.1.3. Atomic Force Microscopy

Measurement name: AFMVP

Master compressed file names:

AMB2022-07_AFMVP_Challenge.zip

AMB2022-07_AFMVP_Solution.zip

Measurement file name example for an AFM experiment done on April 18th, 2022 using a 25 LPPI Ronchi ruling and is the first replicate:

AMB2022-07_AFMVP_2022-04-18_none_25LPPI_none_none_none_none_n1

5.1.4. Laser Scanning Confocal Microscopy

Measurement name: LSCMVP

Master compressed file names:

AMB2022-07_LSCMVP_Challenge.zip

AMB2022-07_LSCMVP_Solution.zip

Measurement file name example for a cure depth LSCM experiment done on April 18th, 2022 using Resin 1 (high viscosity, low reactivity), a 25 LPPI Ronchi ruling, a 0.6 s exposure time, is slide number 3, is located in row 2, column 3, and is the first replicate:

AMB2022-07_LSCMVP_2022-04-18_Re1-HL_25LPPI_0600ms_3_r2_c3_n1

Measurement file name example for a photomask dimension LSCM experiment done on April 18th, 2022 using a 25 LPPI Ronchi ruling and is the first replicate:

AMB2022-07_LSCMVP_2022-04-18_none_25LPPI_none_none_none_none_n1

5.1.5. Radiometry

Measurement name: RadVP

Master compressed file names:

AMB2022-07_RadVP_Challenge.zip

AMB2022-07_RadVP_Solution.zip

Measurement file name example for a radiometry experiment done on April 18th, 2022 using an open aperture and is the first replicate:

AMB2022-07_RadVP_2022-04-18_none_none_none_none_none_none_n1

5.1.6. Beam Profilometry

Measurement name: BeamVP

Master compressed file names::

AMB2022-07_BeamVP_Challenge.zip

AMB2022-07_BeamVP_Solution.zip

Measurement file name example for a beam profilometry experiment done on April 18th, 2022 using an open aperture and is the first replicate:

AMB2022-07_BeamVP_2022-04-18_none_none_none_none_none_none_n1

Measurement file name example for a beam profilometry experiment done on April 18th, 2022 using a 25 LPPI Ronchi ruling and is the first replicate:

AMB2022-07_BeamVP_2022-04-18_none_25LPPI_none_none_none_none_n1

5.1.7. Optical Spectroscopy

Measurement name: OptSpecVP

Master compressed file names:

AMB2022-07_OptSpecVP_Challenge.zip

AMB2022-07_OptSpecVP_Solution.zip

Measurement file name example for an optical spectroscopy experiment done on April 18th, 2022 and is the first replicate:

AMB2022-07_OptSpecVP_2022-04-18_none_none_none_none_none_none_n1

5.8. Digital Caliper (slide thickness)

Measurement name: CaliperVP

Master compressed file names:

AMB2022-07_CaliperVP_Challenge.zip

AMB2022-07_CaliperVP_Solution.zip

Measurement file name example for a slide thickness experiment done on April 18th, 2022, is slide number 3, and is the first replicate:

AMB2022-07_CaliperVP_2022-04-18_none_none_none_3_none_none_n1

6. References

N/A

7. Disclaimer

The National Institute of Standards and Technology (NIST) uses its best efforts to deliver high-quality copies of the AM Bench database and to verify that the data contained therein have been selected on

the basis of sound scientific judgment. However, NIST makes no warranties to that effect, and NIST shall not be liable for any damage that may result from errors or omissions in the AM Bench databases.

Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by NIST, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.