AMB2025-09 Benchmark Measurements and Challenge Problems

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.

Challenge Problem

AMB2022-09: Vat photopolymerization cure depth samples fabricated on a methacrylate-functionalized microscope slide

Cure depth dependence on resin composition and radiant exposure (CHAL-AMB2025-09-CD): Cure depth, and subsequent critical cure energy (E_c) and depth of light penetration (D_p), dependence on resin composition and radiant exposure.

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1. Overview and Basic Objectives

This Photopolymer AM Bench-2025 Challenge is to predict the cure depth versus radiant exposure (often called dose) of prototypical resins with varying monomer functionality and photoabsorber type, for irradiation with narrow-bandwidth (< 5 nm) 385 nm or broad-bandwidth (> 10 nm) 405 nm ot 385 nm light. Fluorescing and non-fluorescing absorbers will be considered. Specimens will be large enough such that size-dependent effects are negligible. Modelers will be provided with reactivity and thermophysical

property data for the resins as well as radiometric data for the light sources. Modelers will predict the detailed working curve(s) as well as extracted critical energy Ec and depth of light penetration Dp for 8 conditions (2 monomers, 2 photoabsorbers, 2 light sources). The primary objective is to determine if and how the presence of a fluorescing and non-fluorescing absorber affect cure depth. Ultimately, the improved understanding and prediction of these relations will enable more repeatable part fabrication.

Experimental data for model calibration and challenge comparison is provided through resin characterization (e.g. real-time Fourier transform infrared spectroscopy and ultraviolet-visible spectroscopy) and system calibration (e.g., radiometry and spectroscopy). 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

All resins were composed of well-studied and widely-used monomers and photoabsorbers with a single, also-ubiquitous photoinitiator for vat photopolymerization additive manufacturing to ensure modelers had an ample body of literature to draw from.

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

Acrylate Monomers:

- Trimethylolpropane triacrylate (TMPTA, Sigma Aldrich, CAS# 15625-89-5, Mw = 296.32 g/mol)
- 1,6-Hexanediol diacrylate (HDDA, Sigma Aldrich, CAS# 13048-33-4, Mw = 226.27)

Photoabsorbers:

- Sudan I (Sigma Aldrich, CAS# 842-07-9, Mw =248.28 g/mol)
- 2,5-Bis(5-tert-butyl-benzoxazol-2-yl)thiophene (BBOT, Sigma Aldrich, CAS# 7128-64-5, Mw=430.56 g/mol)

Photoinitiator:

- Diphenyl(2,4,6-trimethylbenzoyl)-phosphine Oxide (TPO, Sigma Aldrich, CAS# 75980-60-8, Mw = 348.38 g/mol)

Many common 3D printers use either a 405 nm and 385 nm light source to initiate photopolymerization so TPO was selected as the photoinitiator for these formulations and BBOT and Sudan 1 were selected as photoabsorbers.

Each resin was composed of 30 g base monomer (either TMPTA or HDDA), to which 0.1 wt% and 0.4 wt% of photoabsorber (either BBOT or Sudan 1) and photoinitiator (TPO) were added, respectively. To ensure each resin was sufficiently mixed, each resin was placed on a vortexer at 3200 rpm for 1 minute. The vials

were then placed into an oven at 40 degrees C for 1 hour. After 1 hour, each vial was placed onto a vortexter at 3200 rpm for an additional minute.

Table 1: Resin composition broken down into respective components

		HDDA (wt%)	TMPTA (wt%)	TPO (wt%)	BBOT (wt%)	Sudan 1 (wt%)
Fluorescing absorber	Resin 1	99.5	0	0.4	0.1	0
	Resin 2	0	99.5	0.4	0.1	0
Non-fluorescing absorber	Resin 3	99.5	0	0.4	0	0.1
	Resin 4	0	99.5	0.4	0	0.1
Control (no absorber)	Resin 5	99.6	0	0.4	0	0
	Resin 6	0	99.6	0.4	0	0

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.

2.2 Light Engine

The light engine is comprised of a high-power, collimated 405 nm or 386 nm LED (Thorlabs, SOLIS-405C or SOLIS-385C), a commercially-available 405 nm narrow bandpass filter (Semrock LL01-405-12.5) or a custom 385 nm narrow bandpass filter (Semrock), a custom steel aperture with diameter of 2.47 mm which were mounted with the sample setup including a methacrylated glass slide, the resin in question, and a silicone isolator illustrated in **Figure 1**.

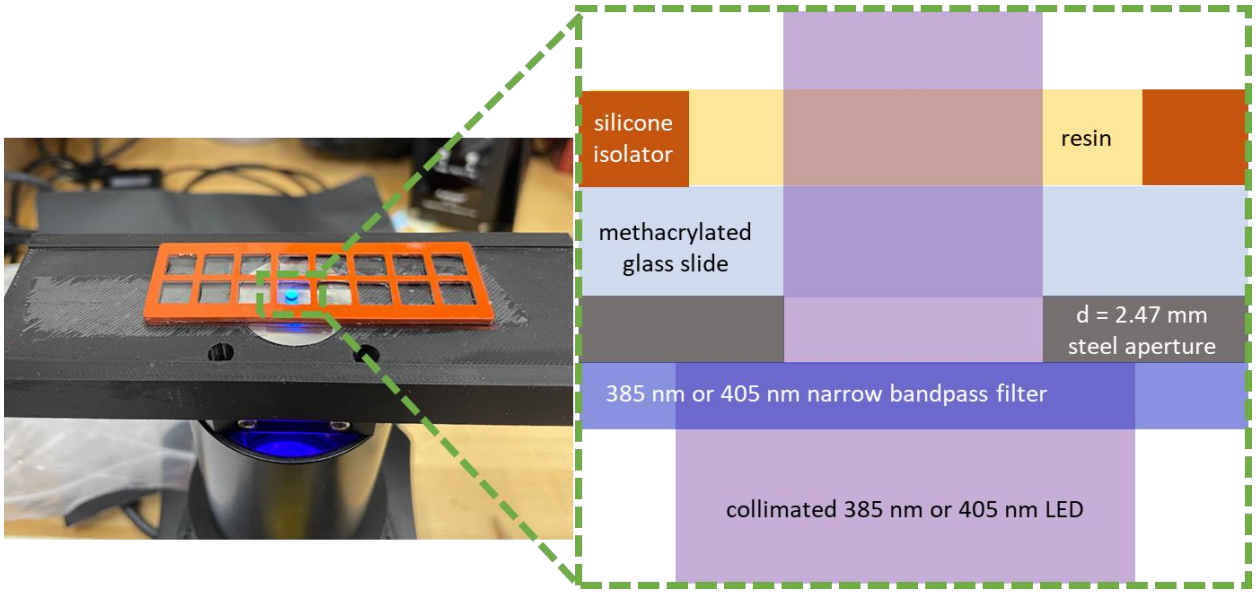

Figure 1: Projection light engine used for all cure depth exposures with an expanded schematic of the sample setup.

2.2.1 Light source

A high-power, collimated 405 nm or 385 nm LED was used for all photopatterning (Thorlabs, SOLIS-405C or SOLIS-385C). The light engine intensity and bandwidth were measured using radiometry and photospectroscopy, respectively.

2.2.2 Aperture

A custom 2.47 mm steel aperture was used for all cure depth experiments.

2.3 Printing process

All samples were printed using the following procedure:

1. Place silicone isolator (1 mm tall) onto methacrylated slide and mount onto light engine
2. Align first isolator well to the light engine
3. Deposit ~150 uL resin into each isolator well (enough to produce positive convex surface tension between isolator well boundaries)
4. Expose for designated radiant exposure
5. Translate glass slide + isolator + resin sample to next, fresh isolator well
6. Repeat Steps 4 and 5 until all 16 cure depth exposures are complete

2.4 Post-Processing

All samples were post-processed using the following procedure:

1. Remove sample from light engine mount
2. Remove isolator using tweezers (being careful to not agitate the exposed regions within the wells)
3. Place sample onto dripping station to allow gravity to remove unreacted monomer for 24 hours

4. While holding glass slide at a 45 degree angle, deposit 250 uL of isopropyl alcohol onto slide to remove the remaining excess monomer without damaging or delaminating the samples
5. Place samples into UviTron light oven for 180 s.

2.5 Specimen Naming Convention

All specimens were named using the following convention:

AMB2022_{experiment date}_{resin used}

Here, AMB2022 is for Additive Manufacturing Benchmark 2022 and the bracketed regions indicate a specific, unique identifier for the specimen. For example, a specimen made on February 18, 2025 using Resin 1 would go as follows:

AMB2025_2025-02-18_Re1

3. Measurement Descriptions

The AMB2025-09 benchmark elucidates fundamentals of vat photopolymerization additive manufacturing by measuring the shape of patterned features subject to varying exposure duration, light engine wavelength, and resin characteristics. The calibration data seek to provide the most essential properties to predict the cure depth and corresponding working curve output of critical cure energy and penetration depth.

Resin Characterization

- Fourier transform infrared spectroscopy measurements of polymer conversion vs exposure duration for a constant exposure intensity
- Ultraviolet-visible spectroscopy was conducted to determine the absorption profile of each resin

Light Engine Characterization

- Radiometric measurements of the filtered LED light sources to determine optical power
- Photospectrometry measurements of the filtered LED light sources to determine optical bandwidth

Printed Feature Characterization

- Cure depth measurements were conducted utilizing an optical coherence tomography system to ensure non-destructive measurement of the samples

3.1 Resin Characterization

3.1.1 Resin Reactivity: Real-Time Fourier Transform Infrared Spectroscopy (RT-FTIR)

Real-Time Fourier-Transform Infrared Spectroscopy was conducted using a Thermo Fisher Nicolet 6700 FT-IR. The following acrylate peaks at 1630 cm^{-1}, 1400 cm^{-1}, and 800 cm^{-1} were monitored for double bond conversion to determine the kinetics of curing.

All samples were prepared between two salt plates to ensure sensitivity to relevant FTIR acrylate absorption peak and were optically thin (<2 um) to ensure the absorptive properties were minimized. Resins were then placed in the spectrometer and the kinetic runs were started, monitoring the associated acrylate peaks with a sampling interval of 0.52 s and a resolution of 4 cm^{-1}. All RT-FTIR experiments were conducted employing the same light engine used for all 405 nm and 385 nm cure depth exposures.

3.1.2 Resin Absorptivity: Spectrophotometry

All resin absorptivity was determined using an Invitrogen Nanodrop One Spectrophotometer (Thermo Scientific). Approximately 1 µL of resin was dispensed onto the sampling region, the detection arm was lowered, and then an absorption spectrum was taken as shown in Figure 2.

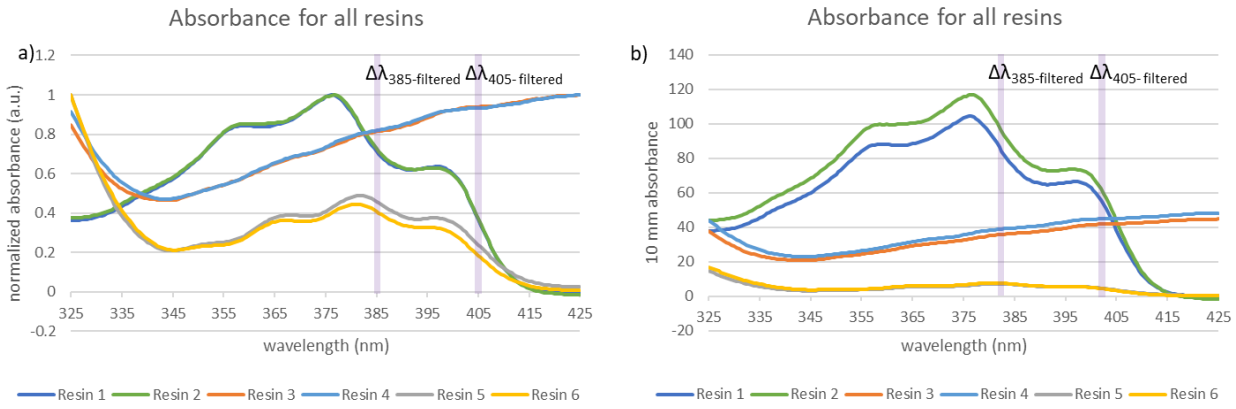

Figure 2: Absorption spectra for all 6 resin formulations.

3.1.3 Resin Photo-Thermal Properties: Photo-DSC

Each resin's photo-thermal properties were determined using a TA Instruments Q2000 Photo-DSC. Due to the low transmission in the light-guides for the instrument, the maximum intensity delivered to the samples for the 385 nm and 405 nm light sources were 1.158 mW cm-2 and 1.615 mW cm-2, respectively.

3.2 Light engine characterization:

3.2.1 Light engine intensity calibration: Radiometry

Radiometry (ThorLabs, PM100D console, S170C photodiode) was used to determine the optical power of the 405 nm LED and 385 nm LED at the photopatterning plane. The optical power at the sample plane was 8 mW/cm^2 for all 405 nm exposed samples and 8.8 mW/cm^2 was chosen for all 385 nm exposures to mirror recent interlaboratory cure depth studies. 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 3**). 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 3** for all manufacturer-supplied power meter specifications.

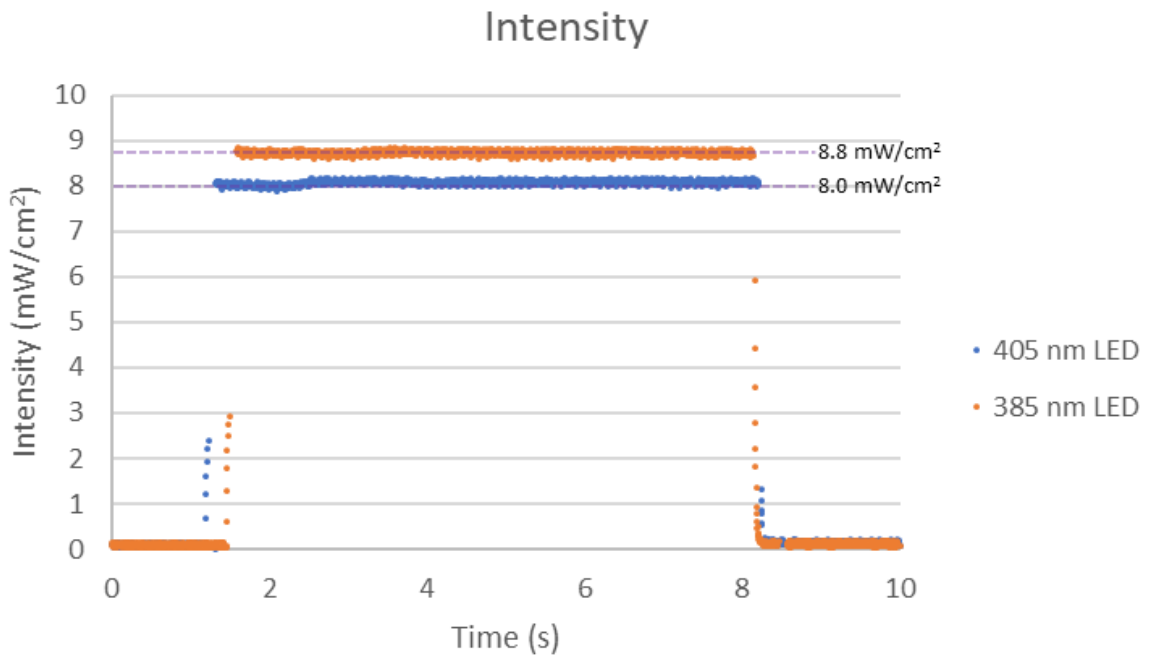

Figure 3: Optical intensity for both the 405 nm and 385 nm filtered LED sources.

Table 3: 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 or 385 nm

3.2.2 Light spectrum: Photospectrometry

The light engine photospectrum was calibrated using an Avantes spectrometer. See Figure 3 for all bandwidth information for both sources after the paired narrow bandpass filters.

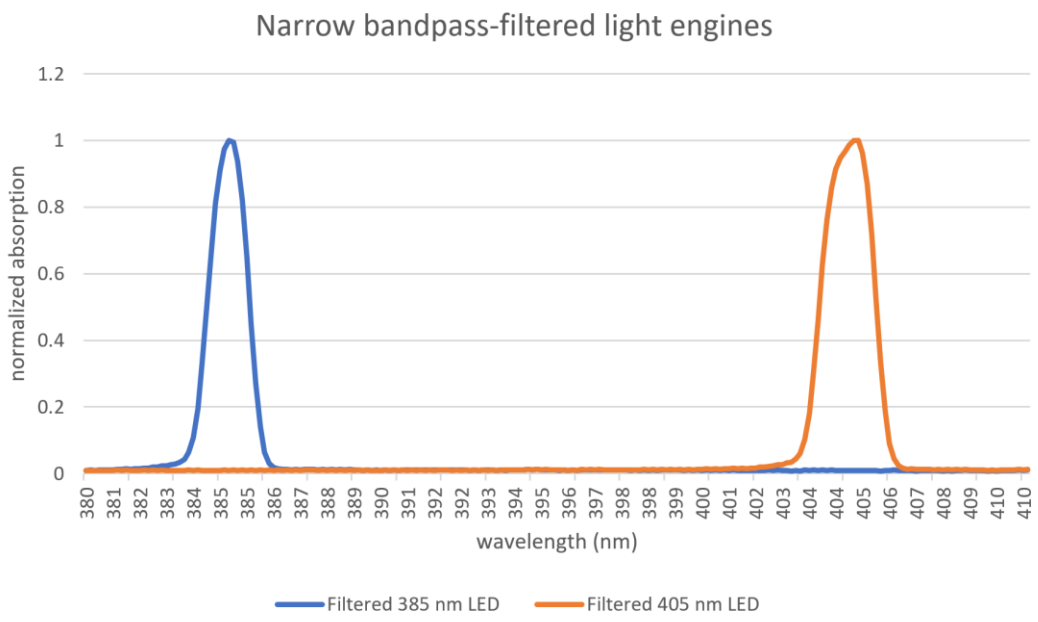

Figure 4: Plot of both the 385 nm and 405 nm light source bandwidths after filtering with their respective narrow bandpass filter.

3.3 Cure depth measurement

Cure depth measurements were conducted utilizing a ThorLabs optical coherence tomography system. Due to the varied reaction kinetics of each resin, the exposure times varied from 1 s to 12 s.

4. Description of Benchmark Challenge Problems

4.1 CHAL-AMB2025-09-CD

The cure depth challenge asks the modelers to predict the critical cure energy (Ec) and penetration depth (Dp) from the Jacobs equation, as measured by optical coherence tomography, as a function of exposure source, exposure duration, and resin type. The resins exhibit varying absorptive properties and functionalities, and thus different fluorescing and non-fluorescing absorptive species in the different resins may affect cure depth.

Solutions shall be judged in two sections using as much as or as little calibration data as you choose:

1) This resin-dependent relative solution will account for 60 % of the score and will be determined by the minimum RMS error in predicting the critical cure energy (Ec) for each resin and exposure source.

2) This resin-dependent relative solution will account for 40 % of the score and will be determined by the minimum RMS error in predicting the critical cure energy (Dp) for each resin and exposure source.

1. Description and Links to Associated Data

All data available to support the AMB2025-09 challenges are contained in the “Vat photopolymerization cure depth samples fabricated on a methacrylate-functionalized microscope slide (AMB2025-09)” dataset available here: NIST MIDAS Database

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 AMB2025-09 dataset webpage.

2. References

[1] Boddapati, A., Rahane, S.B., Slopek, R.P., Breedveld, V., Henderson, C.L. and Grover, M.A., 2011. Gel time prediction of multifunctional acrylates using a kinetics model. Polymer, 52(3), pp.866-873.

[2] Dobson, A.L. and Bowman, C.N., 2024. A Comprehensive, Multidimensional First-Principles Model for Free-Radical Photopolymerizations in Bulk and Thin Films. Advanced Functional Materials, 34(22), p.2312607.

[3] Niederst, L., Allonas, X., Morone, M. and van den Branden, S., 2024. Employing Singlet-Singlet Energy Transfer for Boosting the Reactivity of Type I Photoinitiators in Radical Photopolymerization. Angewandte Chemie International Edition, 63(52), p.e202412625.

[4] Kowsari, K., Zhang, B., Panjwani, S., Chen, Z., Hingorani, H., Akbari, S., Fang, N.X. and Ge, Q., 2018. Photopolymer formulation to minimize feature size, surface roughness, and stair-stepping in digital light processing-based three-dimensional printing. Additive Manufacturing, 24, pp.627-638.

[5] Jacobs, P.F., 1992. Rapid prototyping & manufacturing: fundamentals of stereolithography. Society of Manufacturing Engineers. pp. 29-35.