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UV+EB TECHNOLOGY

Increasing Adoption of Photopolymer Additive Manufacturing

Premiere PAMA Mini Mag for 3D Printing/Additive Manufacturing

RADTECH
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PAMA

PHOTOPOLYMER ADDITIVE MANUFACTURING ALLIANCE

Welcome to the first PAMA "mini" magazine!

PAMA was formed as a collaboration between the National Institutes of Standards and Technology (NIST) and RadTech, a nonprofit trade association, as additive manufacturing (AM) begins to accelerate on its journey into the manufacturing supply chain. The shift from advanced prototyping toward the use of AM in the production of commercial and consumer goods only has begun to gain traction. As AM hardware and materials become more commonplace in industrial markets, we feel it is important that our industry begins to self-regulate to keep AM users (and their end customers) safe while at the same time developing shared language, methodologies and testing standards that will enable customers to make well-informed decisions when comparing different technologies.

PAMA doesn't necessarily target specific industries, but instead is a multi-stakeholder organization which spans across the length of the entire AM supply chain (i.e., chemical producers, resin formulators, hardware OEMs, print shops, end-users/customers) and across multiple industry verticals (aerospace, automotive, dental, medical device, construction materials, etc.). This is a unique differentiator between PAMA and other classical industry organizations that are working to organize best practices targeting one specific process or vertical.

As you will see in these pages, PAMA's various technical committees are busy developing activities and on-boarding new members to help ensure we are targeting the right questions – and we are excited to have nearly 30 organizations already represented in our group. We hope that you enjoy this mini-magazine, and we look forward to including you in our work advancing the photopolymer additive manufacturing space!

David Walker

David A. Walker, PhD
Executive Chairperson
Photopolymer Additive Manufacturing Alliance (PAMA)

Premiere PAMA Mini Mag

Want to Get Involved in PAMA? Read On.

The Photopolymer Additive Manufacturing Alliance (PAMA) is a collaboration between the National Institutes of Standards and Technology and RadTech, a 501-c-6 nonprofit trade association with over 500 members, dedicated to the advancement of ultraviolet and electron beam technologies. PAMA is an alliance of industry, academic, governmental and NGO organizations with the goal of bringing commonly accepted standards and practices to the field of photopolymer additive manufacturing (PAM). Through voluntary self-regulation and work in collaboration with government agencies, PAMA seeks to help ensure PAM technologies are adopted in a safe and responsible manner.

The activities of PAMA are guided by a roadmap prepared by NIST, developed to offer strategic guidance for PAM stakeholders and to fuel collaboration among the PAM community to accelerate innovation. The roadmap is the final report of a workshop held in October 2019 at the NIST in Boulder, Colorado, with input from research, industry and regulatory communities on the PAM research and development agenda.

PAMA Executive Advisory Board Chair and End User Board Members

David A. Walker, PhD
Chair of the PAMA Board; CEO & Founder, Prismatic Manufacturing

Vince Anewenter
Director, Rapid Prototyping Center Consortium, Milwaukee School of Engineering

Michelle Bockman
Chief Simplification and Transformation Officer, Stanley Black & Decker, Inc.

Carl Dekker
President, Met-L-Flo, Inc.

Working Committees Drive PAMA Activities

Working committees bring together stakeholders throughout the PAM community to collaborate, recommend and take action on specific areas within PAMA's range of interest.

Materials Characterization Working Committee

The PAMA Materials Characterization (MC) Committee serves the interests of stakeholders by ensuring that the necessary measurement infrastructure exists and is disseminated to characterize resins, processing, part-resolution, part-performance and additional materials characteristics to ensure customer needs are met.

Jason P. Killgore, PhD, NIST
Chair of PAMA Materials Characterization Committee

Mission: The mission of this committee will be accomplished with a significant emphasis on outreach and education whereby the MC committee will publish documents and videos to engage characterization experts in PAM and related fields to ensure PAMA membership is aware of best practices and emerging methods to advance practical and scientific understanding of PAM. The MC committee will also perform stake-holder-suggested novel research studies to develop new characterization in areas where existing tools are inadequate.

Current Objectives:

- Evaluating and disseminating best practices for working curve measurements
- Assessment of resin lifetime and influence on part fidelity and performance
- Measuring essential resin parameters for the modeling community

Hardware Characterization Working Committee

The PAMA Hardware Characterization Committee's mission is to enable the transformative value of this technology by enlisting PAMA experts across industry, academia and government to understand current limitations to PAMA hardware and how the community can overcome these barriers. The committee also serves as a pre-competitive epicenter for open discussion regarding current and future advancements across the PAMA field to develop novel characterization instruments that readily can be implemented into industry settings.

Callie I. Higgins, PhD, NIST
Chair of PAMA Hardware Characterization Committee

Mission: Photopolymer AM characterization has the unique opportunity to innovate on novel characterization methods due to the complex, diverse nature of the potential applications paces. As photopolymer AM is a founding father of AM, this community has a wealth of experts with deep understanding of this technology that, until now, has yet to be tapped into sufficiently. The committee aims to provide a conduit for researchers and industry professionals alike to learn about current characterization advancements and inform new research directions.

Current Objectives:

- Identify hardware characterization parameters that are common throughout PAM and corresponding methods for characterization
- Educate the PAM community on current best practices for hardware characterization and listing of specifications
- Proselytize utility of characterization techniques for implementation across the industry
- Support ongoing research efforts and collaborations in PAMA hardware characterization through concerted networking events and educational workshops

Environmental, Health and Safety Working Committee

The PAMA Environmental, Health & Safety (EHS) Committee serves the needs of the broader Photopolymer Additive Manufacturing Alliance by focusing on current and emerging issues involving the safe, legal and responsible use of photopolymer raw materials, technology and equipment in additive manufacturing applications. Oversight of these critical 'components' of the AM industrial space is essential to promote healthy, robust and sustainable growth of the technology while avoiding concerns related to human health and the environment.

Michael Gould, Rahn
Chair of Environmental, Health and Safety Working Committee

Jeremy Smith, Nagase Specialty
Co-Chair

Mission: This mission will be accomplished by communicating information most relevant to health, safety and the environment when it becomes available. As information is collected and shared, the principal goal of the committee is to achieve a steady state of "self-regulation" wherein industry participants are open to suggestions and data which will guide best practices. The goals are to promote emerging PAM technology, foster innovation and avoid costly industry setbacks that may occur due to misunderstanding of regulations, and to review and scrutinize current practices in order to get broadest agreement on best practices.

Objectives:

- Collection and dissemination of emerging data that will affect the labeling and classification of photopolymer raw materials, e.g., monomers, oligomers, photoinitiators, stabilizers and additives
- Discussion and clarification of current regulations insofar as they affect critical materials in different parts of the US or global market
- Discussion of and/or crafting of guidelines for material use protocols, particularly for FDA and 'home hobbyist' applications outside of the industrial space
- Consideration of material testing that will promote acceptance of materials or to rebut poorly conducted studies that bring unfair scrutiny of photopolymer raw materials
- Coordination with legal resources to promote and achieve industry goals where regulatory or non-governmental entities oppose or obstruct fair and legal commerce

Government Partnerships & Regulation Committee

The PAMA Government Collaborations Committee explores the development and application of federal, state and regional government initiatives supporting photopolymer additive manufacturing (PAM) to advance or use the technology. The committee also aims to understand the effect of actions taken by governmental organizations on the industry production and subsequent implications for manufacturing competitiveness through an open forum with the manufacturing community.

Dianne Poster, PhD, NIST
Chair of Government Partnerships & Regulation Committee

Cameron Miller, PhD, NIST
Co-Chair

Mission: The committee is a collaborative collection of organizations from government, industry and academia sharing information on initiatives supporting or promoting strategic research and development to advance PAM technology. The committee aims to encourage a dialogue about the government's role and its goals for PAM and to collect and disseminate information on existing or newly developed standards and best practices, materials and engineering processes, and government resources and assistance programs.

Objectives:

- Review existing standards and best practices in PAM and evaluate new needs
- Review of government resources and assistance programs supporting PAM suppliers, equipment

page 6 ▶

◀ page 5

manufacturers and end-users

- Analyze the relationship between federal programs, regional/state programs and private programs supporting innovation in PAM
- Plan for needed initiatives and alternative investment opportunities in PAM
- Identify opportunities for advancing PAM technologies, investment approaches and international collaborations

Market Research Working Committee

Understanding the current state of the additive manufacturing market is paramount for stakeholders across the value chain to make informed business and research decisions. Photopolymer Additive Manufacturing (PAM) technology represents a burgeoning sector of this industry. The PAMA Market Research Committee aims to gather data related to PAM – including materials, print platforms, technologies and industry applications – and report this data in a succinct and actionable form.

Stephanie Benight, PhD, Tactile Materials Solutions
Chair of Market Research Working Committee

Mission: The committee is a collaborative collection of organizations from industry, government and academia focused on gathering information on PAM materials (e.g. volumes, prices, base chemistries, properties, etc.) applications of use and industries involved. A main goal of the committee is to produce a reliable summary of information focused on PAM, make this summary widely available and, in doing so, encourage innovation, sales, adoption and growth.

Objectives:

- Creation of a survey to collect PAM materials usage and industry application data
- Poll PAM stakeholders about futurist goals for additive manufacturing and pitfalls on the horizon that collaborative action can avoid.
- Hold regular meetings to discuss market feedback, progress on the above and real-world examples of PAM material utilization ◆

Leading Groups Recognize Value in PAMA Membership

With PAMA activities kicking off less than one year ago, PAMA is proud to have members from the following organizations:

3D Systems
allnex
Ameralabs
Arkema
Dymax**
Henkel Corp
IGM Resins
Lawrence Livermore National Laboratory
Met-L-Flo, Inc.
MicroTau
Mighty Buildings, Inc.
MIT
Miwon North America
Montana State University
NAGASE Specialty Materials NA, LLC
Osaka Organic Chemical Ind. Ltd **
PrintFoam
Prismatic Manufacturing
Rahn
Rapid Prototyping Consortium–MSOE
South Dakota School of Mines & Technology
Stanley X
TE Connectivity
University of Cincinnati
University of Denver
Vitro3D

***** Denotes PAMA Support as a Founding Member***

RadTech and PAMA Support the US Department of Energy's Industrial Heat Shot™: Ultraviolet Curing Included as a "Key Pathway"

RadTech, the association for UV+EB technologies, and PAMA, the Photopolymer Additive Manufacturing Alliance, support the new US Department of Energy (DOE) Industrial Heat Shot™ initiative. As excerpted from a late September 2022 US Department of Energy Release:

"The DOE has launched Industrial Heat Shot™, a new effort aimed at dramatically reducing the cost, energy use and carbon emissions associated with the heat used in industrial processes. This latest DOE Energy Earthshots Initiative™ seeks to develop cost-competitive solutions for industrial heat with at least 85% lower greenhouse gas emissions by 2035.

The Industrial Heat Shot™ initiative includes as a "key pathway" to achieve targets:
Innovate low- or no-heat process technologies: Develop new chemistry and emerging biotechnology processes to reduce heat demand, such as bio-based manufacturing, electrolysis, ultraviolet curing and advanced separations.

The Industrial Heat Shot™ will support the overarching strategy detailed in DOE's "Industrial Decarbonization Roadmap." The Roadmap emphasizes the urgency of deep decarbonization across the industrial sector and presents a staged research, development and demonstration (RD&D) agenda for industry and government that will deliver the technologies needed to dramatically reduce emissions, increase American manufacturing competitiveness and create high-quality jobs.

As part of DOE's commitment to building a decarbonized industrial sector of the future, the US is joining the Industrial Deep Decarbonization Initiative. Coordinated by UNIDO, this Clean Energy Ministerial global coalition is designed to stimulate demand for low-carbon industrial technologies.

The Industrial Heat Shot™ is an all-hands-on-deck effort across DOE to address the critical technical barriers to the development and widespread implementation of the cost-competitive, innovative technologies we need to fully decarbonize our economy and overcome the climate crisis. To learn more, read the Industrial Heat Shot™ fact sheet and visit the Energy Earthshots Initiative homepage."

RadTech and PAMA jointly plan to establish a task force to offer feedback to the DOE on the execution of the Industrial Heat Shot™ and participate in a DOE webinar that will provide more information on opportunities for collaboration and information-sharing. RadTech and PAMA members who would like to join our working group should contact Gary Cohen at *gary@radtech.org*.

PAMA Partnering with RadTech to Develop BIG IDEAS!

PAMA is a full partner in the development of RadTech's biennial BIG IDEAS for UV+EB Technology Conference, set for March 6-8, 2023, at the Wyndham Bayside in San Diego, California. The event focuses on the BIG IDEAS in the global space for UV/EB technology and will include 3D printing, additive manufacturing, UV LED, printing, automotive, data-driven materials and more. As a partner, PAMA members are working to develop presentations and sessions for the event to advance photopolymer additive manufacturing technologies. The BIG IDEAS conference offers a forum for industry to discuss the future of UV and EB technologies, informing the development of the science and application of photopolymers to enable tech advancement two years, five years and 10 years down the road. Learn more at *https://bigideasconference.com/*. ◆

RadLaunch Spotlights PAM Applications

RadLaunch, a unique idea accelerator for UV/EB start-ups, students and innovators, presented its 2022 class at RadTech 2022, in Orlando, Florida. "RadLaunch serves as critical support to companies working in the UV/EB space as they take their first steps on the long and challenging road to commercialization," said Mike Idacavage, co-chair of RadLaunch. "Many companies that have an excellent idea and perhaps early positive lab results are missing the contacts and network that will take them past the concept phase. One of the most valuable things that RadLaunch offers is an introduction to member companies, such as material and equipment suppliers and end users, that can furnish support and guidance."

The RadTech RadLaunch 2022 award winners honored in Florida are featured here. Applications for the 2023 RadLaunch class now are being accepted at *www.radlaunch.org*.

Camila Uzcategui, PhD,
CEO and Co-Founder, Vitro3D

Volumetric 3D-Printed Dental Aligners: Vitro3D

Making dental aligners at the point of care offers faster treatment and better patient outcomes using a novel and easy-to-use volumetric 3D printing method. The dental aligner market is the biggest user of UV-based 3D printing/additive manufacturing. However, current manufacturing of 3D-printed dental molds to thermoform aligners is slow, wasteful and inefficient. This solution uses a new volumetric 3D-printing method which is more sustainable, 100x faster and produces more accurate aligners for improved patient outcomes. Ultraviolet (UV) photopolymerization is at the core of the technology being developed by Vitro3D. By making use of the rapid reactions that take place during exposure to UV energy, along with the wide variety of UV photocurable materials available, Vitro3D is leading the development work on the next generation of additive manufacturing – volumetric printing. Potentially, all current suppliers of ortho aligners would benefit from the successful commercialization of this technology.

Johnny Hergert, PhD, CTO,
Founder and Materials Scientist, Vitro3D

Custom Bolus for Radiation Therapy: BC Cancer Agency, Centre for the North

Centre for the North is working on a modern process for fabricating tissue replacement (bolus) used during radiation therapy. A bolus is used to modify a patient's radiation treatment for cancers close to the skin, ensuring the tumor receives the correct dose. The historical method is to manually cut out plastic, silicone, wax or other proprietary tissue-equivalent materials onto a patient to conform with the planned treatment field. While Centre for the North successfully has been using FDM 3D printers to produce a better bolus than legacy methods, these printers require a considerable amount of labor and skill. Ultraviolet (UV) SLA 3D printers are easier to understand and operate than FDM printers and have the potential to speed up the process, while creating a bolus that is easier to clean and sterilize for radiation therapy patients in BC.

Nathan Smela,
BC Cancer Agency

Whytneigh R. Duffie, PhD Candidate in Chemical and Biological Engineering, South Dakota School of Mines & Technology

Special Academic Award: Disappearing 4D Advanced Materials

Submitted by South Dakota School of Mines & Technology's Whytneigh R. Duffie, PhD candidate in Chemical and Biological Engineering, and Travis W. Walker, associate professor in Chemical and Biological Engineering, this novel chemical technology platform provides sustainable, biocompatible and high-resolution photocurable resins that enable controlled and predictable disappearing (biodegradability) of materials, while also retaining mechanical integrity of the material. Potential applications include opportunities for precision casting of parts that are difficult to machine; end-of-life disposal of a part or device (e.g., drone, sensor) to prevent reverse engineering of sensitive technology; transient sensors; advanced reactor design; self-healing, sacrificial coatings; and medical devices (e.g., fracture fixation, tissue sealants, drug delivery). A key value proposition identified by potential customers with the Department of Defense is the UV curing of disappearing resins with a 3D printer – this increases material and combat readiness, shortens the supply chain and reduces costs associated with transportation and end-of-life disposal. UV serves as the source of polymerization, ensuring high-resolution and high-efficiency manufacturing. UV offers the ability for custom manufacturing on-demand via 3D printing to provide time-efficient and cost-savings processing that would not be possible otherwise. ◆

3D Printing for Boli in Cancer Radiation Treatment: FDM or MSLA?

By Liz Stevens, writer, UV+EB Technology

At BC Cancer – Prince George (Centre for the North), in Prince George, BC, Canada, researchers are conducting a comparison study of two additive manufacturing methods for creating the boli used during radiation treatments. If a patient needs treatment for cancer that is near the surface of the skin, it often is necessary to fabricate an anatomically customized, form-fitting bolus – a tissue-replacement mass – that is placed on the patient to optimize how the radiation plane strikes and penetrates the target area. Historically, materials such as plastic, silicone and wax, or commercial replacement tissue, have been used. With these methods, however, achieving a custom fit with an optimal shape has been problematic, time-consuming and can yield boli that are difficult to sterilize.

In their study, BC Cancer researchers are comparing fused deposition modeling (FDM) and masked stereolithography (MSLA) to see which method/technology/material combination will produce the best boli in an efficient, affordable and manageable way. *UV+EB Technology* talked with Nathan Smela, radiation therapy service technologist, to learn about BC Cancer's study, which earned a RadTech 2022 RadLaunch Award.

For either FDM or MSLA production of a bolus, the beginning steps are the same. "A CT scan is taken when a patient is first admitted," said Smela. "We then plan a radiotherapy treatment in our treatment planning software. The specs for the bolus automatically are generated and prescribed in that software. We extract the bolus prescription and convert the 3D mesh object that it describes into a STL file (the file format for 3D printing) and use another application to smooth the mesh."

For the printing step, said Smela, the group already had experience with FDM 3D printing. "But while BC Cancer – Prince George has been successfully using FDM printing on an open-air bed with polylactic acid (PLA) filament to produce a better bolus than legacy methods," he said, "a drawback of FDM it that it requires a considerable amount of labor and skill."

BC CANCER
Provincial Health Services Authority

The various consumer-grade FDM printers that Smela et al have used have a steep learning curve. "Operating and maintaining a FDM printer require significant training," said Smela. Operators must learn to calibrate filament rolls and do this for each and every roll, which requires temperature towers and stringing tests.

"The user also needs to ensure a consistent first layer," he continued, "and must avoid filament loading issues, watch for temperature probe issues and check for needed maintenance on belts and bearings." And all of this, Smela emphasized, doesn't include the important tests needed to finetune an FDM printer to its full potential, like calibrating pressure advance, extrusion width tests, testing for acceleration/jerk settings or performing updates to the firmware and slicing software.

Ultraviolet (405 nm) MSLA printers that work with a vat of liquid material are easier to understand and operate than a FDM printer, according to Smela. They also can offer about twice the print speed of an FDM printer and can be used to create a bolus that is easier to clean and sterilize for radiation therapy patients.

"FDM's printing speed is volumetric," Smela added, "meaning that if you double the number of objects to be produced during a single print run, you at least double the print time. With MSLA, the print time is based only on the longest dimension of any or all objects, regardless of the cubic volume of the object or multiple objects." In this case, two objects can be printed in the same time as one object, shaving off valuable print time that Smela can use for making test prints to find the best orientation for printing a bolus.

For this study, Smela is comparing two consumer-grade 3D printers, a $1,200 (CAD) consumer FDM printer vs. a $1,600 (CAD) consumer MSLA printer. While the costs of the printers themselves and their parts/maintenance consumables are comparable, MSLA-printed objects require more post-processing (wash and cure, and sterilization), which calls for additional hardware.

BC Cancer – Prince George (Centre for the North)

The ability to easily disinfect or sterilize a bolus is important since cancer therapy patients may have compromised immune systems or may have communicable illnesses. FDM printing of boli is far from ideal, hygienically-speaking. The process takes place in open air, and an object made from heated filament is a great host environment for pathogens.

"In order to print quickly on a FDM printer," said Smela, "we have to use large layer heights, but this leaves big cavities for microorganisms to thrive in. There also are tiny gaps internally in an FDM-produced object – more places for microorganisms to colonize." To address this problem, Smela wraps each bolus in plastic wrap and routinely cleans it with disinfectant. "An MSLA-produced resin bolus, on the other hand," Smela said, "can be printed with a smaller layer height. It also should have a completely filled interior, making disinfection faster and easier. And with MSLA printing, there also are sterilizable resin options."

Using 3D printing for medical use calls for careful attention to the type of filament or resin used. "The best patient bolus material," said Smela, "would be biocompatible, easy to print, nearly equivalent to water for radiation occlusion, have no odor, be antiviral/antibacterial or sterilizable, and be inexpensive. A super-soft material would be ideal – with a shore hardness in the 00 or 000 range – but the peeling forces of the MSLA process would make material that soft exceptionally difficult to handle and therefore is out of scope for this project." For FDM printing, PLA (polylactic acid) filament was used. Comparable biocompatible, rigid consumer-grade MSLA resin roughly is triple the cost of FDM filament, and this introduces another data point – material cost – to evaluate during the study.

Cost of materials, as well as cost of hardware and software, always is an important factor to consider when choosing between methods and technologies but, for BC Cancer – Prince George and this study, economy carried special weight. "We are operating on a very tight budget," Smela said. "The members involved with the study volunteer their time to ensure that we have enough budget to fully test FDM vs. MSLA printing." That leads the group to be creative with its MSLA post-processing equipment because the preferred cleaning and curing system is beyond the study's budget. To make do, Smela is using an ultrasonic cleaner and a UV LED lamp with a solar-powered turn table.

Smela hopes this study will empower innovation in radiation treatment. "We would like to see this study guide and justify the use of UV polymers in radiation treatment at the clinical level," he said. "We would like to show that a 3D printer could pay for itself for a single purpose, knowing that success in a first use will spur people to consider what else can be achieved with 3D printing."

The group looks forward to seeing this technology grow beyond merely being an intriguing technology, to becoming a multi-purpose tool in search of the next problem to solve. "Given these printers' relatively low cost," said Smela, "we hope to show that they are easy enough to use, thus encouraging other treatment centers to try out the process themselves."

RadTech applauds BC Cancer's exploration and evaluation of FDM and MSLA 3D printing, where the features and benefits of UV can play a part in improving radiation treatment for cancer patients. ◆

Additive Manufacturing of Novel Surface-Eroding, Non-Swelling Anhydride Resins

Whytneigh R. Duffie; Kevin D. Barz; Tsvetanka S. Filipova; Katrina J. Donovan; Timothy M. Brenza; and Travis W. Walker, South Dakota School of Mines & Technology

Several groups have studied the application of water-soluble resin formulations for 3D printing of materials[4], with a common concern being the swelling of the polymer matrix during the dissolution process. In this work, a synthesis protocol was developed to produce unique surface eroding methacrylic-anhydride-based oligomers that later react to form crosslinked networks during the 3D-printing process. Chemical degradation leads to surface erosion of the crosslinked network after 3D printing, which provides ample opportunity for a wide array of applications where swelling of the polymer matrix has previously been a significant drawback in the functionality of water-susceptible 3D-printed parts.

Introduction

Development of 3D printable thermosets that chemically degrade in the presence of water in a controlled, predictive manner has been postulated as a novel strategy to engineer a number of advanced composites, including biomaterials (e.g., drug delivery, fracture fixation, tissue sealants), disposable single-use medical devices, and advanced transient sensor technologies.[1,2] Previous efforts to incorporate surface-eroding oligomers into resin formulations have been further limited by the commercial availability of surface-eroding constituents that are suitable for use in light-based 3D printing. Incorporation of the surface-eroding oligomers into novel resin formulations enables high-resolution, 3D-printable, acrylic-based resins that chemically degrade in the presence of water. Multiple avenues have been identified for development that allow for local photopolymerization of surface-eroding, anhydride-based formulations via 3D digital light processing (DLP). Hydrolysis of the anhydride bonds in the presence of water yields the diacid monomer and poly(methacrylic acid) via a surface-erosion mechanism.[1] Surface erosion occurs when chemical degradation and subsequent mass loss is limited to the outermost layer of the crosslinked product (Figure 1).

Bulk erosion occurs when chemical degradation and mass loss occur throughout the entire volume of the material (Figure 1). Surface erosion leads to higher retention of mechanical properties, less water uptake and swelling, and a more controlled degradation rate.[3]

Methods and materials

Photopolymer resin(s) were prepared with novel synthesized methacrylated-anhydride-based oligomers, reactive diluents and photoinitiators. The synthesized oligomers were characterized using FTIR for functional group confirmation (16 scans), ^{1}HNMR for structural confirmation and determination of molecular weight (300MHz, 1024 scans,

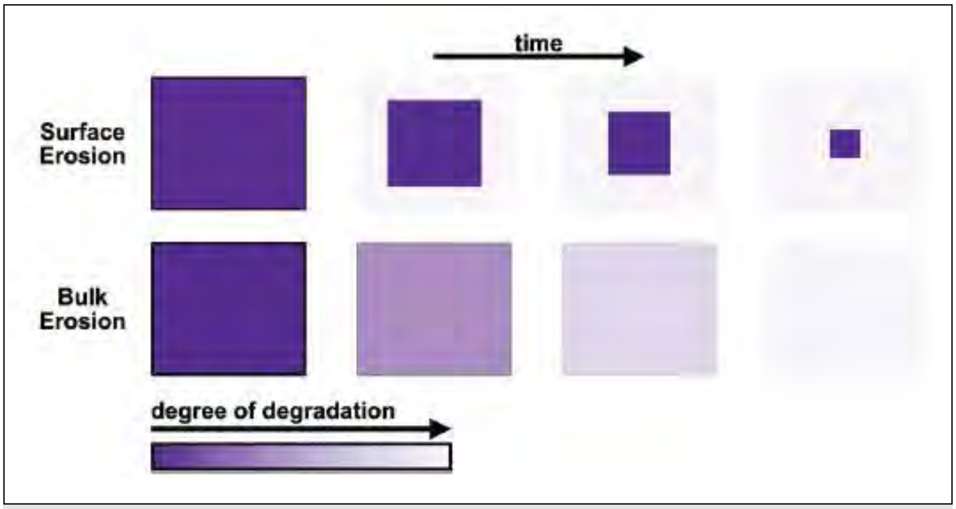

Figure 1. *Surface erosion (top) vs. bulk erosion (bottom) as related to the degree of degradation over time.*

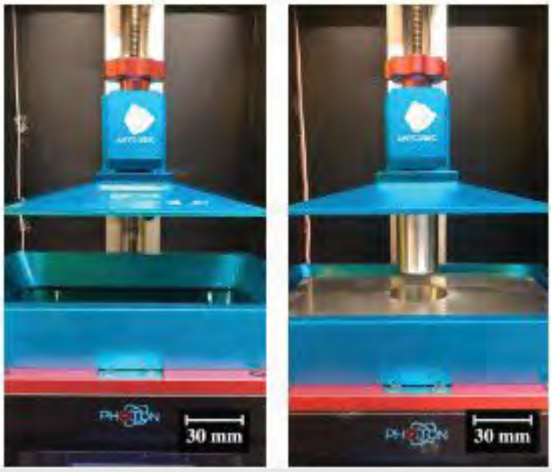

Figure 2. *Image of an Anycubic Photon LCD printer (left), and the modified Anycubic Photon LCD printer (right).*

$CDCl_3$), [13]CNMR for structural confirmation (300MHz, 8192 scans, $CDCl_3$) and Dept-135 NMR for confirmation of peak assignments (300MHz, 8192 scans, $CDCl_3$).

Crosslinked cylindrical disks (15 mm diameter and 1.85 mm height) were produced using an Anycubic Photon LCD printer (Figure 2, left). Printed samples were dried with a lint-free towel and cured in a B9Creations UV cure unit. Degradation studies of formulations containing a reactive diluent with equivalent weight percentages of three different oligomers were performed in triplicate using PBS buffer solution at a fixed agitation of 60 RPM and a temperature of 37° C. After the degraded samples were allowed to dry completely, the disks were massed to calculate the rate of degradation of the disks.

An Anycubic Photon printer was modified in this work to minimize the volume that is required for printing novel formulations to less than 1 mL (Figure 2, right). The modifications to the Anycubic Photon printer included a vat insert with a new area for loading the film, an additional part that was added to the original build plate to serve as the new build area, a 3D-printed sensor piece and an upper-limit switch with 3D-printed housing. The vat insert included a tapered cylindrical cavity at a reduced volume. The cavity that was created to hold the liquid resin included a slight tapering of the inner channel to create turbulence as well as a mixing effect given the dynamics of the printing process. The vat insert was secured with epoxy adhesive around the edges to avoid issues of the build plate lifting the insert

page 14 ▶

◀ page 13

during the printing process. Empty cavities were created on the bottom side of the vat insert to reduce the overall weight of the part. The FEP film insert with a smaller area was created with the same general idea as the original vat. The maximum volume that the new vat can hold is approximately 5 mL. Resin is added and removed from the vat using a disposable pipette.

A cylinder was attached to the original build plate using a set screw, and it can be removed at any time with the correct tool. The current cylinder has a diameter of approximately 25.25 mm, and a height of approximately 35.90 mm. The 3D-printed sensor piece has the same shape as the original metal piece that was provided with the Photon; however, the new piece is longer to accommodate for the length of the cylinder that was added to the build plate. The sensor length (55 mm) was chosen to allow for the (z = 0) position to be set slightly lower than the home position without the sensor piece ramming into the bottom of the printer.

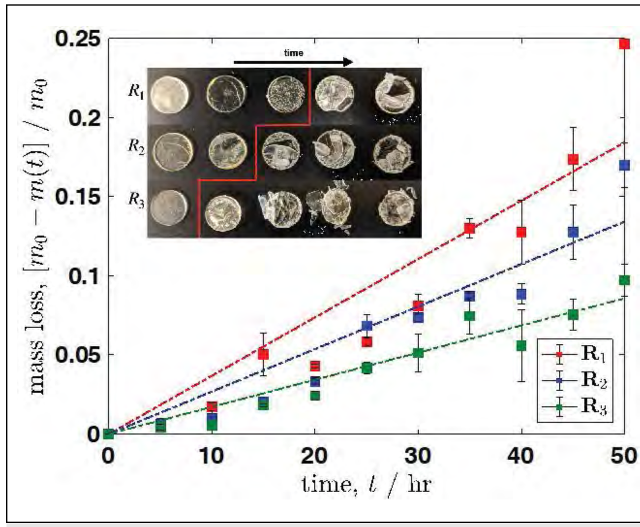

Figure 4. *Mass loss versus time of three resin formulations, where the size of R1 < R2 < R3.*

Results and discussion

To date, the novel synthesis route has enabled the invention of three previously unobtainable oligomers. Percent yields for all synthesized oligomers were over 70%, and oligomers that were chosen for further evaluation were limited to an 'n' value of two, where 'n' represents the average repeat of the anhydride oligomer. These lower-molecular-weight starting materials, available in both solid and liquid states at room temperature, allow for a wide array of applications, including 3D DLP (Figure 3).

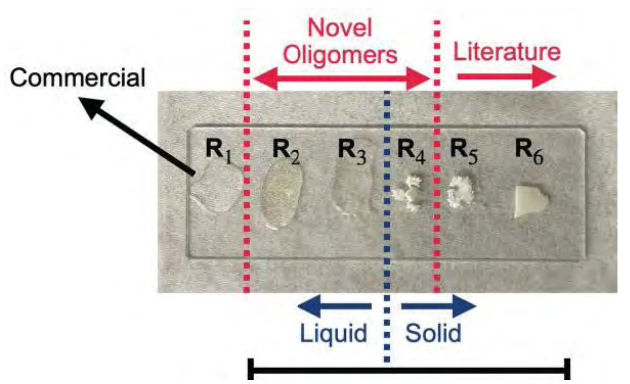

Figure 3. *Methacrylated-anhydride-based oligomers available commercially and as a result of the novel synthesis protocol.*

Oligomers existing in the solid state at room temperature are suitable materials for DLP upon dissolution in light-sensitive liquid crosslinking agents. Thus, solid-state oligomers that are loaded at specific weight fractions into the crosslinking agent undergo co-polymerization upon exposure to light with the addition of a photoinitiator. Several groups have demonstrated the successful copolymerization of solid-liquid mixtures by utilizing methacrylated sebacic anhydride, which is available via a single-stage synthesis route, and loading it into commercially available methacrylic anhydride.[2,3] While methacrylic anhydride is the only commercially available liquid bi-functional anhydride, literature suggests that when choosing homopolymerization of liquid methacrylic anhydride, limitations exist that are a result of limited radical diffusion. The long timeframes that are needed to crosslink the material at small volumes are unsuitable for most applications. While the addition of methacrylated sebacic anhydride was found to improve these exposure limitations, the increase in hydrophobicity of the backbone composition and decrease in crosslink density led to decreased erosion rates and decreased mechanical performance of the material.[3] Thus, constituents that are suitable for loading into methacrylic anhydride are both promising and worthy of investigation for applications that require increased erosion rates and improved mechanical performance. The additional availability of the products from the novel oligomeric synthesis allows the opportunity to further establish a trend of faster degradation rates and increased mechanical integrity as the length between methacrylated-anhydride functional groups decreases.

Disks that were 3D printed exhibited linear rates of degradation and shrinkage along only the surface, based on the observation of surface cracking. These results indicate that the crosslinked products were, in fact, surface degrading. The degradation studies suggested that, at a constant value of 'n,' a decrease in the length of the R-group in the formulation resulted in an increase in the rate of mass loss (Figure 4). In other words, the rate of degradation was found to increase as the hydrophilicity

page 16 ▶

◀ *page 14*

of the R-group increased, as found in the literature.[2,3] Controlled degradation rates provide the opportunity for commercialization in countless applications.

Next steps

By utilizing novel methacrylated-anhydride oligomers that are available solely through an innovative synthesis route, formulation development has shown great promise in providing expanded physical properties of degradable thermoset materials that are suitable for 3D printing. Given that the materials are surface eroding, it is hypothesized in future experiments that the modulus with respect to the thickness of a sample will exhibit very little change over most of the material lifetime. Toward the end of the material lifetime, the thickness of the sample approaches the same order of magnitude as the thickness of the erosion zone on the outer surface of the material. In this case, the modulus with respect to the thickness of the sample rapidly will decline.

Standard characterization techniques provide limited information about the degradation kinetics and mechanism of erosion. Therefore, a frugal microfluidic device has been developed to further access the kinetics and mechanism of surface-eroding polymer degradation (Figure 5). Liquid is introduced into the channel at a constant flow rate and temperature.

Polymer degradation can be assessed readily via image analysis by evaluating the erosion over time with respect to material formulation, polymerization conditions and sample environments. In-depth analysis of the degradation rates

"By utilizing novel methacrylated-anhydride oligomers that are available solely through an innovative synthesis route, formulation development has shown great promise in providing expanded physical properties of degradable thermoset materials that are suitable for 3D printing."

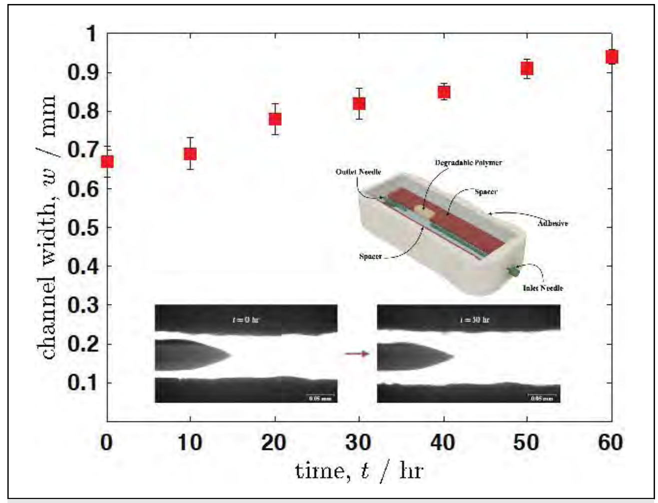

Figure 5. *Preliminary data of a surface eroding polymer material resulting from the frugal microfluidic device.*

and erosion mechanisms with the novel and adaptable flow apparatus can be coupled with previously explored methods of analysis to give insight into the complex mechanisms that take place during erosion. An increase in the number of crosslinks or in the hydrophobicity of the backbone should result in a decreased rate of degradation.

Acknowledgements

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Radiometry: Variations in Consistency Across 3D-Printer Models

By Paul Snowwhite, Katie Snowwhite and Hunter Peczynski, 7D Innovators, LLC

Editor's Introduction: The authors created an experiment to better understand why 3D printers created by various manufacturers perform differently even when using the same print settings or why a print fail occurs after multiple successful prints. Variables initially considered included material, cure source and printer calibration, but what emerged were concerns about consistent radiometry data across the width of the print platform. With better understanding of 3D printers, the ability to develop high-quality materials will be enhanced.

Background of 3D Printing

In the industry of additive manufacturing, 3D printers vary not only in the material used for printing – whether they print resin, filament or even cement – but also in how things get printed, with differences in positioning or curing sources. Three types of resin 3D printers are addressed in this article: stereolithography (SLA), digital light projector (DLP) and liquid crystal display (LCD). SLA, DLP and LCD printers (Image 1) use a process called vat polymerization to cure the resin into layers by using a resin tank and a certain type of light source.

SLA started to be commercialized in the mid-1980s and uses a laser beam to selectively cure layers of resin on the bottom of the resin tank that then adhere to a print platform. The laser works by using galvanometers, which are little mirrors under the resin tank, to guide the laser to the precise position that needs to be cured. In DLP 3D printing, a digital light projector is used to cure the layer of resin simultaneously, using the color black or no light at all to get precise layers. LCD printers use light emitting diodes (LEDs) that shine through LCD panels to cure the current layer of resin. LCD printers are the focus here.

LCD Printers with LED Light Sources

Liquid Crystal Display (LCD) printers have become a popular option for 3D printing as they provide a high-quality product while also being cost effective. LCD 3D printers take advantage of the light-modulating properties of liquid crystals when placed between crossed polarizers and clear electrodes. When a field is applied, the crystal molecules align and, depending on the orientation of the polarizers, light can pass or it is blocked. An array of UV LED panels is used as a cure source, only allowing UV to pass through where resin needs to be cured, reducing the need for galvanometers or mirrors and simplifying the printing process. LCD-based printers have a print quality that depends on the LCD density. The more pixels a display has, the better the quality of the print. LCD printers are scalable in a way DLP printers are not.

A common problem with LEDs is that they start to dim as they come to the end of their lifespan, which is unlike traditional light bulbs that burn out all at once. Each LED will age differently due to manufacturing (high-quality LEDs can have a long lifespan and fairly predictable degradation over time), and there are thousands of LEDs within a singular array. Small imperfections in the LED-chip semiconductor crystals can cause the LED to begin

page 18 ▶

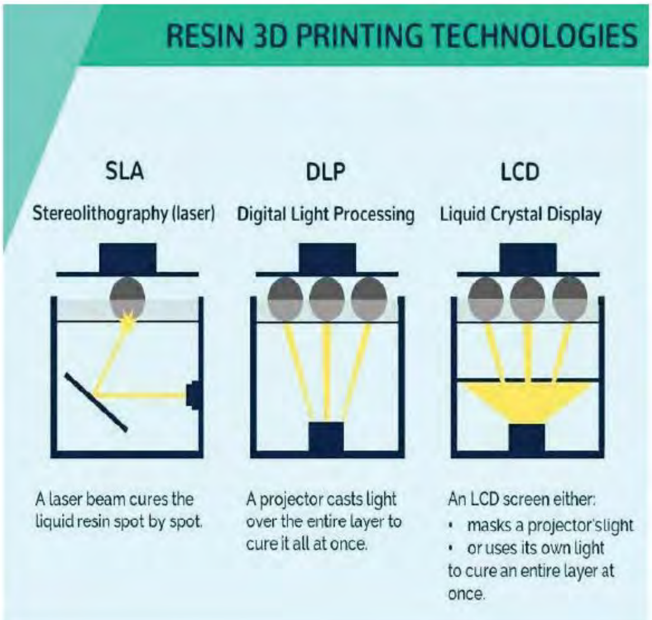


Image 1. *Schematic of SLA, DLP and LCD printers.*

◀ *page 17*

losing brightness. LEDs let out a small amount of heat when turned on and improper cooling can increase the rate of decay.

According to online reference ledlights.org, “The term degradation in connection with LED lighting describes the decrease in luminous flux over the course of a lifetime. The luminous flux slowly decreases due to material changes in the LED chip and clouding of the optics. Degradation is therefore an aging process in which an LED lamp loses its brightness over time and slowly becomes darker.”

Printer Consistency Issues and Observations

When 3D printing, there are many parameters that need to be met for a successful print to take place. Print failures can occur due to incorrect settings, support placement or equipment that needs calibration. Through their use of 3D printers, the authors have seen how important it is to include radiometry in the conversation about print issues.

When printing, failures have been observed on printers that had been consistently printing, were calibrated, had been cleaned, had correctly inputted settings and were using a known material. Sometimes these print fails were minor – such as a deformed support or an unlevel surface that was able to be sanded smooth – however, other times it would be a complete failure, with no model attached to the print platform or half the model missing. After a while, with these failures seemingly coming out of nowhere, it was time to begin investigating and learning more about the 3D printers being used.

It was observed that lifting and uneven models mostly were printed if they took up a majority of the print platform or were placed close to the edges. The starship in Image 2 is a good example of what it looks like when the print is lifting. This print took up most of the print platform horizontally, and the edges of the print lift more as it gets closer to the edges of the platform.

Image 2. *This starship shows lifting at the edges of the print.*

Testing of Radiometer: Consistent Spot on the Screen with Radiometer Turned on/off Between Samples		
Printer Number	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
#1	**4115.4**	7.7
#2	**4352.5**	2.8
#3	**4267.2**	1.3
#4	**4044.0**	6.8
#5	**4036.7**	0.6
#6	**4625.7**	12.7
#7	**4105.3**	9.0
#8	**4242.5**	15.9
#9	**4075.3**	3.6
#10	**4069.9**	2.2
#11	**4217.7**	2.5
#12	**3990.5**	0.1

Table 1. *Radiometer precision testing.*

Some 3D printer manufacturers offer replacement screens and instructional videos for their printers due to these inconsistency issues. Other 3D printing companies don’t offer a replacement option. While it’s understandable that companies don’t want untrained users trying to do their own printer repairs and (possibly) worsening the problem, acknowledging the problem and addressing it would be ideal.

Purpose of Experiment

One of the reasons this experiment was designed is because the authors started to notice that a print would fail on one printer, but it would print perfectly when tried on a different printer from the same manufacturer with the same print settings. In looking for the cause of this inconsistency, variables such as material, cure source and printer calibration were reviewed. In the past, there had been issues with prints peeling up at the edges, but those were attributed to not having a high-enough bottom exposure level. As more research into 3D printers was done, the authors learned that LED-curing arrays begin to dim, which causes them not to work at 100% power consistently or can cause inconsistency of cure over the whole screen.

Overall, the main reason for this experiment was to do more research to better understand the printers, find out why there are inconsistencies in print quality and explain the reasons

Cost Range	Manufacturer
under $1000	A, G
$1000-$5000	E, F
$5000-$10000	B, C
$10000+	D

Table 2. *Commercial cost range of tested printers.*

Printer Manufacturer A: 3 Spots on the Screen					
Printer Number	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
#1	4393	4199	4309	**4300**	97
#2	4521	4463	4488	**4491**	29
#3	4233	4165	4145	**4181**	46
#4	4180	4063	4536	**4259**	247
#5	4331	4101	4688	**4373**	296
#6	4823	4943	5290	**5019**	243
#7	4420	4417	4775	**4538**	206
#8	4375	4276	4564	**4405**	146
#9	4120	4490	4550	**4387**	233
#10	4293	4231	4621	**4382**	210
#11	4116	4277	4141	**4178**	87
#12	4385	4208	4313	**4302**	89
Avg. Per Side (uW/cm^2)	**4349**	**4319**	**4535**		
Std. Dev. Per Side	194	238	312		

Table 3. *Manufacturer A.*

for a print fail after multiple successful prints when there are no obvious reasons for failure. Material developers need to understand these things to develop materials at the highest quality possible for this industry.

Procedure and Equipment

A UV meter (radiometer) was used to measure the output of curing sources in the operation of 3D printers from a variety of manufacturers. The first step was making sure the radiometer was on and in the correct parameters (uW/cm^2) for this experiment. While wearing proper PPE, the display was turned on or the print was started, depending on the printer. Then, the probe was placed on the printer's screen to take a measurement of the UV output, running the probe for five seconds and recording the data. The process then was repeated and, depending on which measurements were being taken, the probe was kept in the same spot for more readings or moved to another spot on the printer's screen and data was recorded. The spot size of the probe was 1 cm in diameter. The average of three measurements was used to get the data reported for each particular printer.

To understand any variations that might occur, multiple spots across a printer's surface were measured – center and edges. The middle measurement for every printer was taken at the center of the screen, a fixed point. For left and right measurements, the size of the printer's screen had to be considered. To get these measurements, the probe was placed so that the outside edge of the probe aligned with the edge of the screen while being along the same horizontal line of the center measurement. This was done for both the left and right measurements.

The precision of the radiometer also was evaluated to ensure accuracy. Several experiments were done to understand the variation with respect to measurement. These were done by measuring printers in the same spot 180 times between three different printers with the radiometer cycled on and off without moving the probe (Table 1). As can be seen, the radiometer's measurements are extremely consistent with low variability. It also is important to note that all printers tested were within the specification per the manufacturer at the time testing was performed.

For the experiments performed, several printers were used. As the commercial cost range data show in Table 2, some printers tested were on the lower end and some on the higher end. The printers varied in how they allowed radiometry testing as well; some give measurement internally, but most don't, and some have an option to test the array's power without an actual measurement produced.

The point of this experiment was not to evaluate which printer or manufacturer had the best printer, but to create discussion about radiometry and 3D printing. Letters have been assigned to the printers within this experiment, and the printers have been categorized by cost in ranges to give a scope of understanding for readers.

Challenge in Measuring Energy Outputs

When beginning the testing for this experiment, it was observed that some manufacturers allow users to check the exposure levels and/or display an image that allows users to check the quality of the LED. With a few printers, it was necessary to start a print with a long exposure time and trick some safety features before being able to check the energy output. Three printer manufacturers have safety features or are designed in a way that do not allow users to check their energy output. With these challenges, the total energy output for individual printer manufacturers is not being compared across manufacturers.

Data and Analysis

The experiment began by looking at data from Manufacturer A. Testing was performed on a total of 12 printers from Manufacturer A, ranging in age from one month to 12 months

page 20 ▶

◀ page 19

Printer Manufacturer B: 3 Spots on the Screen					
Printer	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
New Printer	11739	12304	12151	12065	292
12 Months Old	9922	11462	11426	10936	879
Avg. Per Side (uW/cm^2)	**10285**	**11630**	**11571**		
Std. Dev. Per Side	1384	377	338		

Table 4. *Manufacturer B.*

Printer Manufacturer C: 3 Spots on the Screen					
Sample	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
1	11925	11842	11850	**11872**	46
2	11899	11834	11838	**11857**	37
3	11889	11821	11835	**11848**	36
Avg. Per Side (uW/cm^2)	**11904**	**11832**	**11841**		
Std. Dev. Per Side	19	10	8		

Table 5. *Manufacturer C.*

and in use from ~10 hours to ~200 hours. When looking at Manufacturer A and the data reported for these printers, it is important to note that the assigned printer number is not significant to the age of a particular printer or amount of time used. The assigned numbers are purely for distinction between printers of this manufacturer within the lab.

Manufacturer A's printers show a range of variation across the print platform (Table 3). A correlation study for exposure vs. print issues has not been done, and faster chemistries will be more robust with respect to this error. However, based on some calculations for this system, less than 2% is expected to be great, less than 5% to be acceptable, 5 to 10% could possibly start causing issues and >10% probably results in print issues. These percentages may vary due to differences in formulas and printers; however, these calculations could be applied to other manufacturers. For these 12 printers, six or seven of them could have significant print issues across the surface. The variation also is very high from printer to printer, so if it could be very challenging to reproduce a method over these printers.

Looking at the data produced by Manufacturer B's printers (Table 4), it can be seen that an older model had more issues with consistent prints as it aged. Due to this, a new one was purchased. The older printer meets the manufacturer's specifications (although the specifications do not take into account the changing output of the LEDs as aging occurs), and the new printer is fairly consistent with prints. The data clearly show the new printer has significantly less variation than the old printer (by greater than a factor of three) across the print surface and an even higher difference in irradiance.

Printer Manufacturer D: 3 Spots on the Screen					
Sample	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
1	2856	2425	2920	**2734**	269
2	2808	2415	2901	**2708**	258
3	2852	2424	2890	**2722**	259
Avg. Per Side (uW/cm^2)	**2839**	**2422**	**2904**		
Std. Dev. Per Side	27	5	15		

Table 6. *Manufacturer D.*

Manufacturer C is the only machine that does calibration of its own LED sources internally. However, the data displayed in Table 5 was a measurement taken with the authors' radiometer as per the method listed above to maintain consistency with the rest of the collected data. The printer is impressive with respect to the precision of irradiance, as shown through the data in the table.

As great example of "you don't always get what you pay for," Manufacturer D produces a very expensive printer, and its consistency across the build platform is poor (Table 6).

One of the largest printing surfaces comes from Manufacturer E. This printer was less than a month old with minimal hours printed on it at the time of testing. The data (Table 7) illustrates the difficulty with making a large print, and the variation will make it challenging to use the whole surface, especially as the printer ages. Although the variation was within an acceptable range at the time of measurement (less than one month old and fewer than five hours of use), will the variation worse as the machine ages?

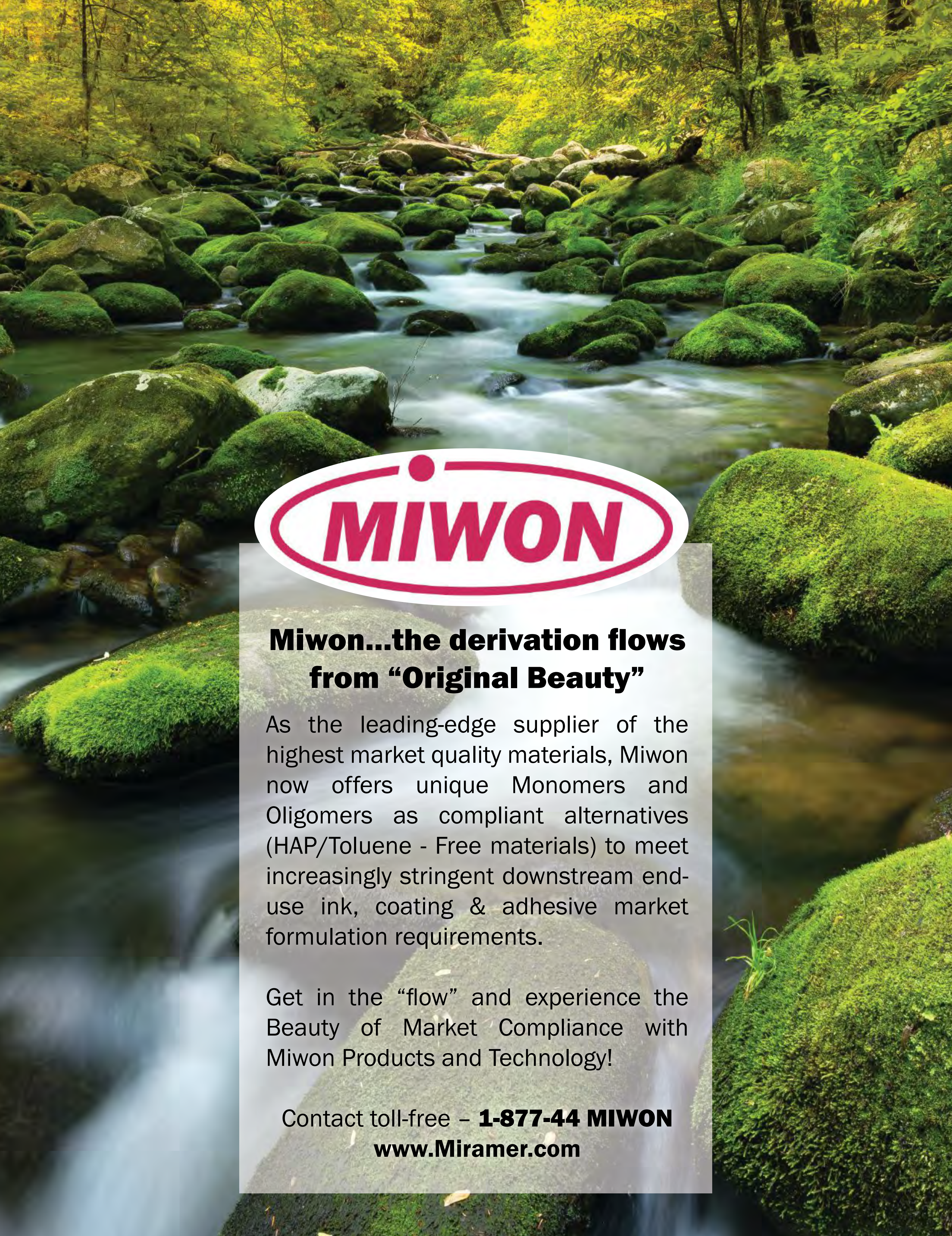
Manufacturer F's printer is relatively new, and it is ok out of the box with respect to irradiance over the entire surface of the printer (Table 8). Similar to E, concern is present that variation will worsen as the equipment ages. And finally, Manufacturer G – one of the older printers in the lab at over 15 months of age with pre-2020 technology – provides data measurements (Table 9) showing what was expected with respect to the variability due to its age.

Conclusion

In conclusion, when selecting a 3D printer, it is important to understand the machine's light source output quality and how the source might age in the future. This study has shown that the most expensive 3D printers don't guarantee a curing source that is the best option – according to the data, money doesn't necessarily buy quality when it comes to prints.

Having consistent radiometry across the entirety of the screen is important, but also difficult to achieve due to

page 22 ▶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◀ page 40

Printer Manufacturer E: 3 Spots on the Screen					
Sample	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
1	3992.5	3790.5	3779.7	**3854.2**	119.9
2	4000.4	3780.3	3784	**3854.9**	126.0
3	3996.4	3719.5	3789.2	**3835.0**	144.0
Avg. Per Side (uW/cm^2)	**3996**	**3763**	**3784**		
Std. Dev. Per Side	4	38	5		

Table 7. *Manufacturer E.*

Printer Manufacturer F: 3 Spots on the Screen					
Sample	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
1	3459	3233.1	3464.6	**3385.6**	132.1
2	3435	3219.8	3416.8	**3357.2**	119.3
3	3432.4	3163.4	3441.1	**3345.6**	157.9
Avg. Per Side (uW/cm^2)	**3442**	**3205**	**3441**		
Std. Dev. Per Side	15	37	24		

Table 8. *Manufacturer F.*

Printer Manufacturer G: 3 Spots on the Screen					
Sample	Left Spot Measurement (uW/cm^2)	Middle Spot Measurement (uW/cm^2)	Right Spot Measurement (uW/cm^2)	Avg. Per Printer (uW/cm^2)	Std. Dev. Per Printer
1	1107.8	1222.2	1022.3	**1117.4**	100.3
2	1108	1210.4	1011	**1109.8**	99.7
3	1101.5	1203.5	1006.2	**1103.7**	98.7
Avg. Per Side (uW/cm^2)	**1106**	**1212**	**1013**		
Std. Dev. Per Side	4	9	8		

Table 9. *Manufacturer G.*

structural differences or age of curing sources. As seen in the data on printers from Manufacturer A, seven of them have significant differences between the middle and edges on the screens.

Understanding the radiometry also can make a difference in print quality and success. The worst-case scenario is that failed prints occur with a failing curing source, but a degraded LED source also might be causing printed models to have different conversion levels if printed on different parts of the print platform – or even a singular model to have different conversion on different areas of its surface if it spans across a print platform. Uneven curing across the entire print also can cause added stress and embrittlement when undergoing post-cure.

After performing this study, some recommendations and guidelines have resulted for people looking to purchase a new 3D printer or create a method for a material. When setting up a method that is consistent for a material on a 3D printer, it's important to take into account the lowest cure settings that work for the material (not the average or highest) as not all print platforms give good results across the surface.

Manufacturer C, which had the best consistency data, was the only printer tested that had internal radiometry metrics. Therefore, it would follow that printer manufacturers whose machines have an internal option to check the radiometry would be recommended. However, simply having a printer that includes a display, internal test or calibration that a user could use to perform testing themselves also is a good option.

This is the authors' first in-depth look into the radiometry of the 3D printers to which they have access, and the entire study has been found to be very interesting. Throughout the experiment, questions have arisen that could be the focus of future investigation. These include the following:

- If a printer is producing good prints but there is variation across the surface of the printer, how does post-cure affect the material's properties?
- Do material physical properties change with the 10% difference in light, or does post cure bring all printed models to the same state?
- The authors' lab has done work on the effects of over-curing and under-curing materials during development, with negative results experienced when a material becomes over-cured and has embrittlement. Could this be happening to parts if a post process is optimized for prints that have been exposed to less energy from a particular printer?
- What is the correlation between the LED source's age and how many hours it has been used to print? ◆

Resources

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