

# 3D Printed Microstructures Erasable by Darkness

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To advance the applications of direct laser writing (DLW), adaptability of the printed structure is critical, prompting a shift toward printing structures that are comprised of different materials, and/or can be partially or fully erased on demand. However, most structures that contain these features are often printed by complex processes or require harsh developing techniques. Herein, a unique photoresist for DLW is introduced that is capable of printing 3D microstructures that can be erased by exposure to darkness. Specifically, microstructures based on light-stabilized dynamic materials are fabricated that remain stable when continuously irradiated with green light, but degrade once the light source is switched off. The degradation and light stabilization properties of the printed materials are analyzed in-depth by time-lapse scanning electron microscopy. It is demonstrated that these resists can be used to impart responsive behavior onto the printed structure, and –critically– as a temporary locking mechanism to control the release of moving structural features.

techniques have been developed, however, only certain techniques such as direct laser writing (DLW), also known as two-photon lithography, can truly fabricate 3D structures on the microscopic scale.<sup>[7]</sup> In order to achieve submicron resolution, DLW exploits the optical phenomenon of two photon absorption, resulting in a tightly focused voxel in a photoresist thus producing arbitrary 3D structures with up to sub-100 nm resolution.<sup>[8]</sup> Owing to its high precision and versatility, DLW has been used to fabricate structures that find applications in cell biology,<sup>[9]</sup> metamaterials,<sup>[10]</sup> microfluidics,<sup>[11,12]</sup> and photonics.<sup>[13,14]</sup>

Although DLW yields impressive results thus far, the printing technique typically relies on using the same classical photoresists, thereby resulting in permanent and “lifeless” objects. Hence, to further expand the applications of DLW, enabling responsive behavior to the printed structure is deemed critical.<sup>[15–17]</sup> One aspect of responsive behavior is degradability, more specifically, the ability to erase 3D structures post-printing and on-demand. Post DLW-degradation can, for example, be used for the repair of microelectronic contacts after prolonged periods of use, for the removal of support materials after fabricating complex 3D architectures, and for degrading cellular scaffolds to trigger cell release and/or to monitor how cells respond to the changing environment.<sup>[18,19]</sup> To achieve these aims, one must design advanced photoresists

## 1. Introduction

Since the pioneering work of Hull in 1986,<sup>[1]</sup> 3D printing technologies have attracted enormous attention. The ability to convert digital designs into tangible 3D objects at the push of a button has revolutionized the way objects are designed and manufactured.<sup>[2,3]</sup> Currently, 3D printing has been extensively applied in a range of fields from architecture to medicine and has enabled fascinating meta-material designs bordering on science-fiction, including invisibility cloaks.<sup>[4–6]</sup> Depending on the dimensions of the envisaged object, different 3D printing

techniques have been developed, however, only certain techniques such as direct laser writing (DLW), also known as two-photon lithography, can truly fabricate 3D structures on the microscopic scale.<sup>[7]</sup> In order to achieve submicron resolution, DLW exploits the optical phenomenon of two photon absorption, resulting in a tightly focused voxel in a photoresist thus producing arbitrary 3D structures with up to sub-100 nm resolution.<sup>[8]</sup> Owing to its high precision and versatility, DLW has been used to fabricate structures that find applications in cell biology,<sup>[9]</sup> metamaterials,<sup>[10]</sup> microfluidics,<sup>[11,12]</sup> and photonics.<sup>[13,14]</sup>

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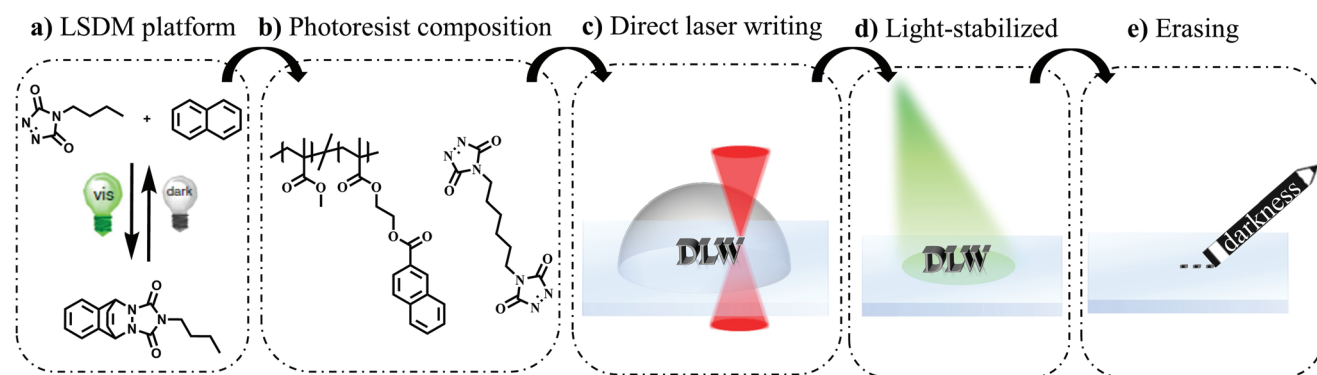
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**Figure 1.** General overview of the presented study. a) The LSDM chemistry platform, which relies on the visible-light-driven dynamic cycloaddition reaction of TAD and naphthalene. b) The LSDM platform incorporated into a photoresist for DLW. c) 3D direct laser writing of microstructures, as well as d,e) their light-stabilized properties and subsequent erasing when exposed to darkness, respectively.

that possess labile linkages, which can be degraded using external triggers. Whereas the introduction of labile chemical linkages is already exploited in areas such as organic materials synthesis, material reprocessing, and drug-delivery systems, their use in photoresists for DLW is still in its infancy and only a few examples have been reported.<sup>[20]</sup> For instance, direct-laser-written structures printed from labile photoresists can be degraded by temperature,<sup>[21]</sup> pH,<sup>[22]</sup> enzymes,<sup>[23]</sup> and light.<sup>[24]</sup>

Over the last decade, our group has been in the pursuit of developing dynamic systems, whereby covalent bond formation, as well as dissociation, can be triggered by noninvasive stimuli. In this context, we previously introduced an unprecedented soft matter materials system termed “light-stabilized dynamic materials” (LSDM) (Figure 1a).<sup>[25]</sup> The chemistry applied, herein, relies on the visible-light-driven cycloaddition of triazolinedione (TAD) to naphthalene, resulting in a cross-linked network. These out-of-equilibrium materials remain cross-linked while irradiated, however, spontaneously collapse through covalent dissociation once subjected to darkness. On the molecular level, the TAD/naphthalene cycloadduct cross-links constantly form and reform, resulting in a photo-stationary state concentration that is shifted far to the adduct side during irradiation. While the LSDM platform has since been applied to the folding of single polymer chains,<sup>[26]</sup> and to the synthesis of microparticles,<sup>[27]</sup> its unique properties have not previously been applied in the critical realm of additive manufacturing.

Herein, we pioneer such a novel photoresist for DLW that is capable of fabricating microstructures that cannot only be erased by darkness but also be stabilized under constant exposure to a visible light photonic field (Figure 1a–e). In doing so, we critically expand the applications of DLW, from which a new class of responsive materials can be fabricated.

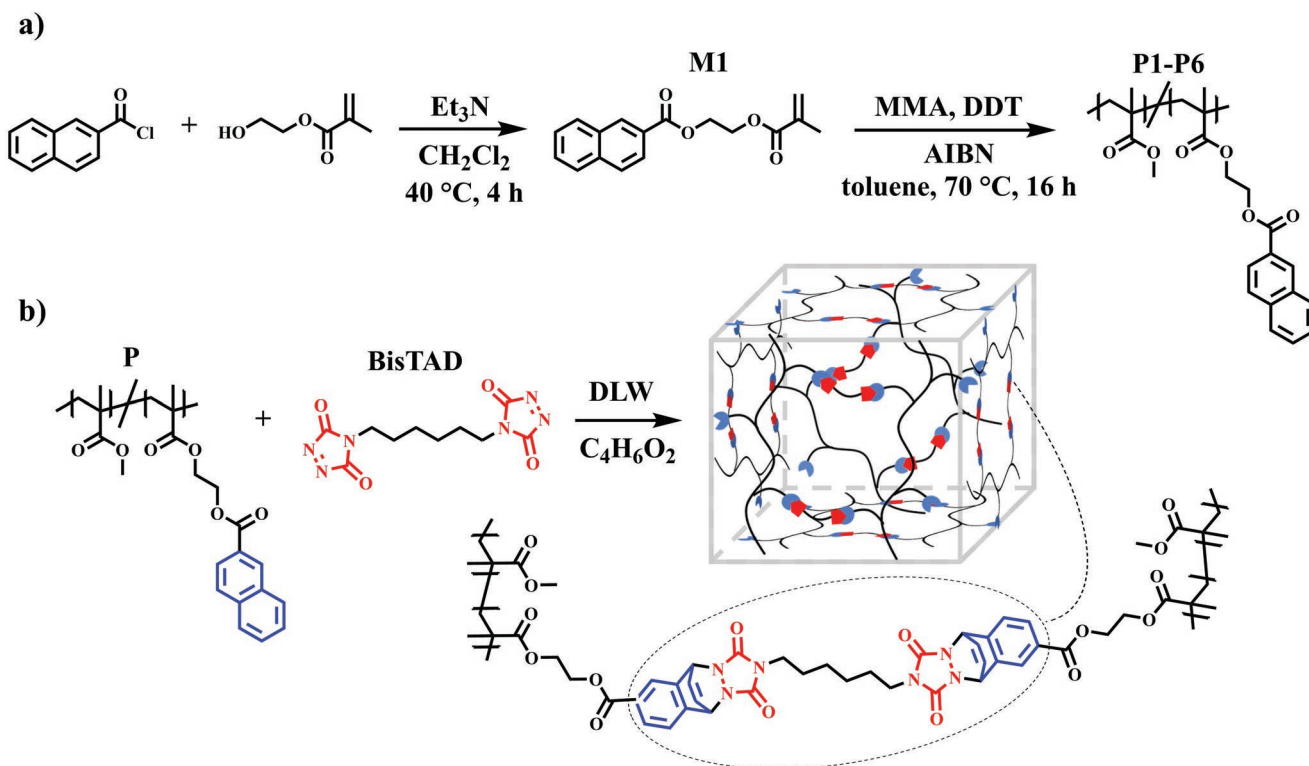
## 2. Results and Discussion

The LSDM-based photoresist design relied on making a judicious choice of the naphthalene co-reactant structure. Indeed, the positioning and the nature of the substituents of the naphthalene core – needed for their incorporation into a polymer that can be cross-linked with a bisfunctional TAD (Figure 1b) – can critically influence the formation and cycloreversion of the corresponding cross-links.<sup>[25,27]</sup> 2-Carboxy-naphthalene

derivatives were identified as suitable scaffolds as their irradiation in the presence of TAD resulted in >90% of clean reversion within 60 h when the cycloadducts are kept in the dark. Thus, the naphthalene monomer (M1) was synthesized in order to incorporate naphthalene units as functional pendent groups into a polymer backbone (Scheme 1a).<sup>[25,27]</sup> To investigate how the number of naphthalene units influences the fabrication of microstructures via DLW and their subsequent degradation, feed ratios of 15 and 25 mol% M1 were copolymerized with methyl methacrylate to afford polymers P1–P3 and P4–P6, respectively. To this end, free radical polymerization using 2,2'-azobis(2-methylpropionitrile) (AIBN) as a thermal initiator was used as the polymerization method owing to its low cost and simple synthetic procedure. In addition, by changing the amount of chain transfer agent and initiator, three molecular weights ( $M_n$ ) between 1500 and 10000 g mol<sup>-1</sup> for each M1 composition were targeted in order to investigate the influence of molecular weight on microstructure fabrication and degradation. The molecular weight and M1 content of the resulting polymers were determined by size exclusion chromatography and <sup>1</sup>H NMR spectroscopy, respectively (refer to Sections S3.1 and S3.2, Supporting Information).

In addition to the synthesis of P1–P6, 1,6-hexamethylene bistriazolinedione (BisTAD) was selected to afford the desired cross-linked networks (Scheme 1b; refer to Section S3.3, Supporting Information), which has been recently shown to be photoreactive through multiphoton absorption at  $\lambda > 700$  nm.<sup>[28]</sup> Subsequently, we exploited the cross-linking system consisting of a naphthalene-containing polymer (P) and BisTAD for the first time for the fabrication of 3D DLW microstructures (Scheme 1b). The LSDM-based photoresists ( $c = 1$  g mL<sup>-1</sup>) were prepared by dissolving P and BisTAD (0.1 and 0.3 molar equivalent of BisTAD relative to mol.% naphthalene in P) in propylene carbonate (Table S3, Supporting Information). Propylene carbonate was identified as the suitable printing matrix to prevent solvent evaporation during prolonged printing times and has good compatibility, i.e., has not been observed to undergo competing reactions with BisTAD.<sup>[25]</sup>

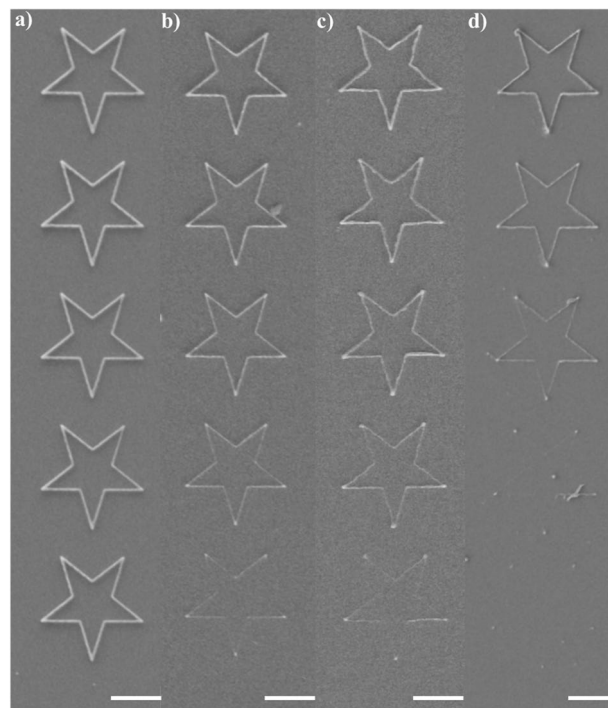
To assess the performance of the LSDM-based resists, an array of 3D micro-stars were printed onto a glass substrate with laser power and scan speeds between 5 and 8 mW and 10 and 30  $\mu\text{m s}^{-1}$ , respectively. Whereas no printing was observed when 0.1 eq of BisTAD was used, writing was possible when the



**Scheme 1.** a) Synthesis of M1 and subsequent synthesis of P1–P6 via free-radical polymerization with methyl methacrylate (MMA), *n*-dodecanethiol (DDT) as chain transfer agent, and azobisisobutyronitrile (AIBN) as the thermal initiator. b) The fabrication of 3D DLW microstructures created from the LSDM-based resist consisting of P and BisTAD in propylene carbonate.

amount of BisTAD was increased to 0.3 eq. Furthermore, no printing was observed when blank solutions of either BisTAD or P1–P6 were subjected to the above printing conditions. After development in propylene glycol methyl ether acetate for 10 min and acetone for 5 min, the 3D micro-stars were imaged by optical microscopy and subsequently immersed in tetrahydrofuran (THF) for 4 days in the dark at ambient temperature (Figure S10, Supporting Information). Important to note is that immersing the microstructures in a solvent such as THF induces swelling and hence facilitates mobility and therefore degradation of the material. To enable the printing of well-resolved structures that can be readily erased post-printing, structures created from the photoresist consisting of P1 with a laser power  $>5$  mW and a writing speed of  $10 \mu\text{m s}^{-1}$  proved to be suitable conditions for the fabrication of degradable structures (also refer to Tables S2 and S3, Supporting Information). To test the lateral resolution of the P1-based photoresist, line arrays with different spacing and thickness were fabricated by DLW (Figure S11, Supporting Information). Line-spacing down to 300 nm was achieved with a laser power of 8 mW and a writing speed of  $10 \mu\text{m s}^{-1}$  (Figure S11a–c, Supporting Information). However, when the laser power was increased to 9 mW, the deposited energy is too high and micro-explosions occur, disabling uniform micro-structure formation (Figure S11d, Supporting Information).<sup>[29]</sup>

In order to investigate the influence of laser power on micro-structure degradation, a laser power between 6 and 8 mW and a writing speed of  $10 \mu\text{m s}^{-1}$  was used to generate an array of micro-stars, which were developed and subsequently immersed in THF in the dark at ambient temperature. Here, the printing quality and subsequent degradation over 7 days were observed in detail

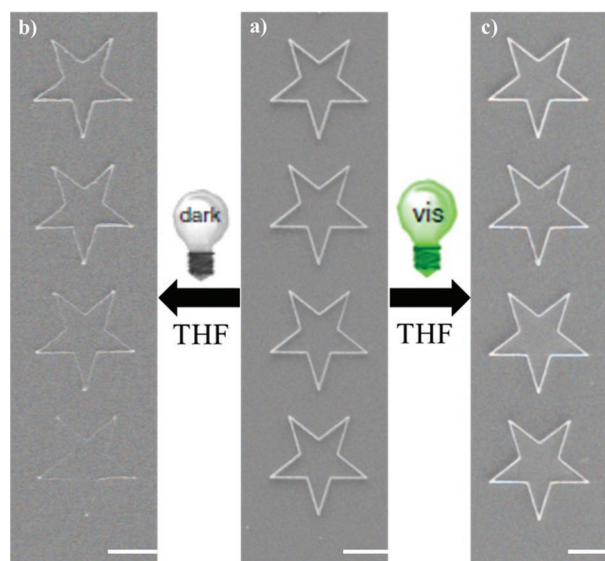


**Figure 2.** SEM images of direct laser written 3D micro-stars, which were created from the P1-based photoresist with a laser power between 6 (bottom star) and 8 mW (top star), in 0.5 mW increments, and a writing speed of  $10 \mu\text{m s}^{-1}$ . a) After development, b–d) immersed in THF in the dark for 2, 5, and 7 days, respectively (scale bars, 10  $\mu\text{m}$ ).

via time-lapse scanning electron microscopy (SEM) imaging (Figure 2). As depicted in Figure 2a, the printing resolution of each micro-star is significantly improved with higher laser power, since higher laser power results in a more densely cross-linked material.<sup>[30]</sup> Consequently, these stars degrade much slower compared to those printed with a lower laser power (Figure 2b–d). Thus, the micro-star printed with a laser power of 8 mW shows only minor degradation after 7 days, whereas complete degradation is observed when the laser power is reduced to 6 mW (Figure 2d). It is important to note that although this study focuses on writing parameters that lead to high printing quality and rapid degradation (e.g., laser power 6.5 mW), altering the laser power during the writing process would also be an elegant approach toward incorporating multiple properties into a micro-structure from a single photoresist.<sup>[30]</sup>

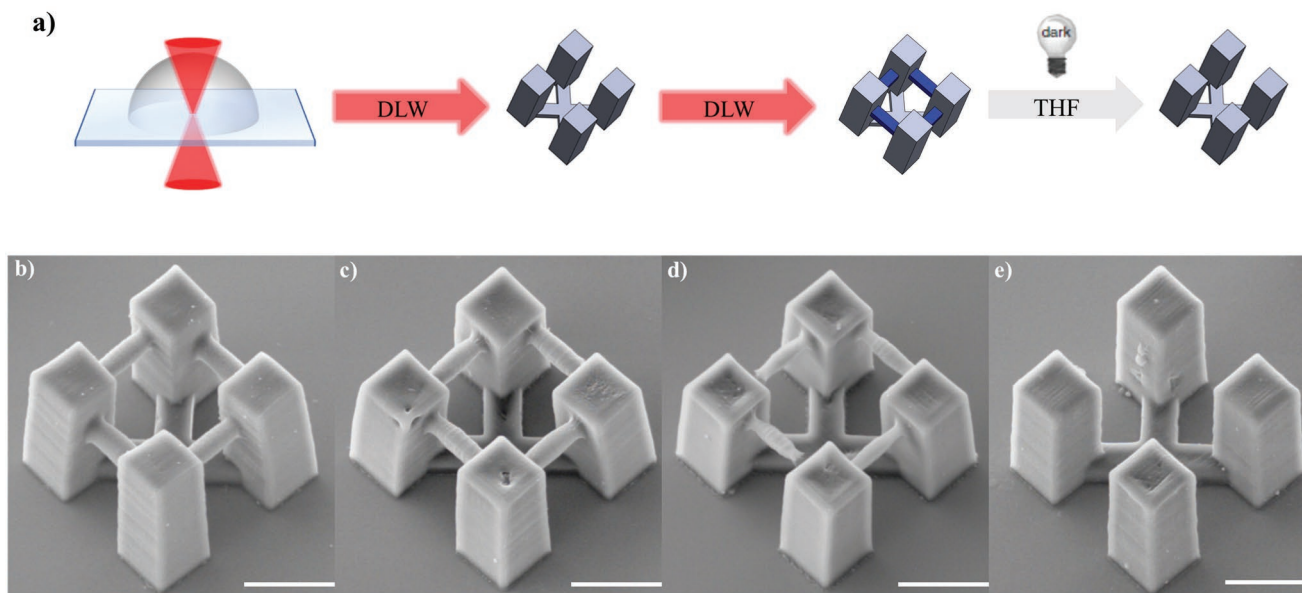
To investigate the light-stabilized properties of the direct laser written material, micro-stars printed with a laser power between 6 and 7.5 mW and a writing speed of  $10 \mu\text{m s}^{-1}$  were immersed in THF and continuously irradiated with a green LED for 5 days (10 W,  $\lambda_{\text{max}} = 525 \text{ nm}$ , refer to Figures S12 and S13, Supporting Information). In contrast to the micro-stars after development (Figure 3a), the stars kept in the dark have significantly degraded after 5 days (Figure 3b). Moreover, no degradation is observed when the micro-stars are continuously irradiated with green light (Figure 3c). In essence, the light stabilizes the material and prevents these micro-stars from degrading.

Having established the optimal photoresist composition and DLW parameters, the developed resist technology was applied to embed spatially resolved responsive features into 3D materials. Here, a classical resist is combined with the developed LSDM-based resist into a multi-material scaffold-type structure, effectively endowing it with responsive behavior. Subsequently, the scaffold was sequentially assembled in two steps

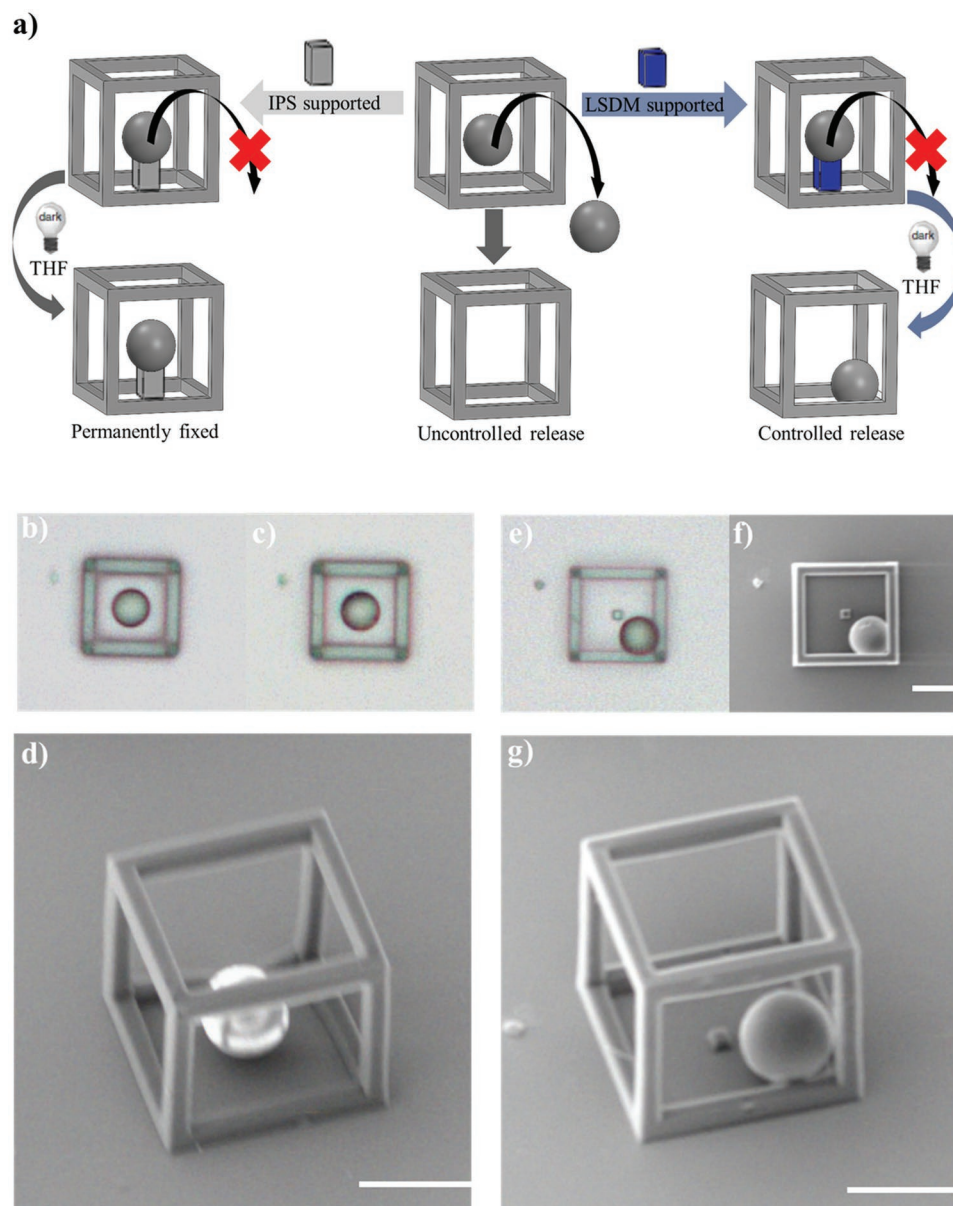


**Figure 3.** SEM images of direct laser written 3D micro-stars printed with a laser power between 6 (bottom) and 7.5 mW (top), in 0.5 mW increments and a writing speed of  $10 \mu\text{m s}^{-1}$ . a) After development, b) immersed in THF in the dark for 5 days, and c) immersed in THF and continuously irradiated with a green LED (10 W,  $\lambda_{\text{max}} = 525 \text{ nm}$ ) for 5 days (scale bars,  $10 \mu\text{m}$ ).

(Figure 4a). Initially, a commercial, acrylate-based, and highly cross-linked resist (IP-S, Nanoscribe) was used to construct the base and four pillars with a laser power of 12 mW and a writing speed of  $3000 \mu\text{m s}^{-1}$ . Second, after immersing in isopropanol for 10 min to wash away excess resist, the connecting bridges were printed from the LSDM-based resist. After development, the scaffold was placed in THF and shielded from the light at



**Figure 4.** a) Two-step writing strategy utilized for the fabrication of responsive multi-material scaffolds. First, DLW is used to print the base and four pillars from a commercial resist (IPS, Nanoscribe). In a second DLW step, the connecting bridges (blue) are constructed from the LSDM-based resist. The multi-material scaffold is then immersed in THF in the dark for several days at ambient temperature to erase the bridges. b–e) SEM images of multi-material scaffolds after development, and immersed in THF in the dark for 3, 7, and 9 days, respectively (scale bars,  $10 \mu\text{m}$ ).



**Figure 5.** a) Overview of the ball-in-a-box model utilized to demonstrate unprecedented moving features. When the ball-in-a-box is printed without support material underneath the ball, it is uncontrollably lost during development (center). Although a support block constructed from the commercial resist (IPS) prevents the ball from escaping during development, it is permanently attached to the support block (left). In contrast, a support block constructed from the LSDM-based resist provides a temporary locking mechanism, whereby degradation of the support block releases the ball in a controlled fashion (right). b,c) Optical microscopic images after development and immersed in THF in the dark for 12 h, respectively. d) SEM image after development. e–g) Optical microscopic and SEM images after immersion in THF in the dark for 36 h (scale bars, 25  $\mu\text{m}$ ).

ambient temperature. As depicted in Figure 4b–e, SEM analysis was employed to monitor the degradation of the bridges over several days. To allow unsupported printing from pillar-to-pillar, a solid tightly-hatched structure was required, leading to the bridges degrading slower than the previously written micro-stars. Nevertheless, the bridges significantly degraded after 7 days and started to detach from the support pillars (Figure 4b–d). Finally, the bridges were completely erased after 9 days in the dark (Figure 4e).

With the utility in 3D printing of darkness-triggered degradable segments into multi-material structures established, the

opposite fabrication strategy was explored, where the LSDM structure was printed first and static structures from the commercial resist were added subsequently. In such an approach, the on-demand degradation holds the potential for release mechanisms to enable unprecedented moving features. Specifically, a ball-in-a-box model was utilized to demonstrate the introduction of moving features (Figure 5a). It is important to note that the box was intentionally designed to allow the ball to readily escape either during the printing or the development process if release is uncontrolled. Initially, a ball-in-a-box was fabricated only from the commercial resist (IPS, Nanoscribe, laser power

12 mW, writing speed 3000  $\mu\text{m s}^{-1}$ ) without any supporting material underneath the ball (Figure 5a, center). While the ball can be printed inside the box unsupported, it is uncontrollably lost during development and thus a support block is required to temporarily lock the ball in place (refer to Figure S14, Supporting Information). Such support blocks are typically written by either commercial resists that have to be removed mechanically post-development or by customized resists that require additional development steps to chemically trigger an erasing mechanism.<sup>[22]</sup> By using our LSDM-based photoresist, however, self-destructive support features that bypass active erasing steps can now be implemented into DLW. Indeed, by using the two-step fabrication process described above, albeit, in reverse, the LSDM-based resist was initially used to construct the ball support block (Figure 5a, right). Subsequently, the ball-in-a-box was printed from the commercial resist ensuring that the ball was printed directly onto the support block. Finally, the completed structure was immersed in THF in the dark at ambient temperature. In contrast to the unsupported ball-in-a-box model, optical microscopic monitoring and SEM evidence that the ball remains in the center of the box for up to 12 h after the fabrication process, since it is still attached to the LSDM-based support block (Figure 5b–d). However, after 36 h when some dark-degradation of the support block has occurred, optical and SEM imaging verify that the ball is released and rolls into a different position within the box (Figure 5e–g). In contrast, when the support block is constructed from the commercial resist (Figure 5a, left), the ball remains attached to the support block indefinitely (also refer to Figure S15, Supporting Information).

### 3. Conclusions

We have pioneered a self-destructive LSDM-based photoresist for DLW applications, introducing the first photoresist that enables the fabrication of microstructures via DLW that remain stable when continuously irradiated with green light but degrade when exposed to darkness. Critically, we demonstrate that the LSDM-based resist can print microstructures exclusively consisting of LSDMs, as well as 3D multi-material structures when combined with a commercially available photoresist in a simple two-step fabrication process. Specifically, multi-material scaffolds where parts of the scaffold can be selectively erased by darkness, and support blocks that control the release of moving features have been printed via DLW. In addition, we demonstrated that the degradation properties of the printed microstructures can be tuned without altering the photoresist itself but by simply changing the writing parameters. Our photoresist constitutes a critical advancement in dynamic 3D printing applications where mild triggers are required to control post-printing properties, ranging from the degradation of cellular scaffolds to the selective removal of support structures and the introduction of movement in complex architectures that would otherwise not be printable.

### Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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### Conflict of Interest

The authors declare no conflict of interest.

### Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

### Keywords

direct laser writing, erasable structures, light-stabilized dynamic materials, microstructures, multi-materials, photoresists

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