Material-Independent 3D Patterning Via Two-Photon Lithography and Discontinuous Wetting

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The fabrication of complex 3D structures composed of micrometer-sized features made of different functional materials is an immensely important and yet highly challenging task. Here, a method is developed to fabricate multimaterial 3D structures with micrometer precision by combining macroscopic 3D printing (digital light processing), with two-photon lithography (2PL) and material-independent discontinuous dewetting. Specifically, 3D inherently superhydrophobic objects are first printed by DLP, followed by creating hydrophilic micropatterns on their surface using 2PL. By exploiting the effect of discontinuous wetting, the selective deposition of solutions of functional materials into microscopic hydrophilic regions on the surface of 3D structures, with high resolution and great design flexibility is demonstrated. Importantly, the method is material-independent and enables the micropatterning of a variety of functional materials dispersed in aqueous solutions including polydopamine, silica, or Ag nanoparticles. As an exemplary application, it is shown that conductive Ag electrodes can be created on the curved surface of 3D-printed mine, silica, or Ag nanoparticles. As an exemplary application, it is shown that conductive Ag electrodes can be created on the curved surface of 3D-printed mine, silica, or Ag nanoparticles. As an exemplary application, it is shown that conductive Ag electrodes can be created on the curved surface of 3D-printed mine, silica, or Ag nanoparticles. As an exemplary application, it is shown that conductive Ag electrodes can be created on the curved surface of 3D-printed mine, silica, or Ag nanoparticles. As an exemplary application, it is shown that conductive Ag electrodes can be created on the curved surface of 3D-printed mine, silica, or Ag nanoparticles. 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Discontinuous dewetting is a powerful method to pattern deposit functional materials on surfaces. In discontinuous dewetting, patterns with extreme wettability difference (e.g., hydrophilicity and superhydrophobicity) lead to the spontaneous break of a bigger droplet and formation of smaller droplets, confined to the hydrophilic regions of predefined geometries and driven by the dewetting of liquid on the superhydrophobic background.\(^{[14-16]}\) Due to its simplicity, scalability, and versatility, this strategy has been used for applications including optical waveguides,\(^{[17]}\) flexible electronics,\(^{[18]}\) and miniaturized biological screenings.\(^{[19]}\) Although discontinuous dewetting has been applied to 2D surfaces, its potential to pattern 3D objects remains to be explored. A recent study used a mask covering 3D aluminum structures\(^{[20]}\) to print inherently superhydrophobic 3D polymer structures by DLP. Ink composition: 30 wt% butyl acrylate, 20 wt% ethylene glycol dimethacrylate, 50 wt% 1-decanol.

To 3D print superhydrophobic objects via DLP, we have developed an ink consisting of a hydrophobic monomer (butyl acrylate), a crosslinker (ethylene glycol dimethacrylate), a porogen (1-decanol), and a photoinitiator Irgacure 819.\(^{[22]}\) The porogenic solvent leads to in situ phase separation during DLP printing, affording an inherently porous nanostructured material (Figure 2a). Such porous structures can not only render the surface of the 3D-printed object superhydrophobic, but also amplify the hydrophilicity of the hydrophobic patterns created by 2PL. This extreme contrast of wettability is critical to ensure the effect of discontinuous wetting and site-specific material patterning. As shown in Video S1 (Supporting Information), the DLP-printed truncated pyramid demonstrated excellent water repellency with very low water adhesion (with a pull-off force of about 3 \(\mu\)N as measured by scanning

![Figure 1. A schematic showing the method for creating multimaterials 3D micropatterned objects detailed in this work. a) Fabrication of a macroscopic superhydrophobic 3D polymer structure by DLP. Ink composition: 30 wt% butyl acrylate, 20 wt% ethylene glycol dimethacrylate, 50 wt% 1-decanol. b) Creation of hydrophilic micropatterns on the top and side surfaces of the DLP-printed objects by 2PL. Ink composition: hydroxyethyl acrylate, crosslinker (ethylene glycol dimethacrylate), a porogen (1-decanol), and a photoinitiator Irgacure 819. c,d) Exposure to and confinement of an aqueous solution of functional materials onto the hydrophilic micropatterns using the effect of discontinuous wetting. e) Formation of micropatterns of functional materials deposited in hydrophilic regions upon solvent evaporation.](image)
droplet adhesion microscopy\(^{(22)}\)). In a second step, hydrophilic patterns were created on the superhydrophobic objects by acrylate polymerization-mediated photografting, using 2PL. The micropatterning was performed by exposing the superhydrophobic structures to a hydrophilic monomer, 2-hydroxyethyl acrylate (HEA), in the presence of a photoinitiator, Irgacure 819 (2 wt%). Due to the incomplete conversion during DLP printing process, considerable amounts of methacrylate groups remain unreacted on the surface of the printed structures as proved by the ATR-FTIR spectroscopy (Figure S1, Supporting Information). Upon laser exposure, these unreacted (meth)acrylate groups can react with the growing chains of polyHEA to achieve covalent surface grafting, which changes the surface wettability and generates hydrophilic micropatterns. It is also possible that polyHEA physically adsorbs to the surface. To evaluate this, the DLP-printed porous structures were immersed into a polyHEA solution, followed by washing and drying steps (see the Experimental Section for details). However, since the produced surface remained superhydrophobic, we can conclude that the produced hydrophilic patterns are due to the covalent surface grafting of polyHEA and not because of its physical adsorption. The optimization of the patterning parameters (noticeably laser power and scanning speed) were carried out based on the evolution of the patterning quality and reliability with respect to the variation of these parameters. It was observed that scanning speed above 10 000 µm s\(^{-1}\) resulted in decreasing patterning quality and reliability. Laser power lower than 35 mW resulted in unreliable, incomplete or unsuccessful patterning, while laser power up to 50 mW lead to an increasingly significant deterioration of the surface morphology (Figure S2, Supporting Information). The optimized parameters (10 000 µm \(s^{-1}\) scanning speed, 35 mW laser power) were chosen to optimize time-efficiency of the process, the retention of the morphology of the surfaces through the patterning process, determined by SEM imaging (Figure 2a), as well as to optimize the definition and reliability of the micropatterning.

The photografting of HEA onto the hydrophobic structure by 2PL was evidenced by time-of-flight secondary ion mass spectrometry (ToF-SIMS). Due to several molecular similarities in the building blocks of the main polymer used for DLP and the poly(2-hydroxyethyl acrylate) used for functionalization, only a small number of fragments in the SIMS data showed the material contrast at this stage: C\(_2\)H\(_3\)O\(^{-}\), C\(_2\)OH\(^{-}\), C\(_3\)H\(_3\)O\(_2\)\(^{-}\), and phosphates. The former three fragments can be cleaved from the side chain in poly(2-hydroxyethyl acrylate) and are represented in green in Figure 2b, whereas the substrate polymer is imaged in red based on the observed fragments C\(_2\)H\(^{+}\), C\(_3\)H\(_5\)\(^{+}\), C\(_4\)H\(^{+}\), and C\(_2\)H\(_3\)O\(^{-}\). Phosphate signals are attributed to the used photoinitiator. Reference spectra obtained from unreacted Irgacure 819 showed a very strong PO\(^2\)\(^{-}\) signal (original data are provided in the repository Radar4KIT). Furthermore, in unreacted Irgacure TOF-SIMS analysis finds the molecular signal for C\(_{26}\)H\(_{27}\)PO\(_3\)\(^{-}\) and C\(_{26}\)H\(_{26}\)PO\(_3\)\(^{-}\) with matching

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**Figure 2.** Demonstration of the overall process using a fluorescent dye (RhB) as an exemplary material. a) SEM images and b) ToF-SIMS maps of the 2PL micropatterned truncated pyramid. Green: Sum of C\(_2\)H\(_3\)O\(^{-}\), C\(_2\)OH\(^{-}\), and C\(_3\)H\(_3\)O\(_2\)\(^{-}\); red: Sum of C\(_2\)H\(^{+}\), C\(_3\)H\(_5\)\(^{+}\), C\(_4\)H\(^{+}\), and C\(_2\)H\(_3\)O\(^{-}\). 128×128 pixel, 5-point averaging in the image. c) Snapshots of a video (Video S3, Supporting Information) showing the deposition and evaporation of a dyed water droplet (RhB) on the hydrophilic-superhydrophobic patterned truncated pyramid. d) Confocal fluorescence microscope images showing various fluorescence patterns formed on the hydrophilic-superhydrophobic patterned truncated pyramid via discontinuous wetting.
isotope patterns. This signal is not found in case of reacted photoinitiator since the initiator is degraded and incorporated into a long polymer chain, leaving behind the PO₂⁻ signal.

To demonstrate the working principle of our strategy and the formation of hydrophilic patterns on a superhydrophobic surface, we first used a water droplet containing a fluorescent dye, rhodamine B (Rhb). When the aqueous droplet contacted the patterned pyramid, only the micrometric region (200 μm diameter circle) functionalized with the hydrophilic poly(2-hydroxyethyl acrylate) was wetted, and a “daughter” droplet remained adhered with a shape conformal to the pattern geometry (Figure 2c; and Video S3, Supporting Information). Such a discontinuous wetting effect results from the combination of the superhydrophobicity of the 3D object, and the high hydrophilicity of the patterned circular region due to the presence of polar groups created by 2PL. The surface is probably superhydrophobic but since it is hard to measure the contact angles on the micrometer sized spots, we will refer to these regions as hydrophilic. Upon droplet evaporation, the RhB from the aqueous solution, remained deposited on the hydrophilic regions and could be readily visualized (Figure 2c). It is worth noticing that the surface structure of the patterned region experienced an obvious shrinkage due to the capillary forces generated during water evaporation (Figure S3, Supporting Information). Thanks to the high-resolution and versatility of 2PL, complex micropatterns (including a star, a spiral, a ring and letters) with very high definition could be created on top of the DLP-printed truncated pyramid and functionalized with RhB, the visualization of these patterns by confocal fluorescence microscopy is shown in Figure 2d.

The selective surface functionalization of 3D objects with micrometer precision promises unique applications in various fields including microelectronics, robotics, optics, microreactors, and bioengineering. Although techniques for microscale surface functionalization on 2D surfaces have been well-established, only a few are applicable to 3D structures, including electron-beam lithography (EBL), 2PL, and dippen nanolithography (DPN). These techniques, however, are developed for a particular type of material (e.g., EBL demands electron-sensitive resist, 2PL needs photoreactive functional species, DPN usually requires molecular inks), and therefore require significant developments for each new material or component. In contrast, as our method combines 2PL and discontinuous wetting for site-specific material deposition, it is almost material-independent as long as the material can be dissolved or dispersed in an aqueous solution. Other materials that can be formed in situ via a reaction inside the droplet can also be used. Therefore, our approach opens new possibilities to micropattern 3D objects with various materials and achieve previously inaccessible properties and functionalities. As a proof-of-principle test for the functionalization of surfaces non-parallel to the substrate, we first created hydrophilic patterns both on the top and side areas of a 3D-printed pyramid. The patterned structure was immersed in an aqueous RhB solution and then immediately taken out and visualized under a microscope. Figure 3 shows the dye patterns left on the top and side surfaces of 3D structures after droplet evaporation, indicating that only the 2PL patterned regions were wetted by the solution, leading to the selective dye deposition.

We next demonstrate the material-independence of our method by selective deposition of several functional materials, including dopamine for polydopamine-based surface functionalization, as well as silica and silver nanoparticles. Polydopamine is a substrate-independent surface coating material widely used to construct functional surfaces with versatile properties such as biocompatibility, antifouling, conductivity, or catalytic activity.  We created polydopamine micropatterns on the DLP-printed objects by simply immersing them into a dopamine Tris-buffer (pH 8.5) solution for 1 h. The formation of polydopamine patterns is visualized by optical microscopy and further confirmed by ToF-SIMS characterization (Figure 4a). Some discrete flakes were also found on the truncated pyramid. This might be attributed to the highly adhesive property of polydopamine, which adsorbs to the polymer globules of superhydrophobic substrates, leading to partial wetting during the submerging process. This can be potentially improved by using perfluorinated (instead of aliphatic) superhydrophobic substrates to reduce adsorption and prolong the lifetime of superhydrophobicity. Importantly, the selective deposition of polydopamine serves as an example to demonstrate the ability of our method to not only pattern materials soluble/dispersible in water, but also materials that can be in situ formed via reactions happening inside microdroplets. Such ability opens an additional possibility to pattern materials that are difficult to dissolve/disperse, for example, conductive polymers or polymer gels.

The precise patterning of micro/nanoparticles is important for both fundamental research and practical applications such as sensing, catalysis, advanced optics, or organic electronics. However, the selective deposition of particles at the microscale is challenging, especially on 3D surfaces. Here, we show that our patterning based on discontinuous dewetting can be applied not only to small molecule solutions, but also to nanoparticles dispersions. Figure 4b shows 2D patterns of silica nanoparticles formed via discontinuous dewetting on a truncated pyramid. Interestingly, the silica nanoparticles self-organize into well-ordered arrays during the droplet evaporation.

Creating conductive patterns on 3D objects is central to the fabrication of advanced structural electronics, and has previously been realized by direct printing of conductive inks. However, the resolution of most extrusion/jetting based printing techniques is still quite limited. Our method, on the other hand, allows for the patterning of conductive materials on 3D-printed objects at the micrometer scale. Thanks to the thermal stability of the DLP-printed structures, Ag nanoparticles were successfully patterned and further annealed (150 °C, 30 min) on the surface of 3D-printed pyramids, as proved by both optical and SEM-EDX microscopy (Figure 4c; and Figure S4, Supporting Information). Electrical measurements showed a conductivity of the Ag patterned regions (line width 50 μm, length 300 μm) with a corresponding resistance of about 350 Ω, while the 3D porous substrate (without patterning) was an insulating material (R > GΩ) (Figure 4d). These results demonstrate the ability of this method to fabricate structural microelectronic devices combining the functionality of electronic circuits with 3D design flexibility, which are useful in advanced antennas, sensors or transistors.
3. Conclusion

In this work, we proposed a methodology enabling material-independent micropatterning of 3D surfaces by combining 2PL and discontinuous wetting. This method allows for precise micropatterning of various functional materials (including dyes, polydopamine, nanoparticles) both on the upper surface and side areas of 3D objects, with very high spatial resolution and geometrical freedom. As a proof-of-concept application, we fabricate conductive Ag microelectrodes on 3D surface. It is foreseeable that our approach is applicable to many more functional materials that are water dispersible or can be in situ formed via reaction inside the aqueous droplet including, inter alia, proteins, quantum dots, conductive polymers, and polymer gels. We believe that the micrometer precision, design flexibility, ability to pattern 3D surfaces and material-independence of this strategy will pave the way to a new generation of multimaterial 3D structures with unprecedented properties and functionalities, opening exciting opportunities in numerous applications from advanced microelectronics, and sensors to microreactors and bioengineering.

4. Experimental Section

Materials and Chemicals: Irgacure 819 (Ig819) was purchased from VWR and all the other chemicals were purchased from Sigma-Aldrich and used without further purification.

DLP 3D Printing of Superhydrophobic Objects: The inks used for DLP 3D printing consisted of 30 wt% butyl acrylate as the monomer, 20 wt% ethylene glycol dimethacrylate (EDMA) as the crosslinker, 50 wt% 1-decaol as the porogenic solvent, and 2 wt% Ig819 as the photoinitiator. All components were mixed in an amber vial and ultrasonicated in a sonic bath (Elmasonic S 30H, frequency 37 kHz and power 80 W) for 30 min to obtain a homogenous ink solution.

The superhydrophobic 3D structures were printed by using a commercial desktop DLP printer (Miicraft Prime 110). The printer is based on a LED projector (385 nm) with an intensity of 1.0 mW cm$^{-2}$ at the vat and a XY resolution of 40 μm. After printing, the 3D-printed objects were immersed in acetone for 24 h to remove the unreacted monomers and porogens.

2PL Patterning of 3D-Printed Structures: The superhydrophobic truncated pyramids were taken out of acetone and rapidly immersed in the patterning mixture, hydroxyethyl acrylate (98 wt%) and Ig819 (2 wt%). 2PL was performed using a Photonic Professional GT instrument (Nanoscribe GmbH), using a laser with a central wavelength of 780 nm, with a 25×/NA 0.8 objective lens (Zeiss LCI Plan-Neofluar 25×/0.8) in
dip-in mode. The focal point was manually set at photoresist–glass substrate interface prior to any lateral stage translation. The volumes for patterning were scanned using galvanometer scanners in the xy-plane and objective translation along the \(z\)-axis, with a scanning speed of 10 000 \(\mu m^{-s}\) and a laser power of 35 mW. The scanned volumes were constructed with a hatching distance of 0.5 \(\mu m\) and a slicing distance of 1 \(\mu m\). If relevant, the volumes were split using 250 \(\times\) 250 \(\times\) 100 \(\mu m^3\) rectangular domains. The scanned volumes were constructed with the xy-origin along their center-of-mass and aligned on the truncated pyramid manually. A semiautomatic alignment of the scanned volumes on the truncated pyramids, using a preliminary manual coordinate spotting, was used for the conductive patterns. The scanned volumes for the surfaces parallel to the glass substrate were constructed as 100 \(\mu m\) tall right prisms of the patterns, located at 60 and 40 \(\mu m\) below and above the theoretical z-location of the surface. The scanned volumes for angled surfaces were constructed as 50 \(\mu m\) tall right prism of the patterns, and angled according to the theoretical angle of the surfaces. All the printing jobs were computed using DeScribe, and from STL files constructed using Blender.

Supercritical Drying: The 2PL patterned objects were first immersed in acetone for 24 h for solvent exchange, followed by CO\(_2\) supercritical drying to avoid shrinkage of the nanoporous structures, which is crucial to render the object superhydrophobic.\(^{[22]}\) Briefly, the patterned objects immersed in acetone were first transferred to the chamber of the supercritical apparatus (Leica EM CPD030). Then, acetone was replaced by liquid CO\(_2\) by repeatedly flushing with liquid CO\(_2\) and releasing the acetone. Subsequently, the chamber temperature and pressure were increased to 35 °C and 90 bar to maintain the CO\(_2\) in supercritical condition. Finally, the chamber pressure was gradually dropped to atmospheric pressure to release the CO\(_2\). The entire supercritical drying process took about 40 min.

Physical Adsorption Test of polyHEA on the DLP-Printed Porous Structures: To clarify whether the polyHEA patterns on the surface are either due to covalent binding or physical adsorption of polyHEA, the DLP-printed porous structures were immersed into a 10 wt% polyHEA (\(M_n 3.2 \times 10^4 g mol^{-1}, M_w 1.4 \times 10^5 g mol^{-1}\), measured by DMAC-GPC) ethanolic solution for 24 h. Then the structures were subjected to the same acetone exchange and CO\(_2\) supercritical drying as denoted above. The dried 3D structures remain their superhydrophobicity as shown in Video S2 (Supporting Information), indicating that the physical adsorption of polyHEA to the porous surfaces is either insignificant in 2PL or is washed away during the drying process.
Deposition of polydopamine. The 2PL-patterned 3D structures were immersed in a 2 mg mL\(^{-1}\) Rhodamine B solution by dissolving RhB in water. 1 wt% silica nanoparticle suspension was prepared by diluting a commercial silica dispersion (5 wt%, d = 900 nm, Sigma) with water, and 2 wt% silver nanoparticle suspension was prepared by diluting a commercial silver ink (30 wt% in ethylene glycol, Sigma) with water. Then, a droplet (3 µL) of the material solution was used to cover the 2PL-patterned 3D structures, and subsequently retracted by a pipette. Due to the effect of discontinuous wetting, a daughter droplet was left on the 2PL hydrophilic micropatterns, while the unmodified regions remained un-wetted. Upon the evaporation of the daughter droplet, the materials in the solution remained deposited in the hydrophilic micropatterns (Figure 1). For Ag nanoparticle patterned objects, a further annealing step was performed at 150 °C for 30 min.

Deposition of polydopamine. The 2PL-patterned 3D structures were immersed in a 2 mg mL\(^{-1}\) 3-hydroxytyramine hydrochloride tris-buffer solution (tris solution: 10 mM, pH 8.5, Sigma) for 1 h and taken out.

Digital Microscopy: Digital microscope images of the samples were obtained by a Keyence BZ 9000. The images were taken at several different angles to reveal the 3D structure.

Confocal Microscopy: Confocal microscope images of the samples patterned with fluorescent dyes (Rhodamine B) were taken by Zeiss LSM 800. Due to the highly porous nature of the 3D-printed objects, they were not transparent enough for making 3D z-stack images, therefore only dye patterns on the top surfaces of the 3D structure were visualized.

Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray (EDX) Mapping: The samples were sputter-coated with a 10 nm gold layer, and imaged using a Zeiss Leo 1530 scanning electron microscope operating at 5.0 kV.

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS): ToF-SIMS was performed on a TOF.SiMM5 instrument (ION-TOF GmbH, Münster, Germany). This spectrometer is equipped with a Bi cluster primary ion source and a reflectron type time-of-flight analyzer. UHV base pressure was <2 × 10\(^{-9}\) mbar. For good mass and lateral resolution the Bi source was operated in “delayed extraction” mode providing unbunched Bi\(^{+}\) primary ion pulses at 25 keV energy. Delayed extraction mode further reduces topography artefacts and charging effects. Despite that an electron flood gun was used to compensate sample charging, the primary ion beam was rastered across a 500 × 500 µm\(^2\) field of view on the sample, and 128 × 128 or 256 × 256 data points were recorded. Spectra were calibrated on the omnipresent OH\(^{-}\), C\(^{-}\), C\(^{2-}\), C\(^{4-}\), and Cl\(^{-}\) peaks. Based on these datasets the chemical assignments for characteristic fragments were determined. Raw data and analysis files are available on Radar4KIT (https://doi.org/10.35097/570).

Conductivity Test: The electrode patterns were contacted by source measurement unit (SMU) needles prolonged with a thin gold wire. Delayed I–V curves from −1 to 1 V were recorded using an Agilent 4156C parameter analyzer. The resistance is extracted from the slope of the I–V curves and is computed on Origin.

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.

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