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pH-sensing multimaterial microrobots 4D printed using automated alignment

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Chemical sensing is an important requirement in many research and industrial applications. To be able to detect chemicals in a microscale region, the sensor in question must be miniaturized. Specialized microrobots are a promising option to accomplish this task due to their small size. With the recent advances in micro 3D printing technologies, namely two-photon polymerization (2PP), the development of microrobots has considerably matured [1]. Moreover, optical trapping techniques that use near-infrared light can be used to precisely transport and manipulate such microrobots [2]. Since microrobots that can change rapidly and reversibly allow for new applications, smart materials enabling 4D printing have also been of great interest in the field [3]. Micro 4D printable pH-responsive hydrogels are useful for microscale sensing, allowing for the identification of microscale regions with different pH levels simply through microscopic observation of their shape change.

In this work, we present the fabrication, characterization, and actuation of a multimaterial microrobot for pH sensing. **Figure 1** shows the fabrication process for printing the microrobots, for which we have developed an automated alignment procedure to print the hydrogel sensing element onto the desired position of the backbone. The alignment algorithm uses binary thresholding, Canny edge detection and a circle Hough transformation to perform rotational and translational alignment. This algorithm thus minimizes alignment errors that may arise by performing the alignment manually. **Figure 2** shows an SEM image of the multimaterial microrobot, as well as optical microscope images of the fabricated microrobot during actuation.

Design of experiments was used to obtain hydrogel printing parameters that result in maximum material actuation from acidic to neutral pH, while maintaining a printing accuracy - *i.e.*, size match between the 3D design and the printed structure - above 90%. The crosslinking density of the hydrogel material is a critical aspect here, and a trade-off between swelling and printing accuracy needs to be considered. While a lower degree of crosslinking facilitates swelling, it can at the same time compromise the structural integrity of the sensing element. Our study confirms this, as the design of experiments shows that the hatching distance and exposure dose have the largest impact on both swelling and printing accuracy. By using the generated model to predict the optimal set of parameters, a 32 ± 3 % length increase of the sensing element was obtained when switching from acidic to neutral pH. This significant size change within seconds shows that the hydrogel is well suited for visually observing local pH changes.

To confirm that the microrobot can be manipulated using optical trapping, printed microrobots were transferred to an optical trapping setup based on two sets of counter-propagating laser beams, as shown in **Figure 3a**. The printed spheres are trapped between the foci of the top and bottom beams. Several traps can be generated using the digital micromirror device to allow for the simultaneous manipulation of multiple traps [4]. A trapped microrobot could be translated (**Figure 3b)** and rotated (**Figure 3c**) over the span of a few seconds**.**

To conclude, we designed and fabricated multimaterial microrobots, examined their swelling behavior, and controlled them using optical trapping. The fabrication was streamlined using automated alignment and the smart material's response was optimized using design of experiments. The microrobots show promise for localized pH sensing due to their responsiveness and maneuverability. Future work will focus on characterizing the sensitivity of the microrobot to pH changes and on testing the microrobots for localized sensing applications in microscale regions.

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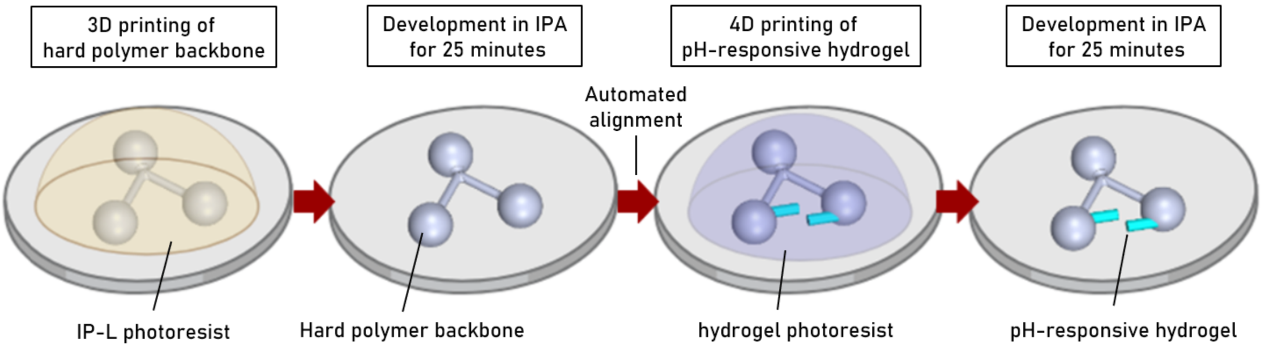

Figure 1. Process flow for fabricating microrobots using 2PP on a Nanoscribe Photonic Professional GT+ system. The hard polymer backbone has three 10 µm diameter spheres that act as optical trapping points for controlling the microrobots.

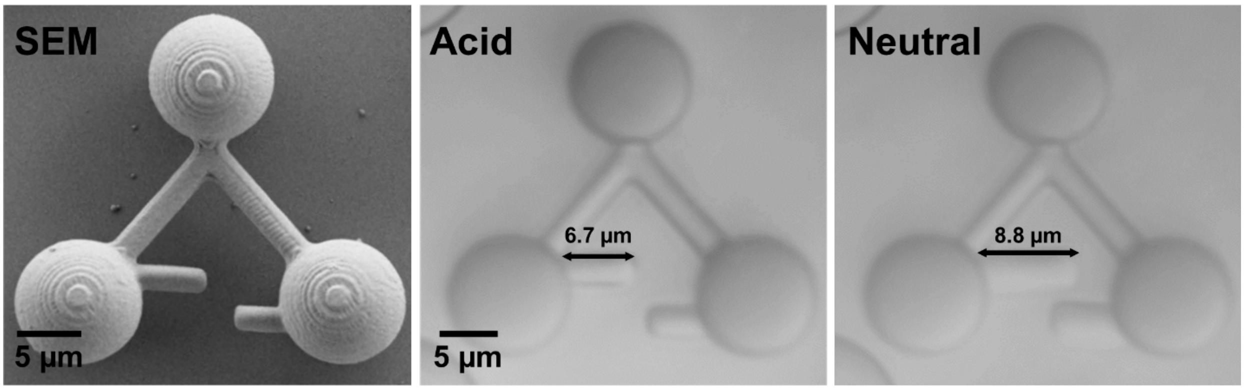

Figure 2. Multimaterial microrobot: Scanning electron micrograph and optical microscope images in different solutions, which show the hydrogel sensing element's change in length from acidic to neutral environments.

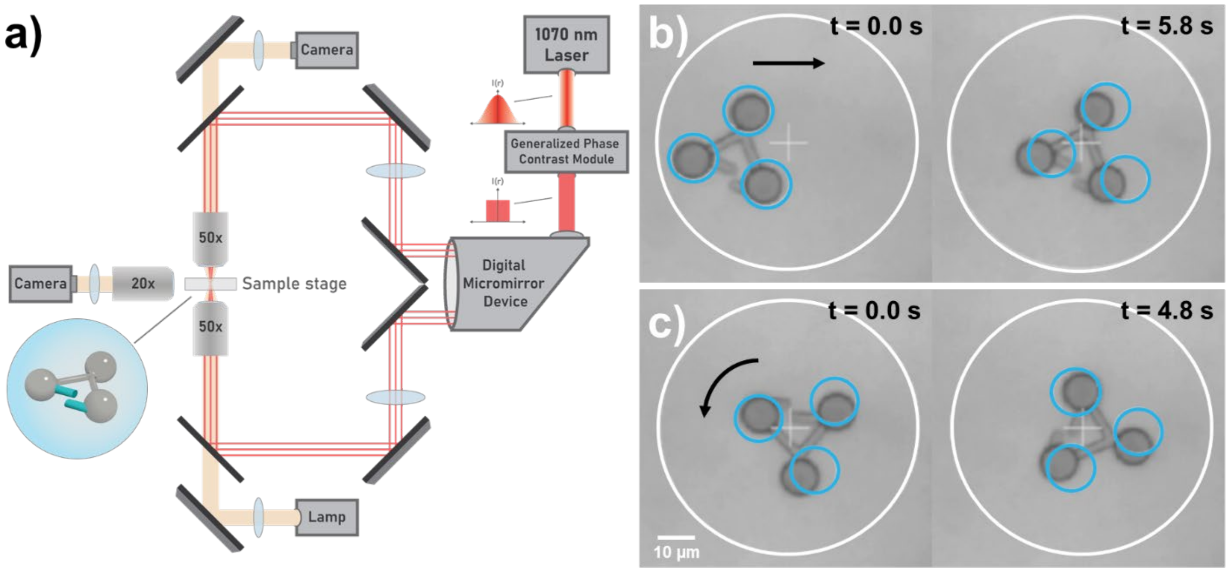

Figure 3. a) Schematic of the optical trapping setup used. b) Snapshots of the microrobot being translated from left to right. c) Snapshots of the microrobot being rotated counterclockwise. The blue circles show the position of the optical traps while the white circle shows the available optical trapping area.