FACILE FABRICATION OF MULTIPLE HYDROGEL ATOMIC FORCE MICROSCOPE CANTILEVERS VIA CAPILLARY FILLING AND ULTRAVIOLET CURING IN AN ALIGNED PDMS MOLD PAIR

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ABSTRACT

This paper reports the facile fabrication of multiple hydrogel atomic force microscope (AFM) cantilevers via capillary filling and ultraviolet (UV) curing in an aligned polydimethylsiloxane (PDMS) tip and beam mold pair. A subsequent single UV exposure replicates multiscale structures, 15 PEGDA AFM cantilevers with their tip radii of ~200 nm, simultaneously. Then, imaging tips of PEGDA AFM cantilevers are sharpened to ~50 nm via oxygen plasma ashing and metal strips are partially deposited by using a shadow mask. Fabricated PEGDA AFM cantilevers are thoroughly characterized and used for nanoscale imaging of HeLa cells, calibration gratings, and DVD media.

INTRODUCTION

Polymeric cantilevers [1, 2] have drawn continuous attention due to their intrinsic low elastic moduli and ease of manufacturing compared to conventional counterparts made from silicon-based materials. Standoff detection of energetic materials by infrared sensitive hydrogel cantilevers was reported [3]. Most recently, hydrogel (PEGDA) AFM cantilevers showed unique capabilities including geometry, property, and functionality tuning by compressible replica molding, combination of materials selection and mold design, and encapsulation of functional nanomaterials [4]. However, the fabrication of PEGDA AFM cantilevers is not yet parallelized and requires a separate tip assembly process for tuning the tip sharpness via mold compression. Due to such a slow fabrication, many promising applications of hydrogel AFM cantilevers are delayed. Here, we report a novel method for fabricating multiple PEGDA AFM cantilevers without requiring the tip attachment.

BATCH FABRICATION

Figures 1 and 2 show the overall concept and detailed sequential steps of the proposed fabrication scheme, respectively. A wafer with 600-nm thick silicon dioxide patterned is etched by using 30 wt% KOH solution at 70 °C for 3 hr to obtain the 150-μm deep base master mold. The negative silicon base master mold is treated with trichlorosilane and replicated with PDMS to obtain the positive PDMS base mold. Then, the positive PDMS base mold is treated with trichlorosilane and replicated with PDMS to obtain the negative PDMS base mold. Another silicon wafer with 600-nm thick silicon dioxide patterned is etched for 30 min at the same conditions to make negative pyramid tip structures and then patterned with SU-8 to make 200-μm long, 40-μm wide, and 15-μm thick negative beam structures that are aligned with KOH-etched tip structures. The wafer is then replicated with PDMS to obtain positive tip and beam structures. The PDMS wafer

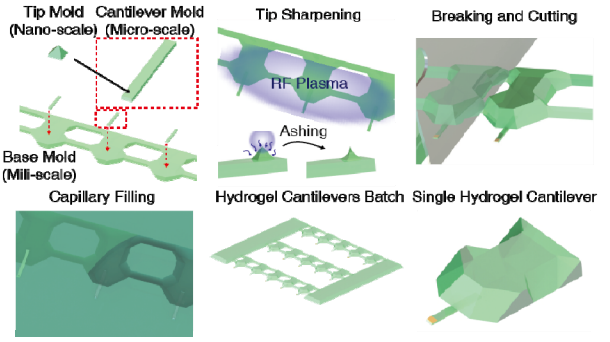

Figure 1: Overall fabrication concept of multiple hydrogel AFM cantilevers via capillary filling and UV curing in an aligned PDMS mold pair.

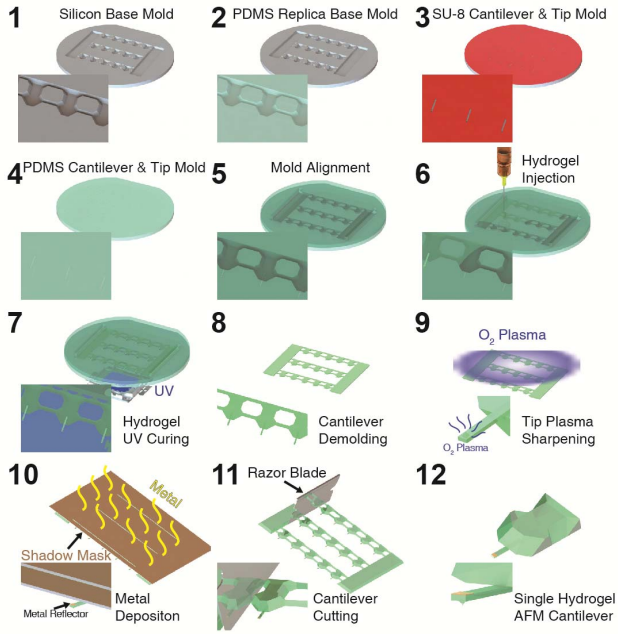

Figure 2: Fabrication process of multiple hydrogel AFM cantilever; (1-2) Silicon base mold fabrication with KOH etching process and PDMS replication. (3-4) Cantilever mold fabrication with SU-8 photolithography and PDMS replication. (5-6) Mold alignment and hydrogel injection. (7-8) Hydrogel curing with UV exposure and demolding. (9) Oxygen plasma sharpening. (10) Metal deposition using shadow mask. (11-12) Cutting a series of cantilevers for a single hydrogel cantilever.

is replicated again with PDMS to invert the tip and beam structures. Among two final PDMS molds, one includes thick base structures and the other contains tip and beam structures (Figures 3 (a) and (b)). After the two PDMS molds are aligned using the setup shown in Figure 3 (c), hydrogel (PEGDA Molecular weight 250 and 1 wt% phenylbis (2,4,6-trimethylbenzoyl) phosphine oxide as a photoinitiator) solution is introduced into the channel by

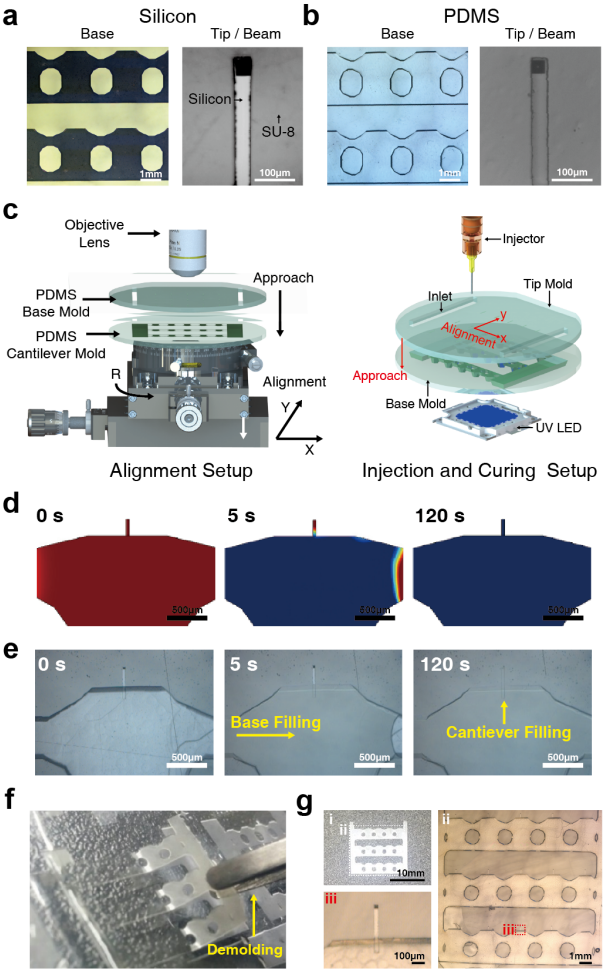

Figure 3: (a) Silicon base and silicon/SU-8 tip/beam master molds. (b) Replicated PDMS base and tip/beam molds. (c) Setup for mold alignment, injection, and curing. (d-e) Simulation and experiment results showing PEGDA filling in the PDMS mold pair. (f) Picture of demolding process. (g) Picture (i) and microscopy images (ii-iii) of demolded hydrogel cantilevers.

the capillary force. The hydrogel filling takes about 2 min in experiment that shows good agreement with the finite element simulation result (Figures 3 (d) and (e)). Then, the introduced hydrogel is cured by UV (405 nm) exposure using the injection and curing setup shown in Figure 3 (c). Cured hydrogel cantilevers are demolded and post-processed using oxygen plasma and metal deposition.

Hydrogel tip can be treated by oxygen plasma to increase the tip sharpness (Figure 4 (a)) [5]. The fabricated batch of hydrogel cantilevers is oxygen plasma treated by using a reactive ion etcher (RIE 80 plus, Oxford Instrument) at 43 W forward power and 30 mTorr with 6 sccm oxygen flow. Figure 4 (b) shows scanning electron micrographs of hydrogel tips before and after the oxygen plasma treatment. The tip radius is improved from 200 nm to ~50 nm. Then, thin metal film is partially deposited near the free end of cantilever through a shadow mask which is prepared by laser machining (DM80FC, 21 Century Corp.). The oxygen plasma treated batch of hydrogel cantilevers is placed under and aligned with the shadow mask as shown

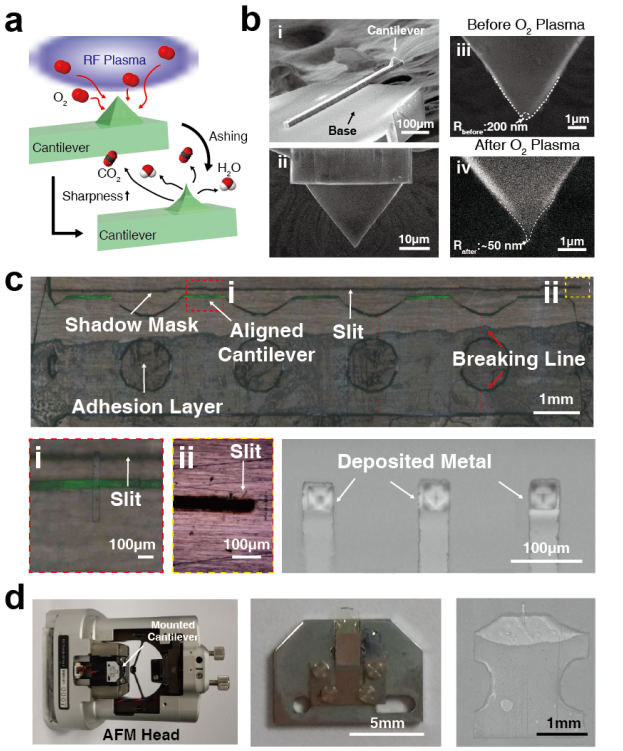

Figure 4: (a) Schematic and (b) scanning electron micrographs of oxygen plasma tip sharpening. (c) Multiple cantilevers aligned onto a stainless steel shadow mask. (d) Individualized hydrogel cantilever before and after mounting in a commercial AFM probe holder.

in Figure 4 (c). 50-nm thick copper is deposited by using thermal evaporator (Customized model, Daedong Hitech) at 3×10^{-6} Torr with 0.6 nm/s rate.

Finally, hydrogel cantilevers are individualized by simple cutting with a razor blade and separated by bending the connection frame ~90° with a tweezer. When the connection frame is being bent, the highest bending stress is generated in the middle, thus hydrogel cantilevers are easily separated. Then, the individualized hydrogel cantilever is mounted on a commercial AFM probe holder (NX10, Park Systems) (Figure 4 (d)).

CHARACTERIZATION

Prior to AFM imaging, hydrogel cantilevers are thoroughly characterized. Figure 5 (a) shows the thermal tune for the calibration of spring constant. The obtained spring constant is 5.85 N/m showing good agreement with the value calculated, 6.12 N/m. Figure 5 (b) shows amplitude and phase spectra near the fundamental flexural mode obtained by frequency sweep of the dither piezo actuator in the probe holder. The resonance frequency measured is 57.85 kHz. Figures 5 (c) and (d) shows force-distance and amplitude (or phase)-distance curves, respectively. From the force-distance curve, we can measure spring constant and sensitivity of the hydrogel cantilever. Both amplitude-distance and phase-distance curves show the bi-stable region between 8 and 16 nm in the distance and stable regions below 8 nm and above 16 nm upon the amplitude modulation for non-contact mode imaging [6]. The stable region above 16 nm is wide enough

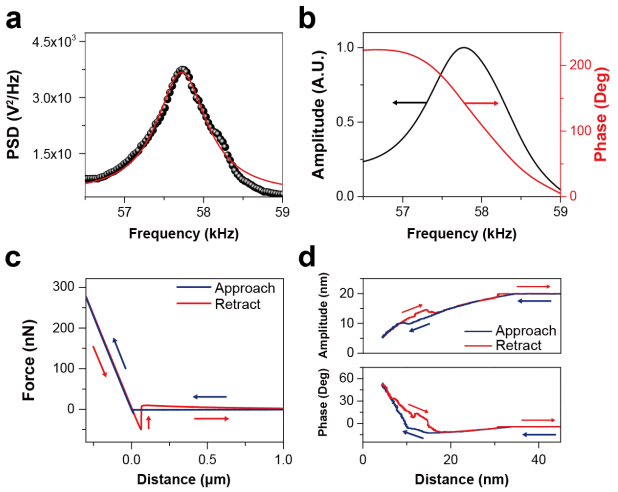

Figure 5: (a) Thermal noise, (b) resonant spectra, (c) force-distance curve, and (d) amplitude (or phase)-distance curve of fabricated single hydrogel cantilever.

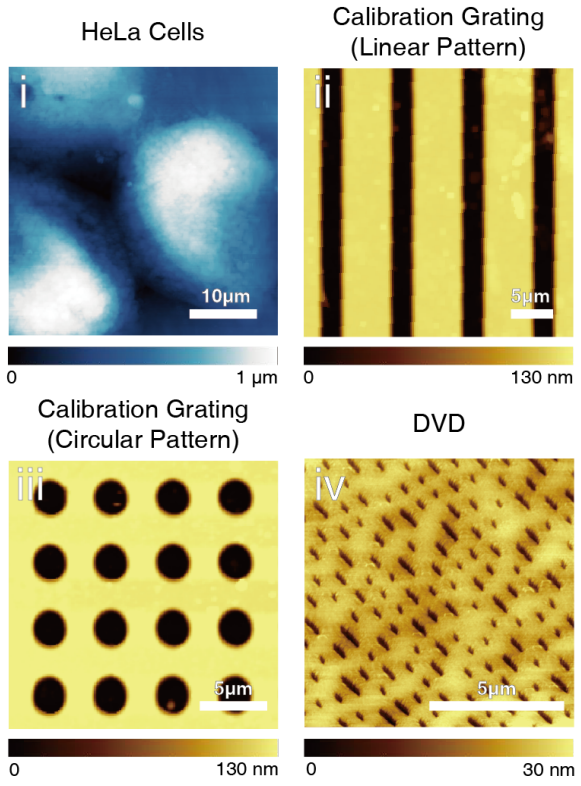

Figure 6: AFM imaging results using hydrogel cantilever; (i) HeLa cells, (ii) Linear and (iii) circular patterns in calibration gratings, and (iv) DVD media.

to guarantee true non-contact mode imaging.

Finally, characterized hydrogel cantilevers are used for imaging fixed HeLa cells in phosphate-buffered saline (PBS) solution, calibration grating with circular and linear patterns (HS-100MG, BudgetSensors), and DVD media with written data bits in air (Non-contact amplitude modulation mode at 20 nm free vibration amplitude, 16 nm set-point amplitude, and 0.5 Hz scan rate) (Figure 6).

CONCLUSION

We report new batch fabrication method for hydrogel AFM cantilevers by applying the UV curing in the aligned PDMS mold pair. The method demonstrated here can be extended to a wafer-level batch fabrication of hydrogel AFM cantilevers and especially useful to make relatively thick structures which cannot be realized by spin casting and UV curing due to the relatively low viscosity.

ACKNOWLEDGEMENTS

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