Surface Modification With Self-Assembled Monolayers for Nanoscale Replication of Photoplastic MEMS

Gyu Man Kim, Beomjoon Kim, Maik Liebau, Jurriaan Huskens, David N. Reinhoudt, and Jürgen Brugger

***Abstract*—A release technique that enables to lift microfabricated structures mechanically off the surface without using wet chemistry is presented. A self-assembled monolayer of dodecyltrichlorosilane forms a very uniform ∼1.5-nm-thick anti-adhesion coating on the silicon dioxide surface, on full wafer scale. The structural layers are formed directly onto the organic layer. They consist here of a 100-nm-thick aluminum film and a high-aspect ratio photoplastic SU-8 structure. After the microfabrication the structure can be lifted off the surface *together with* the aluminum layer. This generic technique was used to make a variety of novel structures. First, aluminum electrodes that are embedded in plastic are made using lithography, etching and surface transfer techniques. Second, using a patterned monolayer as defined by microcontact printing, resulted in a spatial variation of the surface adhesion forces. This was used to directly transfer the stamped pattern into a metal structure *without* using additional transfer etching steps. Third, the monolayer's ability to cover surface features down to nanometer scale was exploited to replicate sharp surface molds into metal coated photoplastic tips with ∼30-nm radii for use in scanning probe instruments such as near-field optical techniques. The advantage compared to standard sacrificial layer techniques is the ability of replication at the nanoscale and the absence of etchants or solvents in the final process steps. [672]**

***Index Terms*—MEMS, nanoscale, replication, self-assembled monolayer (SAM), SU-8.**

I. Introduction

SURFACE micromachined features in microelectromechanical systems (MEMS) are built up, layer by layer, on the surface of a substrate. The functional structures are then typically released by selective removal of a sacrificial layer using a wet etch step to obtain freestanding movable parts of a microactuator device [1]–[3].

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Sometimes, however, wet chemistry may induce manufacturing and process design drawbacks. One problem for example is the risk of permanent attachment of slender structures to the underlying substrate after the final rinsing and drying step. Other potential problems are mainly the etch selectivity between the sacrificial and structural layers, and furthermore the risk of surface contamination, in particular when chemically active surfaces for (bio)chemical sensor or microfluidic devices are constructed.

Dry-etchable organic layers have also been introduced in MEMS fabrication techniques to be used as a sacrificial layer [4], [5]. The absence of fluidics prevents release-related stiction and eliminates furthermore chemical cross-contamination. In another application, the grafting of densely packed organic molecules on microstructure surfaces is used to enhance the lubrication between moving MEMS parts [6], which may also be useful as a release layer.

An entirely different method of constructing surface micromachined devices completely detaches the individual parts from the surface to be used as stand-alone MEMS parts. In this case the surface only serves as support material, which is often prestructured with etched mold features that are to be replicated into the released devices. The substrate wafer containing the mold is later completely dissolved by wet or dry etching. For instance silicon nitride cantilever probes for atomic force microscope (AFM) featuring nanometer sharp pyramidal tips are microfabricated in large volumes using such a wafer-dissolving method [7].

An alternative way would be to use deposited or grown sacrificial layers between mold and functional layer. However, the typically 1-μm-thick sacrificial layers which are needed to allow a high lateral underetch rate, are not suitable for nanoscale replication since the additional layer smoothens the sharp mold and prevents accurate replication at the required scale (10 nm). Only very recently, a novel approach for low-cost wafer-scale fabrication of AFM probes and probe cassettes has been demonstrated without a wafer-dissolving step. The probes were constructed by using photoplastic material (EPON-SU-8) as a functional layer and surface released by using an ultra-thin (20-nm) wet-etched sacrificial Al layer, yielding tip radii in the order of 30 nm [8].

The interest of using molding processes for micro- and nanoscale device fabrication is obvious since it allows simultaneously high-resolution, low-cost processes and wafer-scale compatibility. For instance, in the field of lab-on-the-chip devices [9] with chemically functionalized surfaces, or in

the field of nanoelectromechanical systems (NEMS) [10] with fragile mechanical elements, it would be preferable to use release techniques that eliminate wet-processing steps totally. Dry-releasing a surface micro/nanofabricated device with patterned metal electrodes on the bottom side which are protected during the process, would enable the design and integral manufacturing of novel devices with functional metal electrodes and wiring, on the bottom *and* on the top side of, e.g., thin films. For instance, metallic nanowires can be transferred by mechanical lift-off from a graphite surface and are embedded in a polystyrene film, as recently shown by [11].

In view of making this technique available for silicon microfabrication processes, we developed a release technique using a self-assembled monolayer (SAM) on silicon dioxide, which serves as anti-adhesion layer between functional structures, which are then separated by mechanically peeling them off the surface [12]. Interestingly, the SAM that is covered by a metallic and other functional layer provides enough stability to support most steps typically involved in MEMS processing. Our approach differs from other known methods of using organic sacrificial layers including the grafting technique, since the sacrificial organic layer is only $\sim$1.5 nm thick. This is the basic condition for our application where we aim at replicating surface features down to the nanometer scale. A conformal coverage of surface features at this length scale is therefore a prerequisite, and we will demonstrate that our technique enables, e.g., the integral construction of functionalized elements with nanoscale features as required for advanced scanning probe instruments. In particular, novel nanomolded transparent photoplastic probes for the scanning near-field optical microscope (SNOM) have been made by the new technique [13].

The surface modification with the self-assembled monolayer method is generic and might allow the construction of a variety of novel surface microfabricated devices, including high-aspect ratio plastic devices with embedded micro- and nanoscale metal patterns. Particularly interesting is to use the dry-mechanical lift-off technique on *patterned* SAM layers, defined by microcontact printing (μCP) [14]. We will show that it allows to *directly* transfer the SAM pattern into a metal structure without additional transfer steps. In this paper we will discuss details on the process steps. First a brief description on the SAM formation on planar and pre-structured silicon dioxide surfaces is given, then details on metal layer formation on the SAM and patterning by lithography, on the processing of photoplastic EPON-SU-8, and finally on mechanical release, are provided.

II. Process Details

A variety of process combinations using a SAM as a sacrificial layer are possible and have been studied depending on the final device aimed at. We performed several case studies to build up a knowledge base for further process design and fabrication sequences enabling novel devices to be made. In particular, we have studied SAM formation on planar and prestructured surfaces. We have used homogenous and patterned SAMs, and combined them with patterned and nonpatterned Al layers. We also analyzed combinations thereof. An overview of different cases and combinations involving a SAM release layer, an Al metal layer and an SU-8 plastic MEMS device is shown in Fig. 1. Case 1 describes the situation of a planar surface that is homogeneously coated with an SAM and a patterned Al layer to release the device with embedded metal electrodes. Case 2 shows a planar surface with an SAM structured by μCP, and an unstructured thin Al film, which is disrupted upon mechanical release to transfer the adhesion into a metal pattern. And finally, Case 3 illustrates an oxidation-sharpened pyramidal mold covered down to the apex by the SAM, then covered by Al and a transparent SU-8 probe that is subsequently released from the mold *with* the Al layer attached. In principle, other variations between the cases are possible but are not presented here.

A. Self-Assembled Monolayer (SAM) Formation

Organosilicon derivatives R_nSiX_{3-n}, where X is chloride or alkoxide are well known to form SAMs on hydroxylated surfaces [15], [16], [17]. In our case we start with a (100) single crystal silicon wafer that is covered by a 300-nm-thick thermally grown silicon dioxide SiO_2 layer. Then, a $\sim$1.5-nm-thick SAM is formed on the SiO_2 layer by immersing the wafer for about 2 hours into a solution of 5–10 $\times$ 10^{-3} M *dodecyltrichlorosilane* (DTS) in dry toluene. During this time the process forms a self-assembled network of molecules that are connected to the surface and to each other by covalent bonds. The main observation of a compact monolayer is a drastic reduction of the surface wettability, which is measured by contact angle goniometry. Advancing contact angles of 112° indicated the hydrophobic nature of the prepared layers. A low hysteresis of 17° between the advancing and receding 95° contact angles showed the dense packing of SAMs. For comparison, SAMs of 3-aminopropyltriethoxysilane (APTES) were also prepared. APTES is forming more hydrophilic layers having an advancing contact angle of 67°. The application of different types of monolayers enables the tuning of the surface properties, such as wettability and adhesion, on a wafer scale. SAMs formed by alkyltrichlorosilanes (such as DTS) are stable against detergents and organic solvents and they withstand also acidic solutions, whereas basic conditions may deteriorate the monolayers. The present study applies SAMs of DTS as nonadhesive layers to reduce the adhesion between metal and wafer and to allow mechanically lifting a functional micromechanical device off the surface without release layer etching.

Patterned SAMs of dodecyltrichlorosilane were prepared following the procedure developed elsewhere [14], [18]. It involved the preparation of a polydimethylsiloxane (PDMS; Sylgard 184) stamp patterned with 5-μm-wide lines, the inking of the stamp with the above described solution of DTS in toluene under a dry nitrogen atmosphere, and the printing of this stamp (1 min) on the oxidic surface of the silicon wafer. Hereafter, the wafers were washed with toluene and ethanol, and dried in a nitrogen stream.

B. Deposition and Patterning of Aluminum on SAM

The wafers are then coated by a thin metal layer directly onto the SAM. We used typically aluminum (Al) in a standard resis-

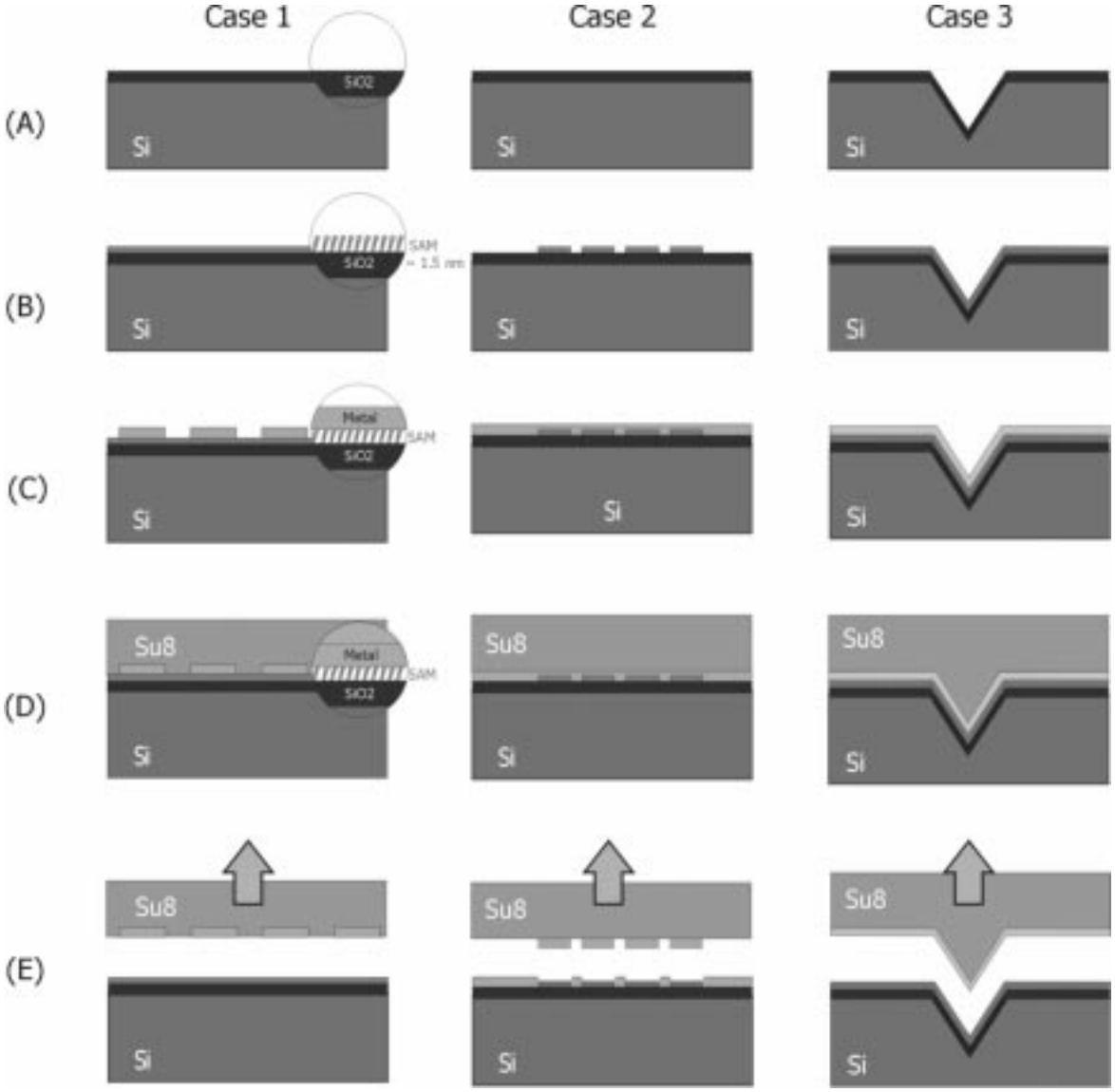


Fig. 1. Overview and process flow of studied "SAMs meet MEMS" experiments. (a) Nanomold making and SiO_2 growing; (b) SAM coating; (c) metal deposition and patterning; (d) SU-8 process; (e) lift-off. CASE 1: Photoplastic MEMS with flat, embedded electrodes fabricated on a planar surface with homogenous SAM coating. CASE 2: For transferring a SAM pattern made by μCP directly into a thin Al film by mechanical layer disruption. CASE 3: Replication of a photoplastic near-field optical probe *with* Al coating from an oxidation-sharpened mold. In principle, other variations of these cases are possible but are not presented here.

tive evaporation tool, but any other metal such as gold, chrome, etc., is possible. The distance between the evaporation source and the substrate was 300 mm and the base pressure was 6×10^{-6} mbar. Deposition time was typically $\sim$3 min for 100-nm-thick layers. Estimated temperature at the sample surface was below 50 °C. The Al layer (on the SAM) was then patterned by standard contact mode microlithography using UV exposure, followed by wet Al etching using phosphoric acid and resist removal. No peculiarities were observed during these steps, indicating that the SAM is not affecting the Al film processing.

C. EPON SU-8 Process

For the construction of the plastic MEMS devices we used EPON SU-8 because of its outstanding properties allowing high-aspect ratio structures to be made with near-UV exposure equipment [19], [20], [21]. In a single spin the negative, epoxy type, near-UV (365 nm) photoresist can be built-up to a thickness up to 500 μm having aspect ratios up to 25. The SU-8 process steps are performed directly on the SAM/Al double-layer. A schematic illustration of the resulting sandwich layer consisting of SiO_2/SAM/Al/SU-8 is also shown in Fig. 1. Because of its capacity to fill surface features very conformal down to the nanometer range, SU-8 is an excellent material for novel scanning probe instruments. Furthermore, due to its high transparency, it can be used for optical applications (Optical MEMS) and nanooptical devices (SNOM).

III. Results

A. Mechanically Releasing the Photoplastic SU-8 With Al Pattern (Case 1)

Because of the low adhesion between the Al and the surface induced by the SAM as molecular "nanocarpet" the photoplastic structure can easily be detached from the silicon wafer

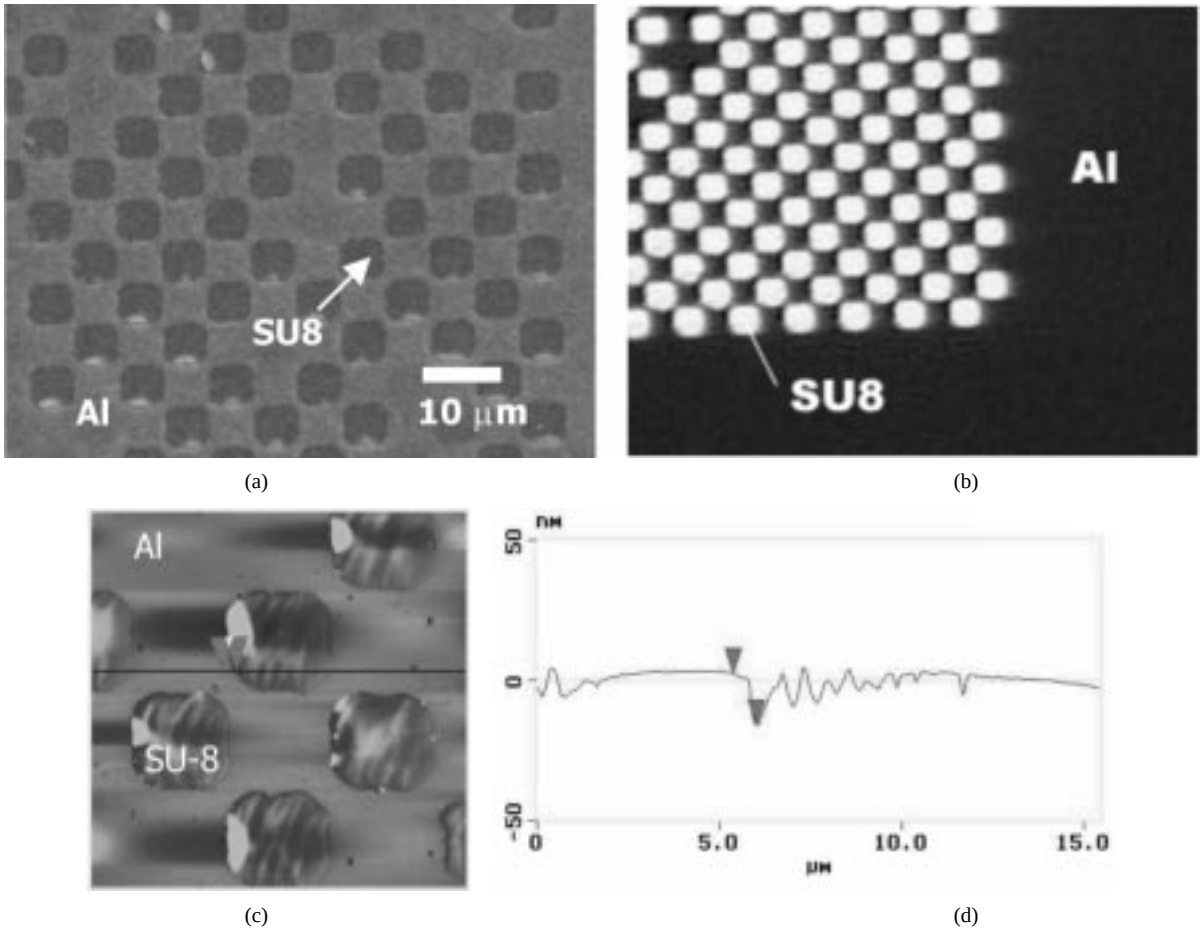
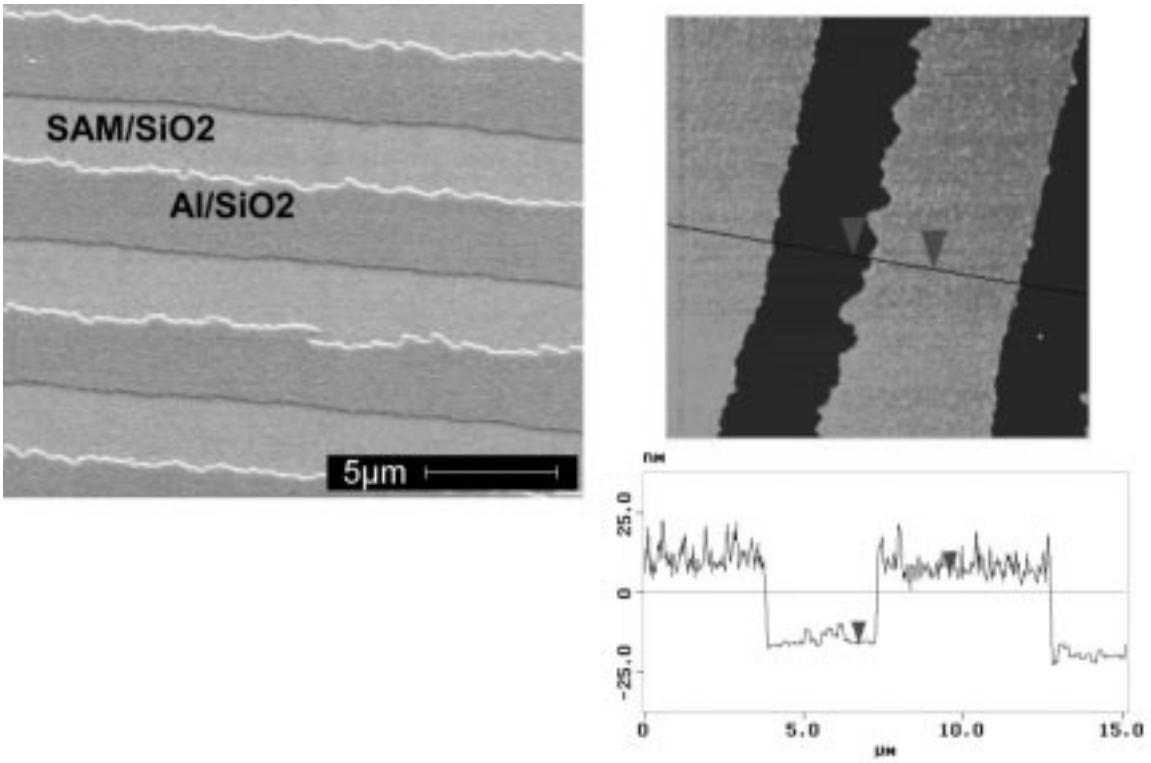


Fig. 2. Results of Case 1. (a) SEM image of a released photoplastic structure with patterned Al layer. (b) Optical microscope image using bottom-side illumination showing the transparent (SU-8) and opaque (Al) regions, the illuminated squares are 5 μm × 5 μm. (c) Atomic force microscope image showing a 15-μm × 15-μm section. (d) Line scan across the checkboard pattern shows that the Al pattern is embedded in the SU-8. The ripples in the SU-8 result from plastic deformation during the mechanical release step.

by means of microtweezers. Fig. 2(a) shows a scanning electron microscope (SEM) image of a released photoplastic structure with a patterned Al layer. The optical microscope image in Fig. 2(b) using bottom-side illumination shows clearly the open (SU-8) and covered (Al) pattern. There are two unique assets to this technique: first the Al pattern is embedded in the SU-8 and, second, the metal surface is very smooth with less than 0.8 nm mean surface roughness as measured by AFM. It therefore allows fabrication of ultra-flat electrode structures, which become increasingly important for nano-scale and molecular electronics. The AFM scan image and corresponding cross sectional analysis over a 100-nm-thick and 5-μm-sized lithographic Al pattern are shown in Fig. 2(c) and (d). The missing pads in the pattern are due to a lithographic and etching defect, and not due to the release step. The ripples in the SU-8 are due to the plastic deformation during mechanical release step using the microtweezers.

B. Direct Formation of a Metal Pattern on Photo-Plastic Using a Patterned SAM (Case 2)

We studied the mechanical release technique not only on plane SAMs but also on patterned monolayers as they can be formed by μCP. In this situation, the surface adhesion force between the Al layer and the surface varies according to the spatial variation of the SAM. In other words, the part of the Al thin film situated on top of the SAM nanocarpet, has a weaker adhesion to the surface than to the SU-8 structure and is hence lifted off the surface together with the SU-8. The part of the Al film directly in contact with SiO_2 surface adheres stronger to the surface than to the SU-8, and therefore remains on the surface. During the release step the thin Al film mechanically disrupts according to the SAM pattern layout. Fig. 3(a) shows a scanning electron micrograph of the metal pattern on the SiO_2 surface after mechanical rupture of a 20-nm-thick Al film. Due to a specific one-directional pull-off movement, one of the edges is less defined than the other. An AFM scan image and single line scan is shown in Fig. 3(b). The 5-μm-large Al lines are still attached to the silicon dioxide where it was not in contact with the stamp and therefore remained *uncoated* by the SAM. The single-sided poor edge definition is attributed to directional forces occurring during the lift-off step. In order to improve the edge definition and to reduce lateral dimensions, further systematic experiments are needed. The presented results show that this technique enables to directly transfer a structured SAM pattern into a corresponding metal structure without intermediate transfer steps. Thereby, the metal pattern on the two separated surfaces is complementary in its shape. The resulting thin metal pattern can be used directly or could form a robust mask for subsequent postprocessing such as



(a) (b)

Fig. 3. Results of Case 2: A μCP SAM pattern (stripes of $\sim 5\ \mu$m width) has been coated with 20-nm-thick Al and 150-μm-thick SU-8. Mechanical removal of the composite SU-8 block *with* the attached Al results in patterned disruption of the thin Al layer according to the layout of the patterned SAM layer, which determines locally the adhesion to the surface. This allows to directly transfer a μCP SAM pattern into a metal pattern without additional process steps. (a) Shows an SEM and (b) shows an atomic force microscope image including a single line scan surface roughness measurement. The poor edge definition is attributed to the directional mechanical rupture of the thin Al film during lift-off.

dry etching or plating. Further studies need to be done to understand the mechanism upon rupture of the thin metal film and also to quantitatively understand the adhesion forces.

C. Low-Cost Photoplastic Probe for Scanning Near-Field Optical Methods (Case 3)

The combination of smooth and nanometer precise separation of metallized SU-8 structures have been used together to develop a nanomold technique for the integrated microfabrication of scanning near-field optical microscopy (SNOM) probes. The novel probe is composed of a light-guiding pyramidal tip (SU-8) covered by an opaque layer (Al). A nanoscale aperture at the tip apex is subsequently made by focused ion beam (FIB). This process is adapted from [8] for low-cost photoplastic atomic force microscope probes and modified here to be able to release the SU-8 nanoprobe from the mold together with the Al layer. A reproducibly microfabricated probe that is microassembled and attached to an optical fiber is shown in Fig. 4. The tip has a curvature radius of about 30 nm as lifted-off from an oxidation sharpened silicon dioxide nanomold.

IV. Conclusion

Ultrathin organic self-assembled monolayers were used to functionalize surfaces on wafer level prior to a replication and demolding processes using functional metal and high-aspect ratio photoplastic layers. Our presented technique is useful for a variety of MEMS related applications, where functionalized integrated probes, and double side patterned MEMS structures are relevant. The unconventional method to define first metal patterns before coating them with a photoplastic and then to lift the structure off the surface, allows electrodes to be made on both sides of thin film structures, the bottom-side pattern furthermore being embedded. In addition, since the metal is not exposed to environment (after being coated with the photoplastic) until the last release step, would allow to define clean metal surfaces as they become more and more relevant when interfacing structures at the scale of biological and molecular scales. Here, self-assembled monolayers are used as a sort of nanocarpet to reduce adhesion between a metal layer and the surface. Our technique enables furthermore to directly transfer a μCP SAM pattern into a metal structure without using additional etch steps, which makes it another variation of a low-cost patterning technique. A more profound and quantitative understanding of the adhesion forces involved between the organic molecular monolayer , the silicon dioxide and the metal film is necessary to make further use of the technique. One of the major advantages shown in this paper is the demonstration that the SAM allows to replicate surface mold structures down to nanometer scale, which will allow the design and fabrication of novel functionalized tip sensors for scanning probe methods.

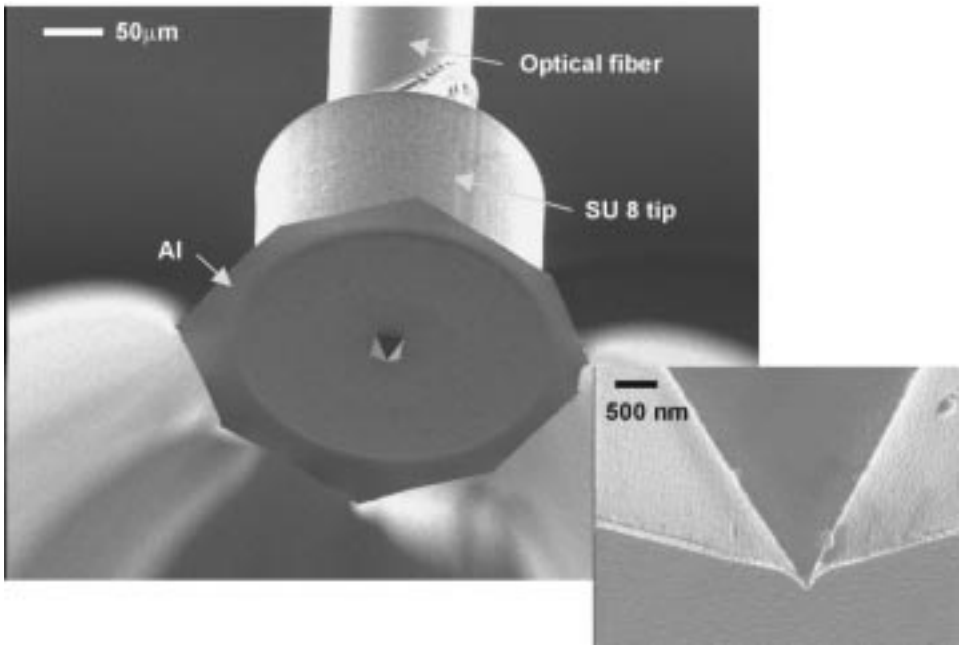


Fig. 4. Results of Case 3. Microfabricated photoplastic SNOM probe microassembled to an optical fiber. The Al layer forms an integral part of the probe and has been made using the combined SAM/MEMS process Case 3. A closeup of a replicated sharp tip with 30 nm tip radius demonstrates the capability of nanoscale replication using the thin SAM layer.

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Jürgen Brugger received the Ph.D. degree from the University of Neuchâtel, Switzerland, in 1995, for a work on microfabricated tools for the atomic force microscope, which included a one-year stay at Hitachi Research, Tokyo, Japan.

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