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Fabrication of Localized Plasma Gold-Tip Nanoprobes with Integrated Microchannels for Direct-Write Nanomanufacturing

Yan Xie, Rajesh Surapaneni, Faisal K. Chowdhury, Massood Tabib-Azar and Carlos H. Mastrangelo
Electrical and Computer Engineering
University of Utah
Salt Lake City, UT, USA
Yanxie411@gmail.com

***Abstract*—We present the microfabrication and characterization of an AFM-tip like device with integrated gas delivery microchannel for the generation of localized microplasmas. The device plasma is generated within a submicron region around its tip for direct-write micro and nanofabrication. The device is fabricated by forming a tall, sharp micromolded gold tip in a KOH etched inverted pyramid followed by thermo-compression bonding and consecutive tip transfer, microfluidic channel patterning and formation of supporting cantilever beam. The tall tip overcomes the height problems of previous designs. Preliminary experiments have been carried out demonstrating the generation of localized microplasma at atmospheric conditions with 1,000V AC stimulation. By mounting the device to a commercialized AFM station and operated in tapping mode, imaging with the same device has also been demonstrated.**

I. INTRODUCTION

With the trend of progressively shrinking the device dimensions, many electronic devices and systems used in modern industry are becoming smaller with their dimensions entering the nanoscale domain. In recent years, the capability to grow, deposit, or manipulate nanostructures such as nanotubes, nanowires and quantum dots has been amply demonstrated, but the techniques used are unable to produce these structures in a reproducible, controlled manner. To realize the potential benefits of these devices and systems, the capability of controlled nano-manufacturing is indispensable.

Controlled nano-manufacturing is defined as the automated, parallel fabrication of individual nanostructures with control over position, size, shape, and orientation at the nanometer scale. Since the development of scanning tunneling microscope (STM) in the late 80's and its close relative atomic force microscope (AFM), many attempts were made by researchers to manipulate nanostructures using atomically sharp tips. When in close proximity to a surface, these devices can locally remove and add material at single point. Single rows of atoms spelling IBM [2] and other words are well known examples of these local probe assembly and manipulation of nanometer and sub-nanometer objects. Atomically sharp tips alone cannot by themselves do more than moving material from one spot to another. A more general capability is thus needed for localized deposition and etching of a diverse set of materials using both reactive and activated gas phase compounds.

II. TALL-TIP PLASMA NANOPROBE DEVICE

In [1], we presented a tip-based device that can process materials at the nanoscale using nano-plasmas. The device consisted of a sharp polysilicon-metal tip integrated with a gas delivering microchannel, both formed on top of a suspension beam. In this device, controlled localized etching and deposition could be accomplished through: (a) an active microchannel gas delivery system that ensures a continuous transfer of the new active species exiting the tip area, (b) a highly localized electric field near the tip and (c) hydrodynamic focusing that improves the plasma localization. The device demonstrated simultaneous capabilities for localized oxidation and plasma etching.

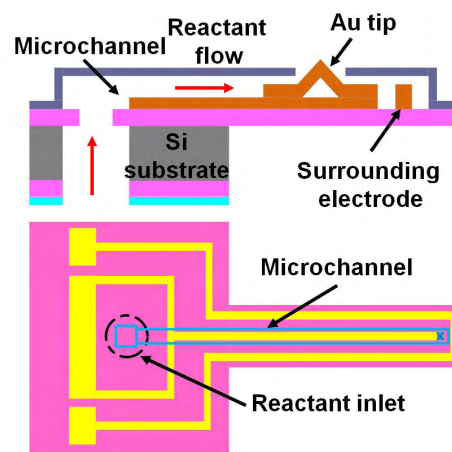

Figure 1. Schematic of new tall-tip plasma nanoprobe device.

This project has been sponsored under DARPA grant N66001-08-1-2042.

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The nanoprobe structure introduced in [1] had two major issues originating from the nature of the materials used in its construction.

The first issue is the realization of a tip that protrudes out of the interior of the microchannel. A protruding tip is necessary to achieve contact of the tip with the processing substrate while clearing the microchannel. In [1] the tip height was determined by the maximum polysilicon thickness practically achievable with conventional LPCVD tubes (~6 μm). This restricted the microchannel height to just about 1 μm requiring a very long sacrificial etch. The resulting shallow microchannel limited the mass transport of reactive gases to the tip yielding slow etching and deposition rates.

The second issue originates from the process used to sharpen the tip. The tip was first sharpened using a timed isotropic wet etch followed by oxidation sharpening. The thicker the polysilicon base film was, the more difficult was to achieve a sharp tip. Due to the non-uniformity of the isotropic tip formation process across the wafer, the yields for devices with sharp tips were low.

In this paper, we present a new, simpler construction method that eliminates both issues. Figure 1 shows the schematic of the new nanoprobe device. The tip height and yield issue is solved using a molded rather than etched tip that is subsequently bonded to the device. In addition, the shallow silicon-nitride based microchannel used in [1] is now replaced with a 16 μm tall polymer channel that can be fabricated and released very quickly. Since we are using molded tips, the depth of the mold is arbitrary and so is the resulting tip height. This permits the fabrication of tall channels that provide a much higher gas transport to the tip region.

III. DEVICE FABRICATION

The new tall-tip nanoprobe device is fabricated using the 8-mask process flow shown in Figure 2. The process starts by molding a sharp Au tip inside an inverted pyramid cavity formed by KOH etching in a (100) silicon wafer. First a 0.5 μm-layer of thermal oxide was grown on a flat substrate to serve as a mask for the mold etching. Next the oxide was patterned and etched using 5:1 BHF. Self-limited 25 μm-deep pyramidal pits were etched in the silicon substrate using anisotropic KOH etching at 60°C. After cleaning the etched samples in piranha, the masking oxide was stripped in 5:1 BHF. Next the substrate surfaces were re-oxidized to form a layer of 0.25 μm of silicon dioxide. This oxide film is needed to protect the silicon mold wafer during the tip release from the mold. The oxidation is followed by sputtering of polycrystalline silicon. The thickness of the polysilicon layer is about 0.5 μm. The polysilicon serves as a sacrificial layer.

Next a 50 nm/2 μm layer of CrAu is sputtered on top of the polysilicon film. The CrAu is lithographically patterned and wet etched to define the bonding patch for the tip. The patterned CrAu patch is next used as a mask to etch the polysilicon below using a SF_6 plasma etch which stops at the underlying oxide film. Next, the molded CrAu layer is freed by sacrificially removing the polysilicon layer using XeF_2. The released floating tip while still seated in the KOH etched

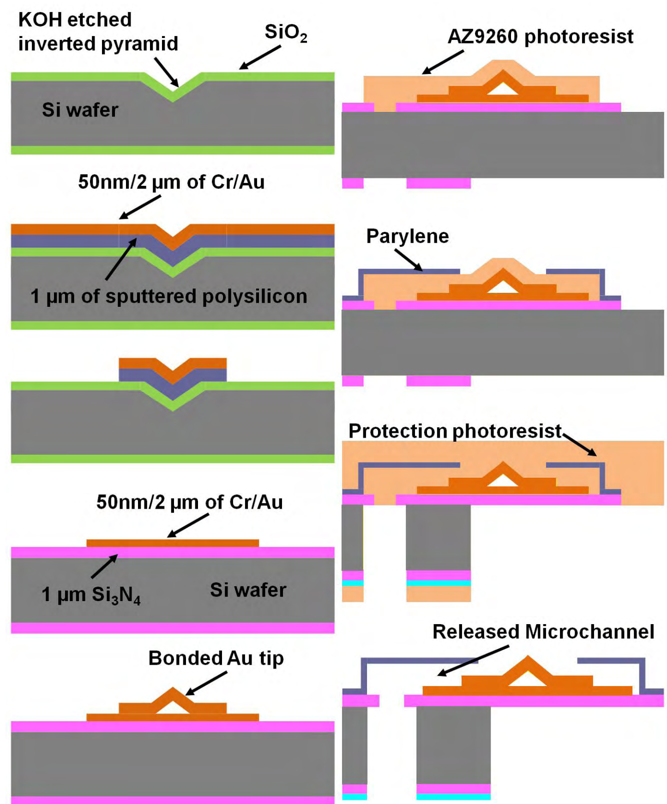

Figure 2. Detailed fabrication procedure of the tall-tip nanoprobe.

inverted pyramid cavity, is Au-Au compression bonded [3] to a second silicon wafer coated with 2 μm of low-stress Si_3N_4 and a 50 nm/ 2 μm CrAu layer. After the bonding, the cavity wafer is removed and the molded tip is thus transferred to the substrate. The tip lead and surrounding electrode are formed by lithography patterning and Au wet etching. The nitride layer is next patterned to define openings for the microchannel gas inlet, and the shape of the released cantilever beam. Next we pattern a layer of 16 μm thick AZ9260 sacrificial photoresist to form the microchannel. The width of the channel is 150 μm. A layer of 1 μm of parylene is deposited to serve as the wall of the microchannel as well as passivation for the electric leads and electrodes. This passivation is necessary to ensure that plasma is only generated around the probe tip. Contact holes for the electrodes and the gas outlet right above the tip apex are next opened by patterning and etching the parylene using oxygen plasma etching. In order to etch the parylene microchannel, a layer of 50 nm of Cr is deposited and patterned using a thick AZ9260 resist layer. The patterned Cr serves as an etching mask for the parylene layer. After the parylene wall is etched, the Cr layer is stripped by wet etching. Next, a second layer of AZ9260 is spin cast on top of the wafer to protect the front side of the wafer.

The wafer backside is next lithographically patterned to define the gas inlet and cantilever beam areas. The backside Si_3N_4 is etched first in CF_4 plasma down to the silicon. Next the wafer backside is etched using DRIE releasing the cantilever and opening up the channel inlet. A layer of 150 nm

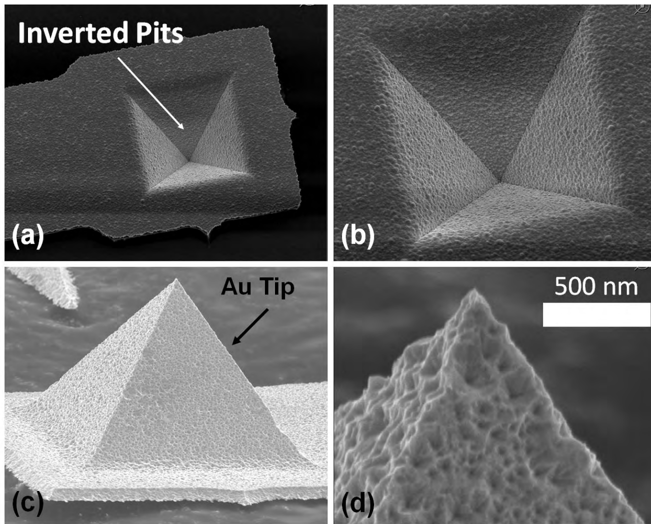

Figure 3. CrAu tip molded from pyramidal pits.

Cr and 16 µm AZ9260 is used as the etching mask for the DRIE etch. The DRIE etch step also forms a trench around each die, helping to separate individual devices. After the devices are singulated by clipping along the DRIE etched trench, the device microchannel is finally released by dipping the entire device in acetone for 30 min. This removes the photoresist sacrificial layer inside the microchannel as well as the resist protective layers. Before the nanoprobe is mounted to an AFM micromanipulator stage, a layer of 0.1 µm Al is also deposited to the backside of cantilever beam to optically determine the vertical displacement of the probe tip to the sample surface.

The basic characteristics of released molded CrAu tips are shown in Figures 3 (a) - (d). When the CrAu is deposited into the KOH pyramidal pit, the metal film conforms to the pit <111> crystal surfaces. When this layer is transferred to another substrate the pit structure is flipped producing a tall pyramidal tip as shown in Figs. 3(c)-(d). Figure 3(d) shows a close up of the tip. The bumped surface roughness is the result of the sacrificial polysilicon film texture. The tip radius is approximately 20 nm.

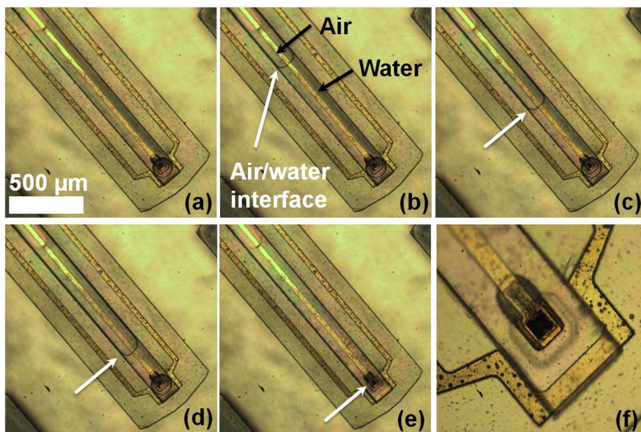

Figure 4. Series of optical pictures demonstrating the successful release of the microchannel.

Figure 4 shows a series of optical pictures of a released microchannel partially filled with water as the sample is dried out. The receding water line demonstrates the complete release of the parylene-wall channel. Figure 5 shows SEMs and optical images of the fabricated devices with a sharp Au tip that protrudes beyond the channel opening. Figure 5 (a) shows a released Au tip molded by KOH etched inverted pyramid bonded to a flat Au coated substrate. Figure 5 (a) also shows the patterned electronic lead and the surrounding electrode formed around the Au-Au bonded tip using wet etching.

In order to test the mechanical strength of the released cantilever nanoprobes, we have fabricated devices with cantilever beams of different shapes. The devices shown in Fig. 4 have rectangular beams. The corresponding width and the length of the rectangular cantilever is 600 µm and 1650 µm, respectively. Figure 5 (b) shows a fabricated device with a triangular shape cantilever beam. Figure 5(c) shows a close up SEM image of a released device near the tip area showing the molded sharp Au tip sitting inside the hollow microchannel. Figure 5 (d) shows an optical image of a device with released triangular cantilever beam. The picture also shows the access hole for the gas inlet.

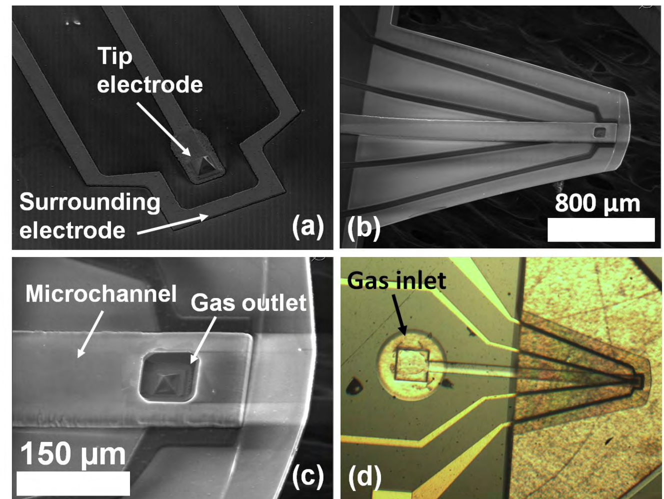

Figure 5. SEM and optical pictures of fabricated tall-tip nanoprobe devices.

IV. Preliminary Experimental Results

Released devices were mounted on test fixtures and subject to the following preliminary tests.

a) AFM Imaging Characteristics: The fabricated nanoprobes can be used to both process and imaging of the sample with the same tip, which greatly reduces the difficulty of locating the desired etch/deposition spot. Figure 6 shows direct AFM imaging of a micromotor stator using the tall CrAu nanoprobe device. In order to obtain the image, the nanoprobe was mounted to a conventional high-resolution AFM manipulator stage. This permits precise positioning of the nanoprobe tip related to the sample with a resolution limit

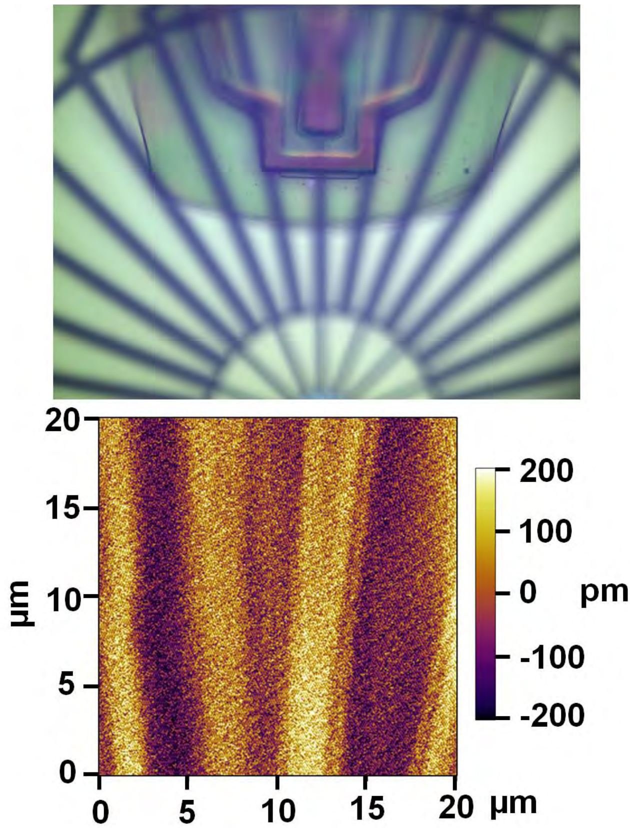

Figure 6. AFM scan over the surface of a micromotor using the protruding tall CrAu tip of our nanoprobe device.

of less than 5 nm. With the device's spring constant and intrinsic frequency measured to be 1.34 nN/nm and 125 KHz, the sample imaging was chosen to be carried out in tapping mode.

b) Local plasma generation: the formation of localized plasmas was tested as follows. Localized plasmas were formed by applying a 1000V AC voltage through the contact pads between the tip and a surrounding electrode patterned on the beam. The test was performed under atmospheric conditions. Figure 7 (a) shows the optical picture of the powered device under normal microscope illumination conditions. Figure 7 (b) shows the generated microplasma using the same device with the microscope illumination off. The generated localized plasma is a source of electrons, ions, and radicals that can be used for microscale and nanoscale fabrication.

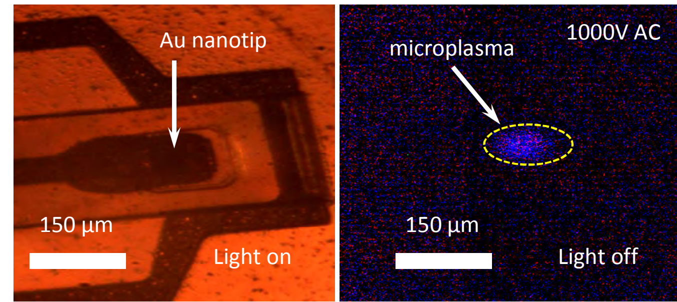

Figure 7. Optical pictures showing the generated microplasma at 1000V AC stimulus.

V. Summary

In this paper we present the microfabrication and initial testing of a tall-tip localized plasma nanoprobe. In this device, a microchannel and electrodes are integrated with a sharp tall CrAu tip molded and transferred from a KOH etched inverted pyramid mold. The process produces pyramidal protruding CrAu tips which are 25 μm-high with tip radius of about 20 nm. Initial tests indicate the probe can be used for tapping-type imaging and the formation of gas microplasmas localized near the tip, demonstrating the device's capability for localized etching, deposition and imaging.

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