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In-plane fabrication of insulated gold-tip probes for electrochemical and force spectroscopy molecular experiments

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ABSTRACT

A new and versatile fabrication process of insulated gold tip probes for atomic force microscopy (AFM) is presented by Wu et al. (In-plane fabricated insulated gold-tip probe for electrochemical and molecular experiments, in: 2013 IEEE 26th International Conference on Micro Electro Mechanical Systems (MEMS), IEEE, 2013, pp. 492–495). The novelty of the process lies in the fact that the length and the thickness of the cantilever are defined by photolithography and Si etching from the wafer top surface. Width of the cantilever is defined by the device layer of a silicon-on-insulator (SOI) wafer. The tip is fabricated in the wafer top plane. E-beam lithography was employed outlining the gold nanowire tip. The chip body is formed with the handling layer of the SOI by deep reactive ion etching in later steps. In a practical operation, the probe chip is rotated by 90 degree. The tip radius of curvature is approximately 20 nm. The high-quality insulation on the probe was demonstrated by performing electrodeposition of gold on the tip-end. The spring constant of the cantilever was obtained by measuring resonance frequency of the cantilever. With this in-plane fabrication process, probes with different spring constants ranging from 0.05 N/m to 13.67 N/m were fabricated on the same wafer.

1. Introduction

An interesting and challenging topic in molecular science is the simultaneous measurement and characterization of the electrical transport- and mechanical properties of single molecules. One of the most popular methods is the break junction method which uses atomic force microscopy (AFM) with a conductive measurement setup [2,3]. In this method, an AFM tip is firstly brought into contact with a molecule on the sample stage and, then, slowly retracted. By using a conductive tip AFM probe, the mechanical and electrical properties of molecule junctions can be measured simultaneously.

The conductive material of the probe tip requires special consideration with respect to binding of the molecules under investigation and to provide low resistivity for electrical measurement at the same time. Several publications on conductive probes using different kinds of metals have been reported [4–7]. Gold is the most often used metal as it allows molecules with a thiol group to bind to its surface. However, unlike platinum-based materials, gold is relatively soft and not compatible with many high temperature processes, which augments the difficulties of the fabrication process.

In order to perform experiment in electrolytic solutions, the conducting tip needs a high-quality insulation on all the conductive parts except the tip apex. This limits parasitic current flow and enhances the difference between tunneling- and Faradaic-currents [8]. For this purpose, modifications of commercial conducting AFM probes have been reported in the literature [7,9,10]. One of the insulated conductive probes was developed by modifying a commercial AFM cantilever by successively coating with a gold layer, and a silicon nitride layer, the tip apex was afterwards opened by focused ion beam (FIB) [7]. Another manually processed probe used photoresist film as the insulation layer and the electro-active area at the tip-apex was opened by using a custom-made photolithography system [10]. A disadvantage of the latter approach is that the photoresist is not electrochemically durable.

Another important parameter of a practical probe for molecular experiments is the spring constant of the cantilever beam. Depending on the application, different spring constants are required. For example, a stiff cantilever (~5 N/m) is required for force-conductance correlation researches in order to measure a force between gold and gold contacts [3]. Whereas a much softer cantilever (~0.1 N/m) is required for gold–molecule–gold connection measurements [10]. The cantilever stiffness also determines the magnitude of the experimentally measurable deflection signal of the laser, and consequently the signal-to-noise ratio in the force measurement. Thus cantilevers with a wide range of spring constants are essential required for various measurements.

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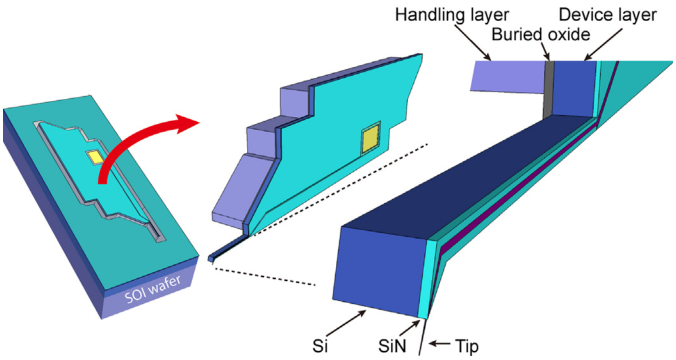

Fig. 1. Schematic view of the unique approach to fabricate an in-plane scanning probe. The cantilever beam length and thickness are defined on the silicon plane.

Changing the thickness and length of the cantilever are the most common method to design different cantilevers with various spring constants.

In this paper, we introduce a versatile batch fabrication process to realize probes with gold tip [1]. The electrode on the probe is totally covered with a high-quality silicon nitride film except for the tip apex. This process allows us to obtain different cantilevers with different thickness and length, resulting in a wide range of spring constants, from a single wafer. The feasibility of the process was demonstrated and the high-quality of the insulation is verified by electrochemical characterization of probes intended for applications in molecular technology researches.

2. In-plane fabrication process

The concept of our "in-plane" probe fabrication is illustrated in Fig. 1. In this approach, the cantilever length and thickness are defined by photolithography and etching. The thickness of the SOI device layer corresponds to the width of the cantilever. After shaping of the probe using dry-etching techniques and liberation with BHF, the probe is rotated 90 degrees and mounted on an AFM. The conductive metal tip and the passivation are realized on the top surface of the wafer, which becomes the side of the cantilever in the final structure. The advantage of this approach is that mechanical and electrical properties, as well as the conducting metal of the probe, can be individually defined, thus it is possible to design a wide range of cantilevers with different characteristics on one wafer.

The fabrication process flow is shown in Fig. 2, starting with a SOI wafer (20 μm device layer) with a silicon nitride (PECVD, 350 °C, 300 nm) passivation layer. The pattern of gold nano-wires is defined by electron beam lithography on a 200 nm thick PMMA layer. Afterwards, a thin gold layer (5 nm Ta and 40 nm Au) is deposited by using electron beam evaporation onto the surface and patterned by using a lift-off process. After removing the PMMA layer, AZ 1518 (1.8 μm) was deposited on the surface. Interconnections with a small overlap on the gold nano-wire, was patterned by photolithography. Layers of 5 nm Ta and 80 nm Pt were deposited onto the surface using electron beam evaporation and patterned by using lift-off process as well. This process combining electron-beam lithography (EBL) and photolithography to make wafer-level fabrication is called "Mix and Match" process [12]. In this way, EBL is employed for the small and critical areas, which require precise and well-controlled exposure of the gold tips. Traditional photolithography is used to make periphery structures such as interconnection leads and contact pads (Fig. 2(a)). This mix and match technique provides an optimum trade off between precision and time budget. After the formation of the gold nanowire and platinum leads, another silicon nitride passivation layer (PECVD, 350 °C, 300 nm) is deposited on the entire top surface (Fig. 2(b)), the contact holes are patterned by photolithography and opened in BHF. Afterward, a 50 nm Cr mask is structured by the "Mix and Match" process (Fig. 2(c2)) as well: small areas to precisely protect the nano-wire structures are formed by EBL and the first lift-off, relatively large areas are defined by photolithography and the second lift-off. With this patterned Cr layer as an etch mask, the cantilever and the chip body are formed by deep reactive etching (DRIE) (Fig. 2(c1)). The Cr mask is then removed in a Cr etchant. The chip body is defined from the backside by photolithography and DRIE (Fig. 2(d)). Protected by photoresist (AZ 1518, 1.8 μm) on the front-side, the cantilevers are released from the backside by BHF (Fig. 2(e)). Finally, the probe chips are further etched in KOH for a short period of time followed by a brief BHF etching in order to control the exposed length of the gold nano-wire tip (Fig. 2(g)).

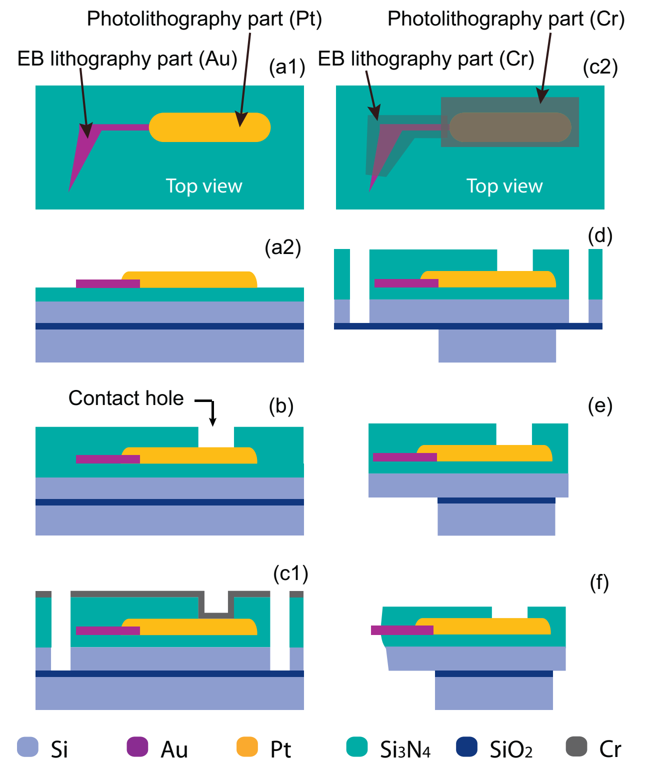

Fig. 2. Schematic view of the fabrication process. Top view (a1) and side view (a2) of the tip and interconnections after the lift-off process. (b) BHF etching to open the contact holes. (c1) DRIE etching through the device layer with the Cr mask which defines the chip shape. (c2) Top view of the Cr mask in step c1. (d) Backside patterned by DRIE. (e) BHF etching releases the chip. (f) KOH etching and BHF etching open the conducting tip apex.

For practical operations, a copper wire is glued onto the contact pad of the probe using conductive epoxy (epo-tek H20E). The contact point is covered with a non-conductive epoxy resin for isolation.

3. Characterizations

Fig. 3a–c shows SEM images of the planar probe with an insulated gold nanowire. The gold nanowire and the electrode connections are located on the side of the cantilever. The metal lines are covered by silicon nitride except at the very end. The radius of curvature of the gold-tip apex is approximately 20 nm. This will result in an active conductive area of around 400 nm^2 after the removal of the overhanging gold material. Part (d) shows a view of the probe from the handling layer of the original wafer. In the image, three