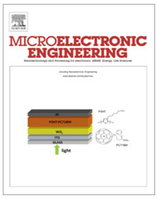
Microelectronic Engineering 119 (2014) 44–47

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee



Batch fabrication of insulated conductive scanning probe microscopy probes with reduced capacitive coupling

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ARTICLE INFO

Article history:
Available online 31 January 2014

Keywords:
Conductive scanning probe microscopy (C-SPM)
SIM
SECM
EFM
SECM-AFM

ABSTRACT

We report a novel fabrication process for the batch fabrication of insulated conductive scanning probe microscopy (SPM) probes for electrical and topographic characterization of soft samples in liquid media at the nanoscale. The whole SPM probe structure is insulated with a dielectric material except at the very tip end and at the contact pad area to minimize the leakage current in liquid. Additionally, the geometry of the conducting layer in the probe cantilever and substrate is engineered to reduce the parasitic capacitance coupling with the sample. The electrical characterization of the probes has shown that parasitic capacitances are significantly reduced as compared to fully metallized cantilevers.

1. Introduction

Since the invention of the atomic force microscope (AFM) by Binnig et al. [1], various techniques based on scanning probe microscopy (SPM) have evolved as fundamental tools in biology and nanobiotechnology [2]. Electrical SPM characterization techniques such as scanning electrochemical microscopy (SECM) [3], scanning impedance microscopy (SIM) [4] and electrostatic force microscopy (EFM) [5] should allow the study of the electrical behavior of biomolecules at the nanoscale in its native physiological environment.

Considering the small signal associated with biological samples, the SPM based electrical characterization techniques for biology should minimize the parasitic conductance and capacitance between the sample and the extended probe cantilever structure. The parasitic conductance could be minimized by insulating the whole probe with a dielectric material except at the very tip where electrical contact with the sample will be made and at the contact pad. In addition, the capacitance coupling between the sample and the probe cantilever could be minimized by engineering the width of the conductor layer in the probe cantilever. Some groups have fabricated conducting insulated probes for SECM-AFM [3,7–9], but still no one with a reduced capacitive coupling.

With the objective of characterizing olfactory receptor proteins for the development of olfactory biosensors [6], we have developed insulated conductive SPM (C-SPM) probes for SIM characterization in liquid environment. Here we report the fabrication of probes for both DC and AC applications. These probes minimize both the leakage current in liquids and the parasitic capacitance coupling associated with the extended cantilever. We also report the characterization results of the AC probes in air. We will report the corresponding results for the DC probes elsewhere.

2. Fabrication and design

We made separate designs for DC probes (for DC electrical measurements in contact mode) and AC probes (to measure the AC response in dynamic mode). The cantilever dimensions for the DC probes range from 300 to 400 μm in length and 30–40 μm in width, with a thickness of 2 μm. These values have been defined to get probes with force constant values lower than 1 N/m, as soft probes are required to study soft biological samples in contact mode. As relatively stiff probe cantilevers are required for dynamic mode applications, the cantilever dimensions considered for the AC probes range from 250 to 275 μm in length and 30–40 μm in width, with a thickness of 4 μm to get force constant values between 5 and 20 N/m. Besides the restriction on the force constant of the probes, there is also material requirement for the tip. As the measurement is made in a biological sample in its native physiological environment, the tip material for the probes should be biocompatible and electrochemically stable. And the tip material for the DC probes should also be wear resistant as the measurement is made in contact mode. To this end we fabricated

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http://dx.doi.org/10.1016/j.mee.2014.01.018
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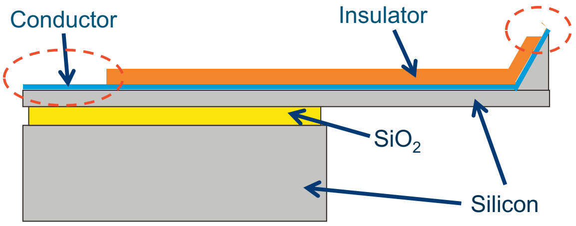

Fig. 1. Schematic structure of the fabricated probes. Two types of probes with either a conducting thin film or a doped silicon layer have been developed.

the insulated conductive DC probes with a conducting layer realized with a high-dose boron ion implantation plus annealing. As wear is not a problem for dynamic mode applications, the conducting layer in the AC probes has been realized by depositing a Cr plus Au thin film using physical vapor deposition and patterning using a lift-off process. Fig. 1 is a schematic view of the insulated C-SPM probes.

The probes have been fabricated in silicon-on-insulator (SOI) substrates as cantilevers with sharp tetrahedral tips at their free end. Fig. 2 shows schematically the fabrication process sequence. The starting material is a (100) oriented n-type SOI wafer with 15 μm device layer, 1 μm buried oxide (BOX) layer and 500 μm thick bulk silicon. First we fabricate the tetrahedral tips by opening a hole in the Si device layer with deep reactive ion etching (DRIE) using the BOX as an etch stop. The top surface is covered by a thin thermal oxide, 100 nm of silicon nitride plus 100 nm thick aluminum (Fig. 2a). This defines the two vertical planes of the tetrahedral tips. The aluminum layer is removed in wet etching and a 400 nm thick thermal oxide is grown on the exposed silicon walls using the silicon nitride layer as a mask. The silicon nitride and the thin thermal oxide are removed using wet etching and the wafers are etched in a 25% tetramethylammonium hydroxide (TMAH) solution at 80 °C with time control to define the third (111) plane of the tetrahedral tips and the thickness of the cantilevers. The structures of the cantilever and chip are patterned using 24 μm thick photoresist and DRIE (Fig. 2b). Then the 400 nm thermal oxide is removed from the vertical side walls of the tetrahedral tips with wet etching and a conducting layer is added to the probes (Fig. 2c). For the AC probes 30 nm Cr and 100 nm Au are deposited using physical vapor deposition and patterned with a lift-off process. For the DC probes the conducting layer is made with a high-dose boron ion implantation plus annealing. Later a 400 nm thick silicon nitride layer is deposited by plasma-enhanced chemical vapor deposition (PECVD) and patterned so that the whole probe is insulated except at the tips and electrical contact pads (Fig. 2d). The patterning is defined by a standard lithography process, which results in an opening at the tip of a typical size of 2–4 μm. Aluminum is added in the DC probes to contact the implanted layer in the pad area. The substrate is etched from the backside by DRIE using an Al mask and the BOX layer as an etch stop [10]. Finally the cantilevers are released by 49% HF vapor etching of the BOX. Fig. 3 shows SEM micrographs of fabricated AC probes at different stages of the fabrication process.

3. Characterization results

The fabricated AC probes were tested by mounting them in commercial AFM instruments to extract their mechanical and electrical properties in air. The resonance frequency and force constant of the AC probes were in the range of 65–120 kHz and 3–20 N/m (calculated by using the Sader's method proposed in [11]), respectively. The AC probes were first tested by measuring the topography of different samples such as carbon nanotubes, interdigitated metal electrodes, and silicon dioxide nano-structures on highly doped silicon. Fig. 4 shows the topography of interdigitated metal electrodes (IDEs) with 150 nm pitch and 100 nm height (left), and the topographic (center) image of a 20 nm thick-silicon oxide nanostructure. From the topography images, the tip radius of the probes was estimated to be in the range of 30–50 nm. An electrostatic force microscopy (EFM) image of the silicon–silicon oxide structure is shown in Fig. 4 (right); the image was acquired in non-contact mode at constant height (45 nm) from the highly doped silicon substrate by applying a DC voltage of 9 V

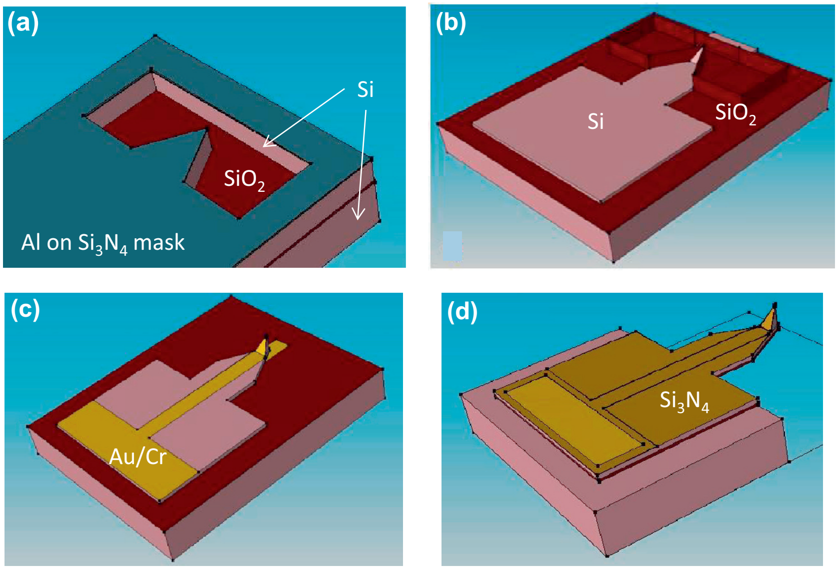

Fig. 2. Schematic diagram for the fabrication of insulated conductive SPM probes. See text for details. (a) A hole is opened in the Si top layer of a SOI wafer by DRIE, stopping at the buried oxide (BOX). (b) The exposed silicon walls are oxidized, the top Si surface is unprotected and anisotropically wet etched to define the third plane of the tetrahedral tip. The cantilever and chip structures are patterned on the top Si. (c) The oxide walls are removed and a metal layer is deposited and patterned. (d) An insulating silicon nitride layer is deposited and opened at the tip and the contact pad. The Si substrate is etched from the backside by DRIE, and the remaining BOX membrane is etched to release the cantilever.