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MEMS Based Nanotools for Detection and Manipulation at the Molecular Level

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1. Introduction

Addressing nanometer scale objects requires probes of comparable dimensions. Classically, these were far-field probes like focused electron or ion beams which have diffraction-limited resolution. The scanning proximity probe microscopes (SXM) like the scanning tunneling [1] or scanning force microscope (AFM)[2], on the other hand, owe their high, atomic resolution to a mechanically constricted interaction defined by a sharply pointed probing tip. Because of their resolution, SXM became an important class of instruments or tools for detecting, analyzing, and manipulation nano-objects down to the molecular and atomic scale. The nanotools treated in here are of the SXM-type. They all feature a sensor-spring for measuring the force acting between the sample and the probe. This signal is used to retrieve either the topography information during a raster scan or to control the vertical placement of the probe above the sample or substrate. In most cases, an additional device is integrated in the probing tip. This allows for simultaneous and local measurements characteristics of a sample as function of its surface topography. Similarly a substrate can also be modified at well-define positions.

Nanotools do not need to be entirely of nanometer dimensions themselves but should have also micro- to millimeter size components. By that, they can act as interfaces between the nano- and macro-world. The ideal range of size of nanotools is therefore in the domain of micro fabrication techniques.

2. Microfabrication

Nanometer size "tool-tips" or other components can be fabricated by using standard optical lithography and related micro fabrication techniques combined with special processing steps. The basic idea of these special processing steps is to use material modifications like under-etching, which are self-limiting at the nanometer scale.

Fig. 1 shows the basic process flow for fabricating plain Si_3N_4-cantilevers with a molded tip [3]. First, single crystal Si <100> wafers are etched in KOH to form a pyramidal etch-pit. The sidewalls of this pit are defined by the {111} plains of Si. Theoretically these plains intersect in a single point if the etch mask is either a perfect rectangle or circle. In reality, a knife-edge of a few 10 nm length is often formed. A thermal wet-oxidation step at about 950 °C can be applied to narrow the pit at the bottom, reducing the problem of the knife-edge to some extent [4]. A homogeneous layer of Si_3N_4 is deposited by Low Chemical Vapor Deposition (LPCVD) and the shape of the cantilever is defined by lithography and Reactive Ion Etching (RIE). Finally, a support of e.g. glass is bonded to the Si_3N_4 layer and the Si is etched away, leaving the AFM probe ready to be used. Other basic processes to form tips are based on under-etching a mask. Different materials can be used like fused silica or single crystal silicon. In the latter case the etching can be isotropic, e.g. by RIE or anisotropic etching such as by KOH. The thickness of the cantilever is defined by etching a membrane from the backside. The shape of the cantilevers is lithographically defined and etched from the front side.

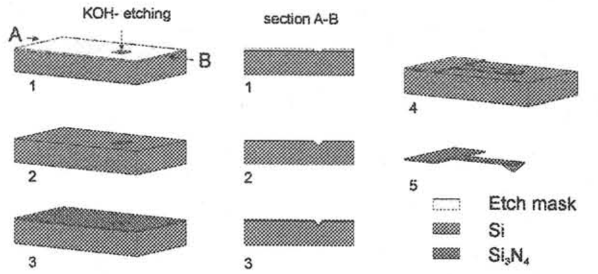

Fig. 1 Process flowchart for fabricating Si_3N_4 canti-levers. The nitride is deposited in a etch pit and the Si is completely etched away. Bonding of a support is not shown here.

3. Examples and Experiments

3.1 Special Tips

For several applications it is desirable to have an electrically conductive tip with insulation up to the apex. For that purpose we have coated sharp, anisotropically etched silicon tips with a low-stress Si_3N_4 layer. The Si is selectively etched from the backside, leaving the empty nitride coat which can than be used as a mold for metal filling [5]. Attacking the nitride on the front-side of the wafer by RIE will open the nitride at the tip apex first due to electrical field-enhancement like on a lightening-rod. Fig. 2a shows a SEM image of such a tip. If prior to the RIE, a second metal layer is evaporated to the front-side, a coaxial tip can be fabricated (Fig.2b). These probes could be used e.g. in an electrolytic thin-layer STM for studying the under-potential deposition of Pb on Ag.

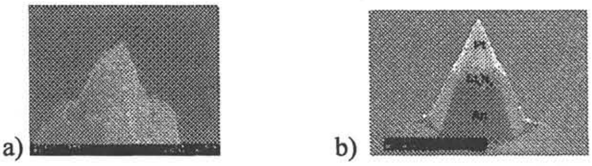

Fig 2 *Isolated Pt-tip (a) with Si_xN_y insulation. A second, coaxial Ag-electrode can be added. (b) shows the implementation of such a second electrode on another tip than shown in (a).*

3.2 Scanning Micropipette

The above-mentioned technique to fabricate a tip shaped Si_3N_4-coat can be extended to include the mold on a cantilever. We left the mold empty and etched the apex again by RIE, which resulted in a micropipette-tip (Fig.3) on a compound Si-Si_3N_4 cantilever.

Fig. 3 *A hollow Si_xN_y tip is mounted on a Si - Si_xN_y - cantilever. (a) shows the cantilever from the backside. The opening in the Si can easily be identified. (b) Presents the opening of such a pipette-tip, also locking from the backside. (c) Is a view from the front side.*

3.3 Scanning Force Microscopy

The standard technique for making Si_3N_4 cantilevers as described in section 2 can be extended to include metallic tips [3]. Leaving some of the insulating layer at the tip-base provides again an insulated tip with an open, metallic apex. This will enable simultaneous measurements of the topography and of local electrical currents or potentials even in aqueous solutions like a physiological buffer. Fig. 4 shows an AFM image of bacteria rhodopsin (purple membrane) recorded with such a tip in a buffer solution. This image was recorded for testing the "AFM–quality" of the metal tip, no electrical information was acquired so far. A sufficient resolution below 1 nm for imaging biological membranes was obviously achieved.

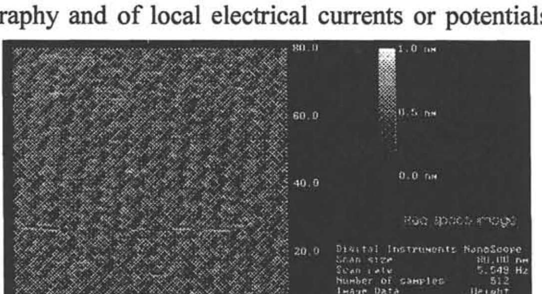

Fig. 4 *AFM measurement with a metal tip in buffer solution of bacteria rhodopsin. The intra-cellular side is shown. (Image courtesy of P. Frederix et al, Univ. of Basel)*

3.4 Scanning Near-Field Optical Microscopy

In scanning near-field optical microscopy (SNOM) a sub-half-wavelength size source or detector is employed to probe the sample. This probe has to be positioned and scanned within its near-field range above the sample surface. An AFM sensor with a transparent tip enables this in an effective way. We have therefore developed such a sensor comprising a silicon cantilever, a fused silica tip and an aluminum coating [6]. With these tips, single, fluorescent-labeled molecules could be detected with a resolution of 32 nm (Fig. 5). The sample was made of diluted (10^{-6} M) goat anti-rat antigens labeled with Alexa Fluor 488 and spin coated on a glass cover slip. The image shows a scan in constant-height with a gap of about 10 nm between the tip and the surface [6].

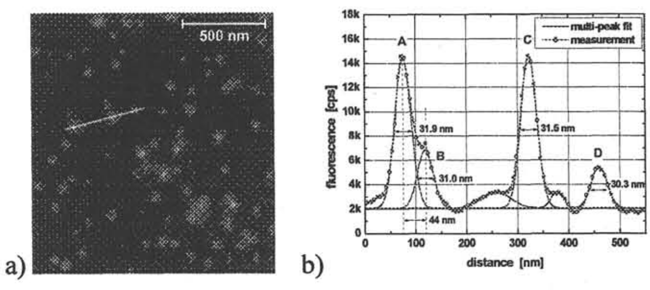

Fig. 5 (a) *SNOM image of fluorescent-labeled single molecules. The intensity cross-section along the white line shown in **b)** demonstrates a resolution of about 32 nm [6].*

4. Summary and conclusion

We have built several multi-functional probes based on AFM cantilevers. By using self-limiting processes and effects that occur on highly curved surfaces, nanometer scale size features can be fabricated at the apex. The interaction with the sample proves to be localized enough to generate images at the molecular level.

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