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# Fabrication of submicron- or nano-sized mesa electrodes via AFM oxidation: Applications to metal ion detection

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## Abstract

Conductive probe AFM local oxidation is a promising lithographic technique for use in fabricating submicron- or nano-scale structures. In this study, a metal ion detector with a submicron size electrode was fabricated by AFM lithography using a pre-programmed voltage and a non-etching method. The square frame of the mesa pattern was functionalized by APTES for the metal ion detection, and the remaining portion was used as an electrode by the self-assembly of MPTMS for Au metal deposition. In this module, no metal lining or lead line was required, because the conductive tip (mobile electrode) was in direct contact with the gold-deposited mesa portion (fixed electrode). The conductance changed with the quantity of adsorbed copper ions, due to electron tunneling between the mobile and surface electrodes. © 2005 Elsevier B.V. All rights reserved.

**Keywords:** AFM; Etching; Lithography; Metal ion detection; Patterning

## 1. Introduction

New methods for fabricating submicron- or nanometer-size structures are crucial for the production of advanced sensors and devices in the future. Scanning-probe-based lithography (SPL) has the potential for producing small features (<100 nm) with high spatial precision, and can be

classified into three methods; nano-scratching [1], dip-pen [2], and anodic oxidation [3]. In nano-scratching, patterns are directly scratched on the substrate with a tip, and, as a result, the tip is easily abraded. In the dip-pen method, the fabrication rate is slow (1–10 nm/s) compared to AFM anodic oxidation (0.1–10 μm/s). Conductive probe AFM local oxidation of the semiconductor and metallic surface is the most versatile and desirable lithographic technique, among the available SPL lithographic techniques for creating submicron- or

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nano-scale structures for applications to nano-electronics.

Functionalized nano-particles and self-assembled organic films can be used as sensor elements. The change in resistance or conductance of the modified surface, upon exposure to target materials, is frequently the basis for the sensing action. To date, a number of methods have been proposed for the detection of metal ions, including: (i) point contact between the two fixed electrodes, which triggers a quantum jump in conductance [4]; (ii) fluorescence detection on the surface functionalized silica particles [5]; (iii) a sensor with a thin-film polymer that changes size upon the reversible binding of analyte ions [6]. Metal ion detection via a point contact is based on electron tunneling or a current jump between two separated electrodes.

Herein, we describe a simple 2D patterning method using AFM oxidation with voltage programming based on the pixel units, which is referred to as pre-controlled automation. Based on this method, a new method for detecting metal ions is described, based on current transmission of the separated compartments between fixed and mobile electrodes via the adsorption of metal ions.

## 2. Experimental

### 2.1. Pre-controlled automation of AFM oxidation

The experiments were performed in a humidity controlled box equipped with an AFM (METRIS-2000, Burleigh Instrument, USA) and an additional power supply (potentiometer). The fabricated patterns were also analyzed with the Dimension 3100 SPM instrument (Veeco, USA). To investigate the growth of  $\text{SiO}_2$  on Si(100), a  $\text{Si}_3\text{N}_4$  resist, deposited on a boron-doped p-Si(100) wafer ( $\rho = 10\text{--}15 \Omega \text{ cm}$ ) by LPCVD, was used as the substrate. The  $\text{Si}_3\text{N}_4$  thickness (12 nm) was determined by ellipsometry measurements (L116B, Gaertner, USA). A silicon cantilever coated with Ti–Pt (CSC21, MikroMasch, Estonia) was used. The force constant ( $k_c$ ) and resonance frequency ( $f_0$ ) were 0.21 N/m and 12 kHz, respectively.

As shown in Fig. 1, a pre-programmed voltage signal is applied to the conductive tip during AFM scanning, and complicated patterns, corresponding to the input signal, can then be easily produced. In the mapping step, the voltage-map is adjustable to several pixel units (i.e.,  $N$  or

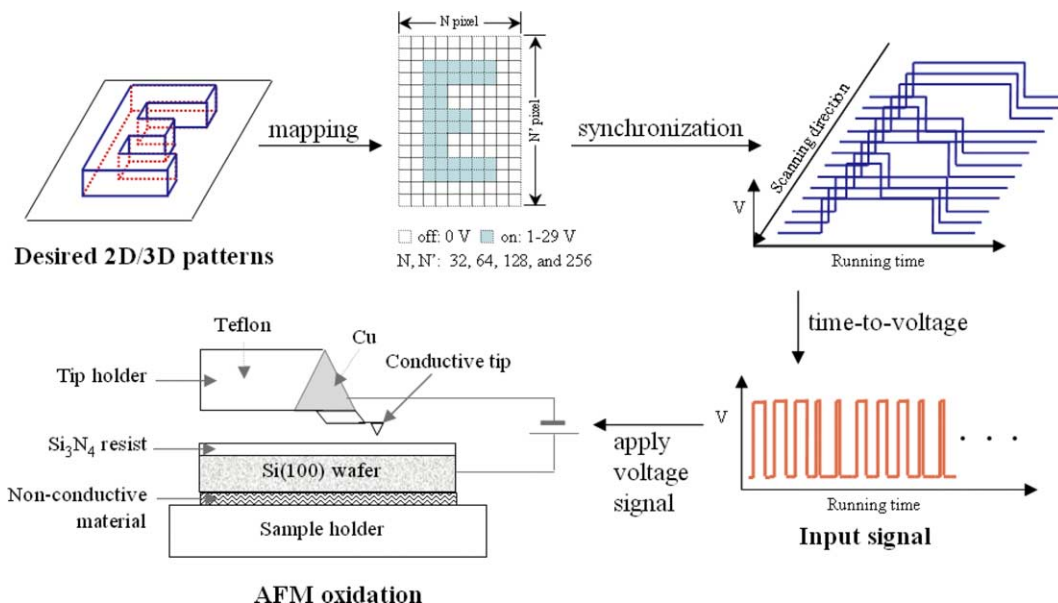


Fig. 1. Schematic diagram of AFM oxidation with voltage programming based on pixel units.

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