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Resistless patterning of quantum nanostructures by local anodization with an atomic force microscope

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Abstract

We report here two processes for fabricating quantum devices based on local anodization induced by Atomic Force Microscope (AFM). The first process involves ultra-thin films of doped silicon-on-insulator (SOI) passivated with hydrogen. AFM-drawn oxide lines create a chemical contrast that is used as a mask for silicon wet etch. Etching is performed down to the buried silicon oxide layer, thus leading to silicon nanowires supported on insulator. We show that this process appears well suited to obtain SOI nanostructures and investigate electrical transport though silicon wires with sub-1000 nm^2 cross-section. In the second process, diffusion of oxygen species is performed through the whole layer of metallic ultra-thin films, which provide a technique for direct writing of insulating regions. This process was applied to ultra-thin niobium films for fabricating mesoscopic structures. A superconducting quantum interferometer useful for nanomagnetism is demonstrated. © 2002 Published by Elsevier Science B.V.

Keywords: Atomic force microscopy; Lithography; Silicon-on-insulator; Nanostructures; Quantum devices

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1. Introduction

Scanning probe microscopes are proven to be valuable tools to achieve atomic and molecular manipulation and patterning at the nanometer scale. Indeed, various scanning probe lithography (SPL) techniques involving the entire SPM family have been developed, taking advantage of both near field

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interaction of such microscopes and sub-angström resolution of tip positioning. These lithography techniques were used to pattern nanoscale structures with well-defined geometry, some of them even reaching the atomic scale [1]. Except results based on the mechanical interaction between an AFM tip and a soft substrate (mechanical indentation of polymer resists [2,3] and of self assembled monolayers [4] or photonic emission from near field optical microscopes [5], most of these SPL techniques take advantage of the spatial resolution of the low-energy electron emission from the microscope tip to perform local physical or chemical alterations on a suitably prepared surface. Following this principle, a wide range of different techniques have been reported: chemical vapor deposition under an STM tip [5–7], local exposure of polymer resists [8], and local oxidation of silicon [9] and of thin metallic films (Al, Ti) [10,11] have been reported.

These last two techniques are entirely compatible with today's microelectronic technology. Furthermore, their near field operation makes them resistant with respect to proximity effect [12,13]. Technological breakthroughs of high-speed parallel AFM lithography [14] have risen hopes that the intrinsically low throughput of SPL can be enhanced so that industrial applications can be envisioned. On the other hand, these techniques appear as the only ones to investigate direct fabrication of quantum devices such as quantum point contacts [10,11], nanowires [15] and single electron devices [10,11] with in-situ control during fabrication. Following these recent advances, we have chosen two methods which both involve SPM-induced oxido-reduction. We have applied them to the fabrication of quantum devices. For that purpose, we have focused on a specific process, pioneered by Dagata et al. [9] that is fully compatible with today's microelectronic technology. It is based on the local oxidation of ultra-thin films induced under the voltage-biased tip of an AFM. Such resistless lithography technique is among the sharpest resolution that could reach 7 nm linewidth [16]. In order to test their real efficiency for general-purpose nanofabrication, we have applied this lithography technique using different types of materials. We have chosen Silicon-On-Insulator (SOI) and ultra-thin metallic layers for their respective semiconducting or superconducting properties in order to implement active functions.

2. Principles of the technique

These anodization techniques have lead in recent years to a wide range of applications, including the first clear experimental demonstration of a single electron transistor operating at room temperature [10,11]. The principle summarizes as follows: diffusion of oxygen is performed very locally under the negatively biased tip into the thin film, which acts as an anode. The electrochemical process occurs in ambient air in the presence of a water contamination layer. Oxide diffusion in the layer is limited by charge space [17].

3. Fabrication of silicon nanostructures

This process was applied to a *n*-doped 15 nm-thick silicon-on-insulator film (SOI) prepared using Unibond® process [18]. Native oxide was first removed by a HF dip leaving hydrogen passivated metastable surface. Oxide lines of width ranging between 15 and 60 nm are then drawn with the biased AFM tip. Such lines create a chemical contrast with respect to the non-patterned Si:H that is

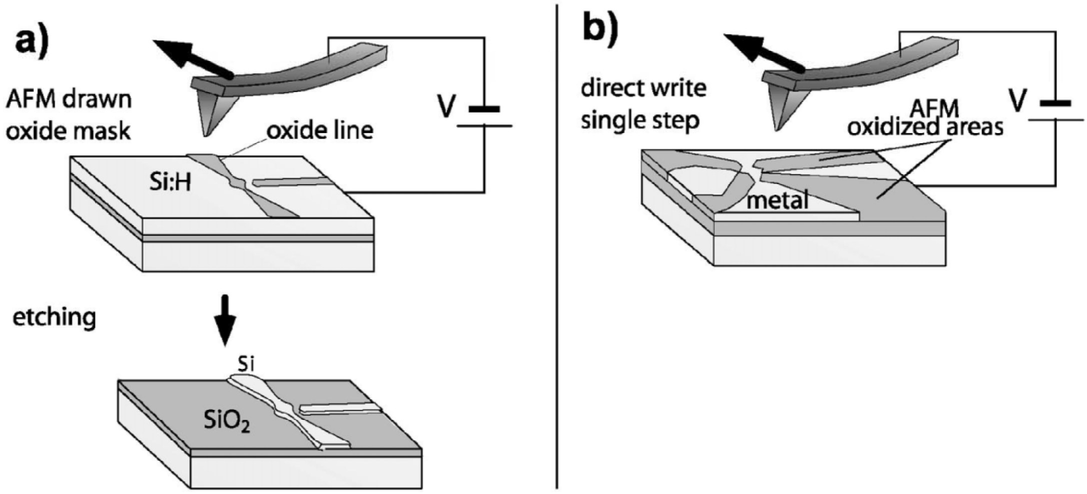

Fig. 1. Schematics of the two different fabrication processes based on AFM induced anodization. Left: masking technique, the oxide pattern behaves like a mask for a subsequent wet etch of the non-patterned silicon film, thus leaving silicon nanostructures supported on oxide. Right: AFM oxidation through the whole metallic layer leads to a direct-write technique of insulating regions.

used as an etching mask in a subsequent silicon wet etch (see Fig. 1, left), which removes all the non-protected silicon areas. This leads to supported single-crystal silicon nanowires. A lateral finger drawn in this way and approaching the nanowire acts as a lateral gate [19]. Fig. 2 is a AFM micrograph of such a fully-processed device. A nanowire with 70×15 nm^2 cross section is obtained. The nanowire-gate gap can be reduced to 30 nm without significant leakage. At room temperature,

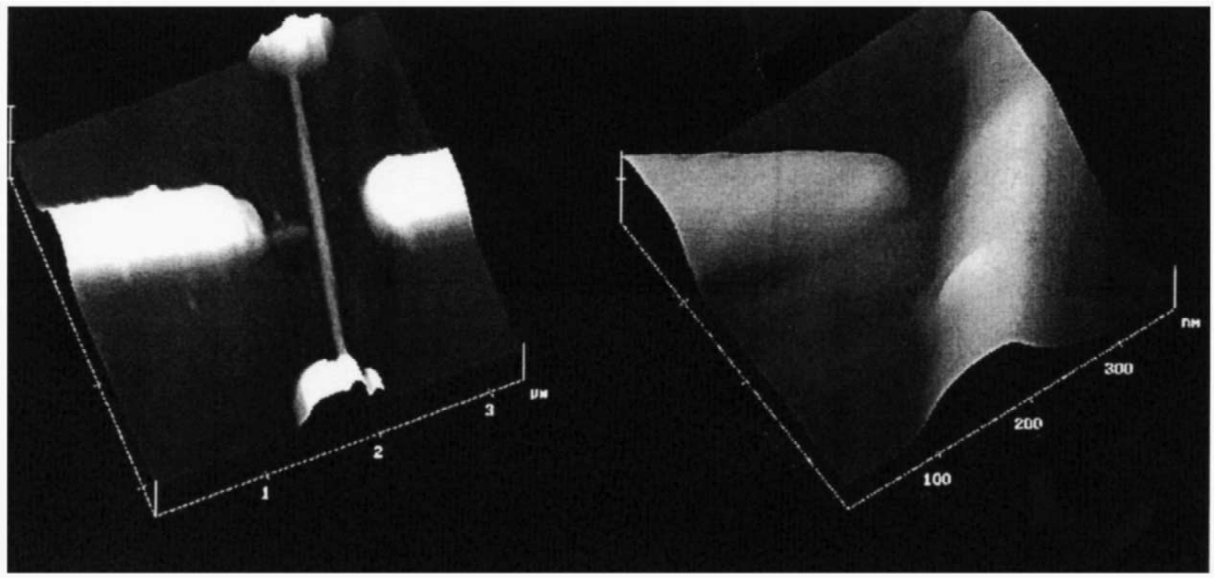

Fig. 2. AFM micrograph of a Si nanowire with a lateral gate made by AFM anodization. Right: zoom of the active region.

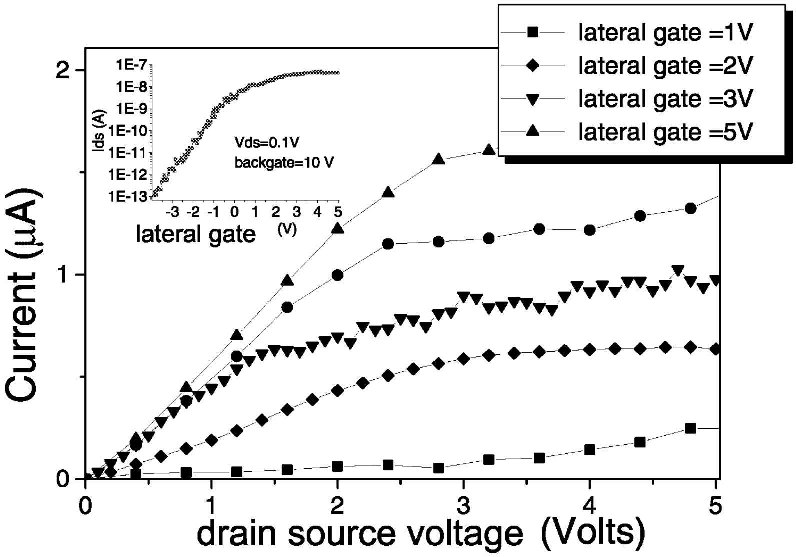

Fig. 3. I–V curve of the device presented in Fig. 2, for different lateral gate voltages Vg. Inset: I–Vg curve showing the channel depletion for negative voltages.

such nanostructures typically behave like field effect transistors (see Fig. 3). A current density up to 10^6 A/cm^2 can be completely switched off by a few volts applied on the lateral gate (see Fig. 3, inset). Low temperature measurements shows that the transport in these structures is strongly non-linear and is dominated by Coulomb blockade effect [20]. This process is a versatile and flexible and appear well suited to explore silicon nanoelectronics.

4. Nanoscale superconducting quantum interference devices

In a second type of anodisation process, diffusion of oxygen species is performed through the whole film layer. Therefore, it allows direct writing of insulating regions (see Fig. 1, right). This general method has been successfully experimented using many materials such as Al [10,11], Nb [10,11], Cr, TiN, Nb. Nanostructures can then be fabricated in a single step, on metal strip lines that have been previously patterned using conventional UV lithography. Moreover, by carefully adjusting the tip bias, the oxide line width can be adjusted to thickness to provide either electrical separation or a tunnel barrier with controlled transparency [21].

In our group, following previous experiments [22], we have focused on Niobium ultra-thin films (<10 nm thick), which are evaporated in UHV conditions on annealed sapphire substrates. Such preparation ensures a high quality epitaxial Niobium film.

We have implemented weak-links by a partial lateral anodization of the film thickness. These weak links behaves like Josephson junctions [23]. An insulating loop can be obtained by scanning the biased AFM tip in parallel lines (see Fig. 4, left). The resulting device is a superconducting quantum interference devices (SQUID) [24,25]. The magnetic field modulation of the maximum Josephson supercurrent (Fig. 4) presents a signature typical of a Josephson junction which length is of the same

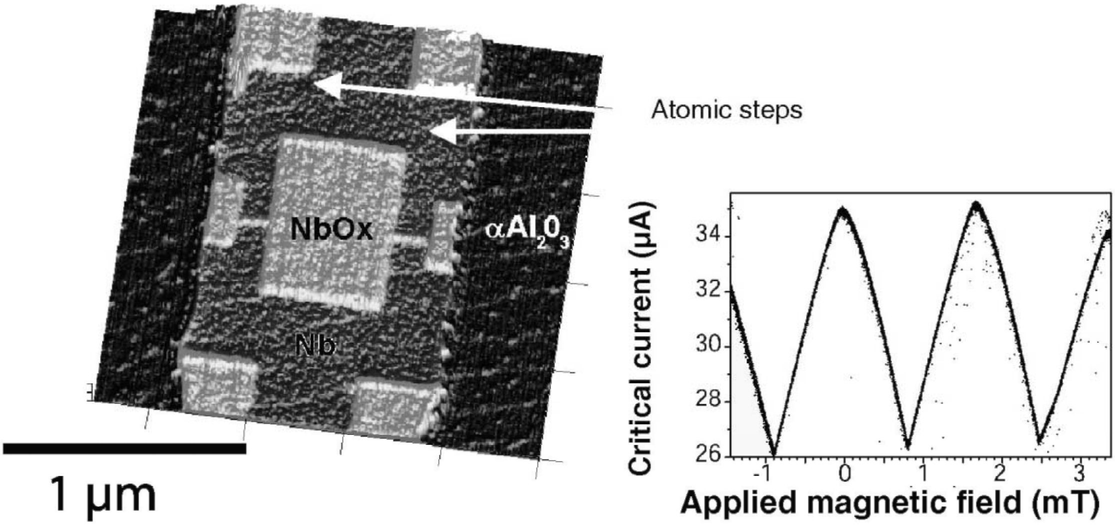

Fig. 4. Left: AFM micrograph of a superconducting quantum interference device made by local oxidation on 6-nm-thick niobium film. Right: Magnetic field dependence of the critical current of the AFM-made SQUID.

order of the superconducting coherence length (that is of the order of 10 nm in these films) [24]. Applications of these novel devices to the magnetometry of nanometer scale particles [26] are now envisioned since it allows patterning with a precise alignment with respect to the probed particle.

In conclusion, we have shown that AFM-induced local anodization is a versatile and reliable technology leads to a positive and a negative lithography process suitable to fabricate metallic and silicon nanodevices. Improvement in the resolution should rapidly lead to fully processed devices of size below 10 nm.

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