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Nanolithography on SrRuO₃ thin film surfaces by scanning tunneling microscopy

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1. Introduction

The feasibility of manipulating the material surface at nanometer length scale, on demand, by scanning probes was first demonstrated by Becker et al. [1] in 1987. Since then large effort has been devoted to this rapidly developing area, later named scanning probe lithography (SPL) [2–8]. Currently, SPL-based technology has attracted much attention in creating nanostructures on a variety of materials, while conventional lithography methods are approaching fundamental size limits [9]. Particularly in modern miniaturization of microelectronic, SPL has offered promising possibilities in the fabrication of nanodevices and development of engineered templates for growth of epitaxial thin films.

The surfaces of many materials such as silicon [4,6], graphite [10,11], and $YBa_2Cu_3O_7$ [12–14] have been modified by SPL. The creation of nanostructures was found to be strongly influenced by bias voltage, tunneling current, scan speed, and ambient conditions. The lithography mechanism varies depending on the experimental conditions and is still under debate. Among others, field-induced evaporation [15] is widely accepted as a critical, or in some cases dominant, scheme in lithographic experiments [16–18]. That is, when the tip-sample proximity decreases to several angstroms, the applied electrical field becomes strong enough to break up the atomic bonding and atoms are ionized and evaporate

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ABSTRACT

Nanolithography on SrRuO₃ (SRO) thin film surfaces has been performed by a scanning tunneling microscope under ambient conditions. The depth of etched lines increases with increasing bias voltage but it does not change significantly by increasing the tunneling current. The dependence of line-width on bias voltage from experimental data is in agreement with theoretical calculation based on field-induced evaporation mechanism. Moreover, a three-square nanostructure was successfully created, showing the capability of fabricating nanodevices in SRO thin films.

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away from the sample or tip surfaces. The field-induced evaporation is a thermally activated process and the rate of evaporation is given by $\kappa = v \exp(-Q/kT)$, where Q is the activation energy and v the frequency factor (which is about 10^{13} s^{-1}). Field evaporation theory has successfully explained many phenomena discovered in SPL experiments, such as threshold bias voltages and reversibility of material transfer [4].

In the present work, we have performed scanning tunneling microscope (STM) lithography on SrRuO₃ (SRO) thin films surfaces under ambient conditions using self-fabricated iridium (Ir) tips. SRO is a conductive perovskite material, which has an orthorhombic structure with the space group Pbnm and lattice parameters a=5.5670 Å, b=5.5304 Å and c=7.8446 Å [19]. Epitaxial functional oxide thin film based on the SRO template could be crucial for the application of nanoelectronic devices.

2. Experimental methods

SRO (110) thin films (~50 nm thick) were epitaxially grown on (001)-oriented SrTiO₃ (STO) substrate by off-axis radio frequency magnetron sputtering. The films deposition was conducted in a mixed atmosphere of oxygen and argon (O₂:Ar=4:10) at a total pressure of 100 mTorr. The substrate temperature was controlled in a range between 750 and 850 °C. The electrical conductivity of the SRO thin films was measured to be 1.2×10^5 Sm⁻¹. STM measurements on the SRO surfaces revealed a step-and-terrace topography with terrace widths of 150–200 nm and step heights of one to two unit-cells. The





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Fig. 1. SEM image of an electrochemical etched Ir tip with an apex of $ROC \sim 50$ nm. The magnification is 100 000. The inset shows the overall shape of the Ir tip at magnification 2000.

root-mean-square roughness of the terraces surfaces was found to be ~1 Å. X-ray diffraction analysis indicated excellent crystalline quality, the full width at half maximum (FWHM) of the rocking curve around the (110) peak was determined to be less than 1°. STM tips were prepared from Ir wire with a diameter 0.25 mm by electrochemical method. Details of tip preparation will be documented elsewhere. Scanning electron microscope (SEM) characterization approved that the Ir tips with macroscopic radius of curvature (ROC)~50 nm were routinely produced. Fig. 1 shows SEM images of a typical electrochemical etched Ir tip.

The nanoscale lithographic experiments were conducted on a commercial STM system (Nanoscope III Multimode, Digital Instruments) at room temperature. The images of modification patterns were obtained right after the lithographic process by the same tips. Both imaging and lithography operations were performed at constant current mode with feedback loop switched on. Scanning parameters for normal imaging were positive tip bias voltage 500 mV and setpoint tunneling current 500 pA; for lithography, the etching parameters were positive tip bias voltage ranging from 1.8 to 2.5 V, setpoint tunneling current 60 pA, scan speed 500 nm/s, and 100 scan repetitions per line. During the lithographic process, the tip movement was defined by a NanoscriptTM programme.

3. Results and discussion

Fig. 2(a) displays a typical data set for the line etching experiments in which eight lines were etched by scanning the STM tips back and forth over a selected area. The etching sequence was from up left to down right, as indicated by the numbers above each line. The etching parameters were bias voltage 2.6 V, tunneling current 60 pA, scan speed 500 nm/s and 100 scan repetitions per line. It should be noted that in this experiment the bias voltage must be applied positive to iridium tips in order to obtain successful line etching on SRO thin film surfaces.

Fig. 2(b) presents quantitative data of the dependence of line-depth on etching sequence. It is obvious that there is an evolution process of line-depth from line 1 to line 4 in the upper row. After four lines, the etching is more stable and reproducible. The two end parts of each line were removed in depth measurement because there is a time lag when the tip is changing direction at the ends of each line, leading to deeper etching.





Fig. 2. (a) STM image $(350 \text{ nm} \times 350 \text{ nm})$ of a typical eight-line pattern from the line etching experiments. Each line is $\sim 100 \text{ nm}$ long. The etching sequence is indicated by the numbers above each line. The etching parameters are bias voltage 2.6 V, tunneling current 60 pA, scan speed 500 nm/s and 100 scan repetitions per line. (b) Line-depth vs. etching sequence using the same etching parameters in (a).

Additionally, we define a successful etching if etched line is continuous for at least 70 nm with a minimum average depth of half a unit cell. Therefore, only successful lines from the bottom row were analyzed in this work.

The analyzed line-depth as a function of bias voltage and tunneling current is plotted in Fig. 3. The line-depth increases from 0.6 to 7.7 nm as bias voltage increasing from 1.8 to 2.8 V, while the increase of the tunneling current does not change the depth significantly. This observation, therefore, suggests that etching mechanisms such as electron migration [2] and local heating [17] could be ruled out because both are strongly current dependent. The slow increase of depth with tunneling current is consistent with field-induced emission in which the electric field *F* is related to tunneling current *I* by $F \propto -1/\log I$ [6]. In other words, the field-induced evaporation is probably a dominant

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