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Ultimate nanopatterning of Si substrate using filtered liquid metal alloy ion source-focused ion beam

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ABSTRACT

In this work we study the influence of the major focused ion beam operating parameters: ion chemical species, beam current, lens voltage and ion dose on the ultimate nanopatterning resolution. We propose a two-step process based on first ion milling of a SiO_2 sacrificial layer and second SiO_2 chemical etching for the fabrication of nanopatterns with ultimate size/density and ad libitum shape. Examples of resulting patterns are presented.

1. Introduction

Liquid metal alloy source focused ion beam (LMAIS-FIB) technology offers unique capabilities to create patterns with ultimate size and shapes designed on demand due to the maskless local nanopatterning process while using specific nonpolluting ion sources. The extended applications foreseen in nano- and microelectronics are dependent on the reproducibility, speed and resolution of the process of pattern fabrication. Ultimate nanopatterning resolution can be achieved when using optimized experimental conditions based on the complex relations between the various operating parameters which influence ion-matter interaction processes. Focused ion beam (FIB) technique is commonly used for micromachining and preparation of transmission electron microscopy (TEM) samples. Recently new processes involving local patterning and subsequent deposition in the predefined patterns at the nanometer scale were demonstrated [1–6]. The main advantage of LMAIS-FIB is the flexibility of its direct local one step patterning as compared to the costly and complex electron beam lithographic technique that requires numerous steps [7]. However, when the heavy incident ion beam, bombards the surface of a substrate, complex phenomena are produced by the swelling, deposition, etching, and implantation occurring simultaneously [8].

The FIB column operates similarly to the column of a scanning electron microscope (SEM). Both instruments rely on a focused beam to create a specimen image; an ion beam for the FIB and an electron beam for the SEM. For both instruments, the intensity of the secondary particles (electrons or ions) produced during the scanning of the beam on the surface is displayed to construct an image of the sample. The ion source type used in most of the commercial and research systems designed for micromachining applications is the liquid metal ion source (LMIS) [9] using Ga^+ ions. Such sources offer good resolution and stability performances but Ga^+ ions have detrimental effects for many applications (contamination, heavy implantation, etc.). New metal alloy sources (LMAIS) such as AuGe and AuSi have been developed to overcome these limits [10–13]. LMAIS are generally not liquid at room temperature and should be heated at the eutectic temperature of the metal alloy. In LMAIS-FIB, the ion beam is constituted of the elements of the alloy. A mass filter separates and selects one of the source elements that is used for the nanopatterning process.

In this study we investigate the influence of LMAIS-FIB parameters on ultimate nanopatterning resolution in particular the influence of the ion source (Ga^+ and Au^{2+}) and the ion current and dose; using ultra-high resolution ion beam (about 4 nm) when probe currents are below 10 pA. These operating conditions have not been studied previously. Patterns of different sizes and shapes fabricated using Ga^+ and Au^{2+} were compared. We focus on LMAIS-FIB operating conditions for ultra-small patterns. We show that both the size and

Table 1
Primary ion beam parameters for experiments.

Acceleration voltage	30 kV
Extracting voltage	7.00 kV
Suppressor voltage	0.80 kV
Beam current	2.40 μA
Heating current	2.50 A
Probe aperture	10–20–50 μm
Mass aperture	10–20–50 μm

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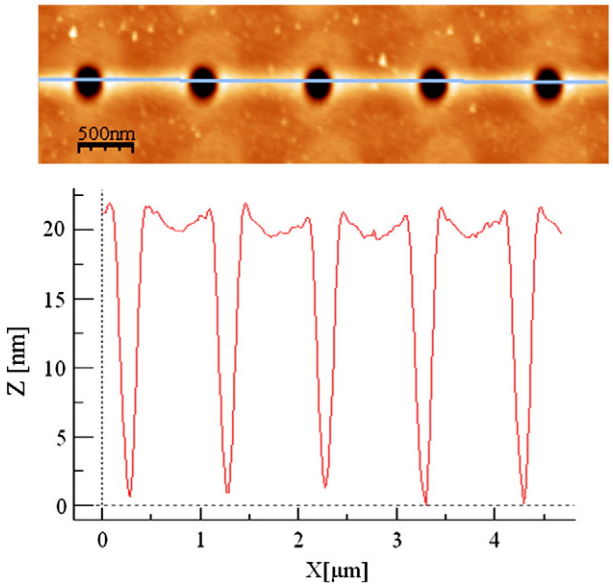

Fig. 1. AFM line profile of the patterned surface that evidences the conical shape of the holes.

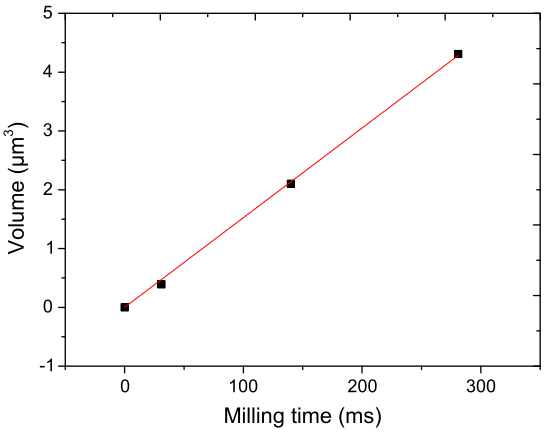

Fig. 3. Variation of volume as a function of milling time.

the position of each pattern can be controlled at the nanometer scale by varying different FIB milling parameters. Finally, we develop a two-step process based on first ion erosion of a sacrificial SiO_2 layer in optimized operating conditions and second the SiO_2 chemical etching. This process allows the fabrication of patterns with ultimate resolution of 15 nm (~4×10^{11} patterns/cm^2) and ad libitum design.

2. Experiments

The samples used in this study are monocrystalline Si (111) and (100) substrates. The substrate preparation is detailed in the following paragraph. This work was carried out using a commercial COBRA-FIB from Orsay Physics system integrated SEM Tescan Lyra. This Tescan LYRA dual-beam equipment uses a liquid metal alloy ion source (LMAIS). A Wien filter allows the ion selection with a resolution of 2 meV. The energy can be varied between 5 and 30 keV. For this work the LMAIS used is an eutectic AuSi alloy heated at 380 °C. The patterns were performed with Ga^+ and Au^{2+} ions selected with the mass filter (3.27×10^{-25} kg) using a mass selection aperture of 10, 20 and 50 µm. Table 1 lists the main operating conditions. The dwell time was increased from 0.8 to 500 µs, while the probe current was varied between 1 and 500 pA and the objective lens voltage was between 16.477 and 16.497 kV. Patterns with different hole size, depth and periods were fabricated by FIB.

Before the FIB process, the samples were cleaned in order to remove metallic and organic contamination. The cleaning process used follows a modified Shiraki recipe: i) 5 min in HNO_3 (65%) heated at 70 °C, ii) 1 min in deionized water and iii) 30 s in HF (49%):H_2O (1:10).

The experimental parameters varied were the ion beam and dose, milling time, probe and mass apertures, and objective voltage. The distance between the patterns was varied between 0.1, 0.5 and 1.0 µm. After each process step the samples were systematically ex-situ investigated by atomic force microscopy (AFM) and scanning electron microscopy (SEM). We used the AFM images to determine the hole depth, and radius. SEM was used to check the validity of the AFM measurements of the radius. TEM cross section samples were also realized using FIB preparation. TEM samples were observed in a Jeol JEM2010F at 200 kV. A PSIA XE-100 AFM was used in non-contact operation mode, the tip model used is NCHR-50 for very high resolution imaging and its typical radius is about 8 nm. The depth of the patterns was limited to some tenths of to rule out any effect of AFM tip radius on their size measurements.

The rate of sputtering is evaluated by measuring the volume of the patterns as a function of the experimental parameters. The latter was

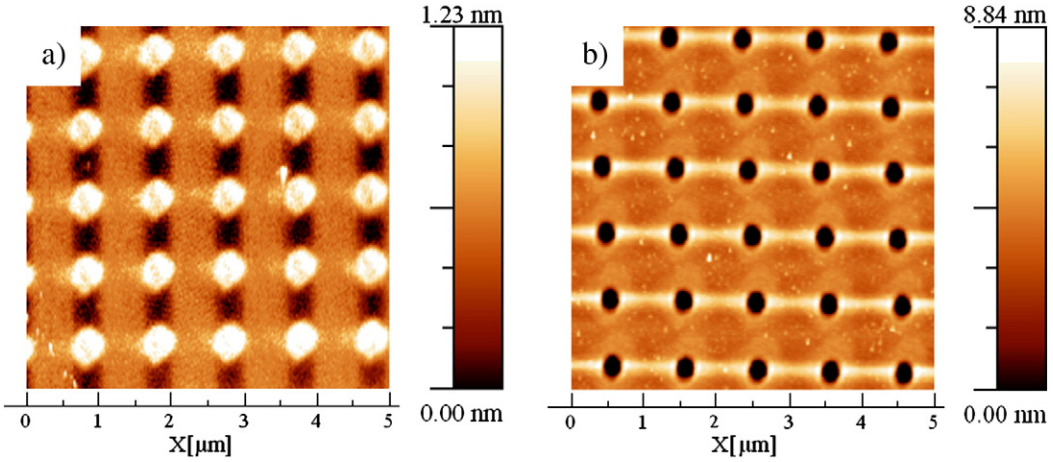

Fig. 2. AFM images of the patterns obtained under different FIB irradiation conditions (a) 0.05×10^{15} ions/cm^2/s and (b) 4.09×10^{15} ions/cm^2/s.