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Full Length Article

Site-controlled fabrication of silicon nanotips by indentation-induced selective etching

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ABSTRACT

In the present study, the indentation-induced selective etching approach is proposed to fabricate site-controlled pyramidal nanotips on Si(100) surface. Without any masks, the site-controlled nanofabrication can be realized by nanoindentation and post etching in potassium hydroxide (KOH) solution. The effect of indentation force and etching time on the formation of pyramidal nanotips was investigated. It is found that the height and radius of the pyramidal nanotips increase with the indentation force or etching time, while long-time etching can lead to the collapse of the tips. The formation of pyramidal tips is ascribed to the anisotropic etching of silicon and etching stop of (111) crystal planes in KOH aqueous solution. The capability of this fabrication method was further demonstrated by producing various tip arrays on silicon surface by selective etching of the site-controlled indent patterns, and the maximum height difference of these tips is less than 10 nm. The indentation-induced selective etching provides a new strategy to fabricate well site-controlled tip arrays for multi-probe SPM system, Si nanostructure-based sensors and high-quality information storage.

1. Introduction

To explore new micro/nanofabrication methods with high precision, low cost and high flexibility is of much concern [1,2]. Especially, site-controlled nanofabrication is of importance for the formation of high-quality quantum structure arrays and creation of quantum devices [3–5]. Due to its simplicity, low-cost and flexibility, scanning probe microscope (SPM) showed robust performance in local anodic oxidation [6,7], manipulation of single atoms [8,9], dip-pen nanolithography [2,10], surface texturing [11,12] and so on. As its basic ability, SPM lithography provides a maskless and straightforward approach by mechanical scratching to produce site-controlled nanostructures including nanochannels and protrusive hillocks on the surface of various materials, where mechanical interaction plays a dominant role in the material removal [13,14]. The mechanical scratching can easily result in the wear of a diamond tip [15]. Moreover, the scratch-induced structural damage beneath the nanochannel area, such as amorphization and lattice distortion, cannot be avoided. Such structural damage will degrade the physical and mechanical properties of fabricated nanostructures [16,17]. An alternative SPM-based nanofabrication can be realized by friction-induced selective etching, and protrusive structures or deep grooves can be produced by post etching of a scratch on material surface, such as silicon, quartz, glass and so on [18–20]. For producing protrusive nanostructures, the amorphous layer and/or deformed crystal layer beneath the scratched area can act as an etching mask in KOH aqueous solution [21–23]. In contrast, the scratched area after tribochemical removal of surface oxide can promote the chemical attack from the etching solutions and result in deep groove [24]. It is also noted that high-aspect nanostructures can be produced on monocrystalline silicon through friction-induced selective etching of a scratched Si_3N_4 mask [25]. Various patterns, including slopes and hierarchical stages, can be produced by programming the loading mode and scanning traces. The friction-induced selective etching provides an active way for site-controlled nanofabrication without any masks.

Since the nanoindentation can lead to a series of crystal transformation beneath the contact area [26], the pressed area is expected to act as a mask in post etching. In the present study, a simple nanofabrication method through indentation-induced selective etching was proposed to prepare pyramidal nanotips on silicon surface. The effect of indentation force and etching time on the

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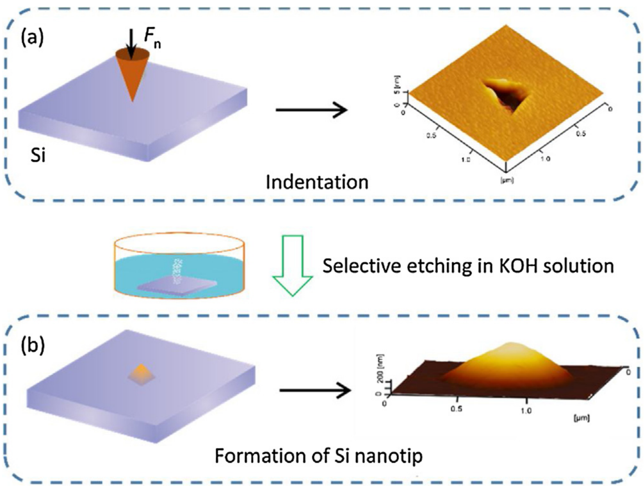

Fig. 1. Schematic diagram for the fabrication process by the indentation-induced selective etching. (a) Schematic diagram and AFM image of an indent produced on Si surface under an indentation force F_n. (b) A pyramidal tip created on Si surface after selective etching in KOH aqueous solution.

formation of pyramidal tips was investigated, and the fabrication mechanism was addressed. As a maskless and straightforward nanofabrication approach, the indentation-induced method provides a new strategy to fabricate well site-controlled nanoscale tip-arrays and high-density storage structure.

2. Materials and methods

2.1. Materials

The Si(100), Si(110) and Si(111) wafers were purchased from MEMC Electronic Materials, Inc., USA. To eliminate the effect of the native oxide layer on the fabrication, silicon wafers were dipped in 5 wt.% HF solution for 2 min to etch off the oxide layer [24]. By an atomic force microscope (AFM; SPI3800N, Seiko Instruments Inc., Japan), the surface root-mean-square (RMS) roughness of the silicon wafers was measured as about 0.1 nm over a 1 μm × 1 μm area before and after etching in HF solution. Then the samples were ultrasonically cleaned with the acetone, ethanol and deionized water for 3 min in turn to remove surface contaminations.

2.2. Fabrication methods

As shown in Fig. 1, the fabrication process consists of nanoindentation and post etching. With a Berkovich tip and an in-situ nanomechanical test system (TI900, Hysitron Inc., USA), a series of patterned indents on silicon surface were produced in air under various applied normal loads F_n of 2, 3 and 4 mN (Fig. 1a). Then the indented surface was immersed in KOH aqueous solution for various etching time, and pyramidal nanotips were produced in-situ from the indentation area on silicon surface (Fig. 1b). A mixture of 20 wt.% KOH solution and isopropanol alcohol (IPA) with the volume ratio of 5:1 was used as the etchant for selective etching, and IPA was employed to improve the surface quality [24]. The temperature for the selective etching was set as 25 °C ± 0.5 °C. All AFM images of the fabricated nanostructures on silicon surface were scanned by a Si_3N_4 tip (MLCT, Veeco Instruments Inc., USA) in vacuum with a nominal tip radius of ~20 nm.

3. Results and discussions

3.1. Effect of etching time on indentation-induced selective etching

The etching time has a strong effect on the formation of pyramidal nanotips during indentation-induced selective etching. An indent with a depth of about 6 nm was firstly produced by indenting under F_n = 2 mN. Then a pyramidal nanostructure was detected on the indent area after dipping in KOH aqueous solution, as shown in Fig. 2. It was noted that the formation of nanotips was obviously etching time-dependent, and the height of the nanostructure increased firstly and then decreased with increasing etching time. The protrusive nanotips almost collapsed completely after etching for 60 min. The variation of the height of these nanotips with etching time was plotted in Fig. 3. The height increased from −6 to 280 nm with the increase in etching time from 0 to 20 min, and 20 min-etching led to a maximum height. Since the deformation layer beneath the indented area can be etched away gradually with the increase in the etch time [22]. Long-time etching can also

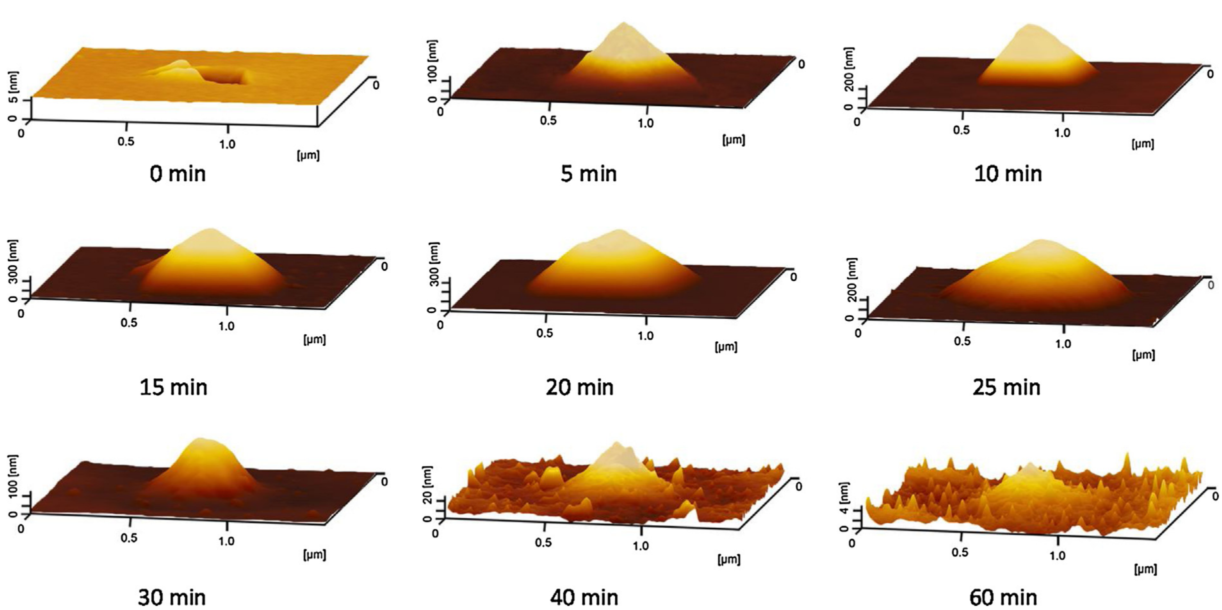

Fig. 2. Indentation-induced selective etching under various etching time. The indentation force for the indentation is 2 mN.