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Fabrication of nanopore on pyramid

Seong Soo Choi[a,*], Myong-Jin Park[a], Tokutaro Yamaguchi[a], Sung-In Kim[b], Kyung-Jin Park[b], Nam Kyoo Park[c]

[a] Department of Nanoscience and Instrument Center, SunMoon University, Ahsan 336-708, Republic of Korea
[b] Team for Measurement and Analysis, National Nanofab Center, Daejeon 305-806, Republic of Korea
[c] School of Electrical Engineering, Seoul National University, Seoul, Republic of Korea

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ABSTRACT

There have been tremendous interests about the fabrication of metallic nanopore due to the ultrafast genome sequencing biosensor capability. In this report, the fabrication of the nanopore on the Au coated SiO_2 pyramid has been examined using various high energy electron beam irradiation and focused ion beam (FIB) milling techniques. The microfabricated Au nano-apertures on pyramid were irradiated with various high energy electron beam and FIB techniques. The formation of the nanopore dependent on the probe current was also examined using electron probe micro-analysis (EPMA). The nanopore on the Au coated SiO_2 pyramid is found to be an Au—Si mixture. The Au nanopore on the crater type hole was also fabricated using FIB Ga ion beam scanning. The shrinking rate was found to be the fastest compared with those fabricated with the other electron beam techniques.

1. Introduction

There have been many studies related to fabrication of solid state nanopores using various techniques such as ion sculpting and electron beam exposure due to the possible ultrafast genome sequencing capability [1–4]. So far, fabrication of a nanopore on the thin membrane using various high energy electron beam techniques was carried out, but its physical dynamics of nanopore formation has not been clearly stated. When a high energy electron beam hits the nanometer thick membrane, the electrons will experience atomic sputtering and atomic displacements from elastic scattering by atomic nuclei, and inelastic scattering by atomic electrons. The inelastic scattering will result in heating of the substrate [5–7]. Electron beam irradiations using field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), electron microprobe analyzer (EPMA) have been utilized in fabricating the nanopore [8–11].

For TEM electron beam bombardment, resizing of the nanopore becomes dependent upon the membrane thickness and the diameter of the nanopore. Depending upon the ratio of the pore diameter to the thickness of the membrane, the aperture will be widening, or be closing. Even without electron beam irradiation, thermal annealing of Au thin film at 563 K can also provide the aperture opening and closing depending on the ratio of the pore diameter to the membrane thickness. These phenomena are attributed to the surface tension of the viscous membrane [12,13].

Electron beam induced melting was previously reported [6,7]. The temperature rise from high energy electron beam exposure can make the membrane viscous. In order to test the surface tension modeling on the liquid membrane, the nanopore formation using direct thermal heating at the melting temperature of silicon oxide was also examined [14]. However, the radiation damage

* Corresponding author. Tel.: +82 41 530 2855; fax: +82 41 530 2871.
E-mail address: sscphy2010@gmail.com (S.S. Choi).

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from scanning electron beam with MHz scan frequency such as field emission scanning electron microscopy (FESEM) is reported to provide the *solid state phase* change of the bulk specimen, rather than *liquid phase* surface modification [15,16]. For FESEM electron beam exposure on the nanometer thick membrane, the size of the nanopore is reported to *shrink always* regardless of the thickness and the diameter of the nanopore [10,11]. This phenomenon can be attributed to *solid state* surface modification; radiation induced dissociation of atomic bonding and atomic migration [7,15]. For atomic displacement during electron beam irradiation, the energy transfer should exceed the displacement energy. The displacement energy for the Si and the Au is 15 eV and 34 eV, respectively. The corresponding threshold electron energy for the Si and the Au is 150 keV and 1320 keV, respectively [7,11]. It is reported that there is a strong attractive interaction between Au and Si in the liquid state and a repulsive interaction between Au and Si in the solid state [17,18]. Hence, the Si atom can form an Au—Si mixture with a low liquid eutectic point at 636 K. During high energy electron beam irradiation, the temperature rise of the Au/SiO_2 specimen can be high enough for the surface to become viscous. The dissociated Si atoms mobilized from SiO_2 by radiation damage during electron beam irradiation can also form the Au—Si liquid mixture layer at the Au/SiO_2 interfacial region. The Au—Si alloy would flow into the pore region, and then result in shrinking of the nanopore.

Nanopore formation using focused Ga ion beam milling techniques has also been carried out on the 200 nm thick Au freestanding membrane. Upon high energy Ga ion impingement on the sample surface, atomic mixing occurs in the melted region within picoseconds. The ions diffuse in the viscous membrane and form the nanopore. This phenomenon can be attributed to the thermal spike model [3,19]. The pore closing can also be attributed to either creation of stressed viscous surface layer and atomic transport to the pore, or mobile surface atom diffusion to the pore [2]. In addition, nanopore formation using FIB ion sculpting technique can provide a carbon-free nanopore. Initial scanning of the specimen surface for 1 s or less can remove the carbon-contaminated surface layer [7]. Hydrocarbon nanopore formation using electron beam irradiation was also reported [20]. Removal of the hydrocarbon on the specimen can be carried out using either O_2 plasma treatment, or UV irradiation with double wavelength [21,22].

Recently, mono-layer graphene nanopore in addition to SiN and SiO_2 nanopore was utilized as a nanopore sensor device for electrical detection [1,23]. However, the plasmonic Au nanochannel or Au nanopore can be utilized as an optical biosensor due to the plasmonic "hot spot" at the Au nanopore apex [24,25]. Furthermore, a nanopore on the apex of the pyramid can be more stable than a nanopore on the graphene during device processing.

In this experiment, a nanopore was fabricated on the apex of an Au coated oxide pyramid. The pyramidal nanostructure has the advantage of better mechanical stability over than that of graphene, SiN, and SiO_2 nanometer thin membranes. In order to examine the dynamics of nanopore formation, various electron beam techniques along with FIB scanning technique were performed to fabricate the nanopore. During high energy electron beam irradiation, the Si—O bonding of the SiO_2 membrane can be broken, and the Si atoms along with the Au atoms can migrate toward the nanopore regions. The Au evaporation rate due to the high Au vapor pressure of $\sim 3 \times 10^{-5}$ Torr at the 1337 K melting temperature would be a considerable factor for the dynamics of nanopore formation under $\sim 10^{-8}$ Torr operating pressure [26,27]. The vaporization of the stainless steel during high energy electron beam irradiation was also reported before [28]. Hence, we investigated the nanopore formation dependence upon the probe current, the primary voltage, and the scan rate using EPMA, FESEM, and TEM. In addition, pore formation on a free standing Au membrane was also carried out using FIB technique.

2. Experimental process and results

2.1. Microfabrication of Au aperture on the pyramid

Pyramidal nanometric size apertures on top of oxide pyramids were initially fabricated using conventional Si microfabrication techniques. Then, 200 nm Au thin film deposition using sputter deposition technique was carried out. The electron beam irradiations, or FIB drilling was followed in order to fabricate the nanopore. The hollow pyramidal structure with the nanopore on its apex can be utilized as a bioreactor and genome sequencing device. The detailed fabrication process is presented elsewhere [29,30].

Fig. 1 presents the initial oxide pyramid in (a) and an oxide aperture with ~200 nm diameter (b) after the long backside Si bulk etching by TMAH. Fig. 1(c and d) presents the images of FESEM and TEM, respectively, for the 200 nm thick Au deposited nano-aperture after the two step sputter metal deposition. The dotted circle, as shown in (c), indicates the Au aperture with 20 nm × 40 nm after the two step Au deposition. A ~30 nm diameter aperture surrounded by ~65 nm wide thin Au membrane is shown in TEM image (d) using 200 keV TEM (JEM-2010).

2.2. Aperture reduction using various high energy electron beam exposures

It is very difficult to control the nanopore using deposition technique only as in Fig. 1. Hence, in order to better control the size of the nano-aperture, the various electron beam techniques are applied, such as FESEM (JSM-6400), TEM (JEM-2010, JEM 3011 HR) and EPMA (JXS-8100) and FIB (FEI Helios). FESEM provides the electron energy with 1–30 keV and current density with ~nA, and a probe diameter of the order of nanometer size. TEM can provide the high electron voltage (~200 kV or 300 kV) and the current (~order of

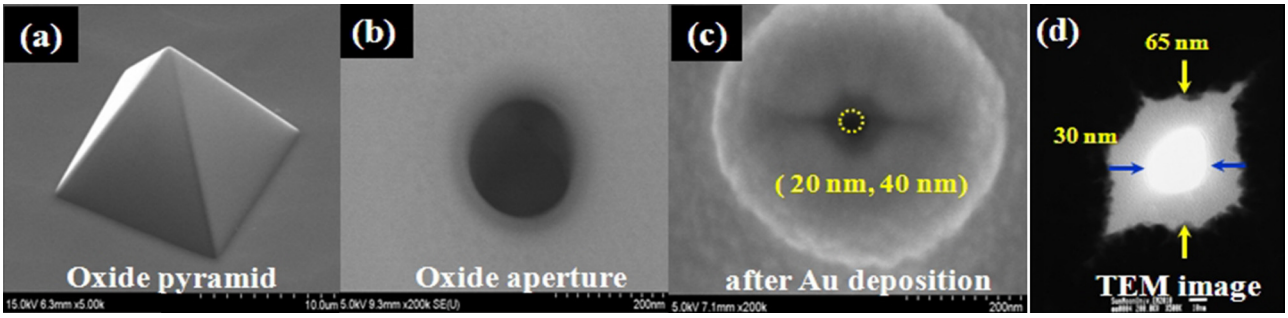

Fig. 1. FESEM pictures show the oxide pyramid (a) and the oxide aperture with ~200 nm on the apex of the pyramid in (b). The 20 nm × 40 nm SEM image of nano-aperture on the Au nanoflower is shown after two step sputter-deposition in (c). The TEM image for the same nano-aperture is shown in (d). The 30 nm wide Au nanopore is surrounded by the thin Au gray membrane with 60 nm × 65 nm size. Au atoms from the dark Au surroundings were diffused and formed the thin membrane.

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