Effect of protective layer deposition in cross-sectional analysis of focused ion beam in germanium substrate

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Abstract. In this work, a single micro-size circular and rectangular holes were fabricated in phosphorus/tin implanted germanium substrate using ion milling of FIB, for potential application in photonic crystal and FINFET. One of the important analysis that need to be done in the future is a cross-sectional analysis in order to evaluate the milling rate of material, redeposition of milled material, and the quality of the milled surface in the pattern structure. However, a well-known practice in preparing a cross-sectional sample for transmission electron microscope analysis using FIB involves a deposition of protective layer on the substrate surface before performing the cross-sectional ion milling, in order to prevent the surface damage. Taking this into consideration, this work performed a preliminary study to analyze the effect of depositing a protective layer onto the hole for performing FIB cross-sectional analysis of the fabricated hole structures. Two-step deposition technique of platinum layer involving electron beam, followed by ion beam was performed on the hole prior to the cross-sectional milling. Hole structure without protective layer was also prepared as a comparison. It is found that although the depth of the milled area can be evaluated in both cross-sectioned holes with/without protective layer, an evaluation on roughness and redeposition are difficult, specifically in the one with protective layer. While the existence of secondary damage might affect the quality of milled surface during the cross-sectional milling in the sample without protective layer.

1. Introduction

Micro- and nanoscale patterning of the functional material is crucial in semiconductor integrated circuits due to the demand of miniaturization that can realizes device notable performance capability. Combination of optical/electron beam lithography with ion etching is the most prominent techniques for the structures patterning in fabricating micro- and nanoelectronic devices. However, this requires costly high-resolution-masks, and not worthy for a low-volume fabrication of micro- and nanodevices, or for the fabrication of single test devices. Ion “Direct-Write” lithography, also called as focused-ion beam (FIB) lithography is the mask-less technique that use a system that enables direct write of shape/pattern, follows by the direct milling of the shape/pattern to fabricate the structures on substrate [1].

A “dual beam” is a single system consists of an FIB and scanning electron microscope (SEM). It enables removal of materials for modification and patterning of the sample using ion beam, and
performing a non-destructive imaging analysis using electron beam in the same equipment. One of the prominent features from this combination is a cross-sectional analysis of a specific material or shape/pattern where a selected region-of-interest will be cross-sectionally milled by FIB and the exposed cross-sectional structure can be directly observed using SEM. Another additional feature of dual beam system is that both ion and electron beams can be utilized for deposition of materials such as platinum (Pt), carbon (C) and tungsten (W).

Milling in FIB is also known as sputtering, and structure patterning as well as cross-sectional milling, can be achieved by the continuous sputtering process that occurs during ion beam exposure on the sample [2]. Meanwhile, material deposition by FIB can be achieved by flowing the precursor gas on the surface of sample. When the ion or electron beam hits the surface of sample, the adsorbed precursor gas will decompose and the desired reaction product remains on the surface [3].

In this work, a single micro-size circular and rectangular holes were fabricated in phosphorus/tin implanted germanium (Ge) substrate using ion milling of FIB in the dual beam system, for potential application in photonic crystal and FINFET. One of the important analysis that need to be done in the future is a cross-sectional analysis in order to evaluate the milling rate of material, redeposition of milled material, and the quality of the milled surface in the pattern structure. However, a well-known practice in preparing a cross-sectional sample using FIB for transmission electron microscope (TEM) analysis involves a deposition of protective layer on the surface before performing the cross-sectional ion milling, in order to prevent the surface damage. Taking this into consideration, this work performed a preliminary study to analyze the effect of depositing a protective layer onto the hole for performing cross-sectional analysis of the fabricated pattern structures.

2. Experimental Work
A p-type Ge substrate (100) co-implanted with n-type dopant ions of phosphorus (P) and non-dopant ions of tin (Sn) with energy and dose concentration of 40 keV/6.0×10^{14} cm^{-2} and 130 keV/4.2×10^{14} cm^{-2}, respectively [4,5]. Laser thermal annealing using KrF excimer laser (λ= 248nm) with laser fluence of 300 mJ/cm² was then performed to activate the dopant atoms and recrystallized the doping layer. By using the annealed substrate, a single circular and rectangular hole structures (Figure 1) were milled in the dual beam system of FEI Helios Nanolab 650 using gallium ion (Ga⁺) as the source for ion beam. The setting parameters of ion milling current and voltage were 80 pA and 30 kV, respectively. The depth of milling was set to 1.0 μm. In order to evaluate the effect of protective layer deposition, a two-step deposition technique of Pt layer involving electron beam, followed by ion beam was performed on the holes prior to the cross-sectional milling. Hole structures without protective layer were also prepared as a comparison. The cross-sectional area was observed and evaluated using FESEM.

Figure 1. Pattern of circular and rectangular holes for FIB milling
3. Results and Discussion

Figure 2.1 and Figure 2.2 show the FESEM images of the milled circular and rectangular hole structures in Ge substrate. The FESEM viewing tilt angle is at 0°. Roughness on the top milled surface can be observed together with the bottom surface. FESEM imaging at tilt angle of 52° was then performed in the rectangular hole of Figure 2.3 (a) in order to get insight on the damage at the bottom and sidewall surfaces. From Figure 2.3(b), it is found that a nanoporous structure of damage was created at the bottom milled surface which is considered caused by the generation of point defects in the sample due to ion irradiation [6]. In addition, surface redeposition can also be observed on the surface and around the outer side of each holes (Figure 2.1 and Figure 2.2). These occur as an energetic Ga⁺ ions that hit the substrate results in the sputtering of the targeted area/material. The sputtered material then leaves its original position as secondary ions or neutral atoms and accumulated on the milled surface [7]. Although it is impossible to completely eliminate this damage, it can be minimized by using appropriate parameter setting during milling process [8]. Hence raised the importance of cross-sectional analysis to evaluate the damage dependency to the ion milling parameter.

![Figure 2.1 FESEM images of two circular hole structures milled by FIB (Magnification 17.5K)](image1)

![Figure 2.2 FESEM images of two rectangular hole structures milled by FIB (Magnification 12 K)](image2)

![Figure 2.3 FESEM images of rectangular hole at 0° and 52° tilt angle](image3)
As previously stated, a well-known practise in preparing a cross-sectional sample using FIB for TEM analysis involves a deposition of protective layer on the surface of substrate before performing the cross-sectional ion milling. In addition, a two-step deposition technique of Pt layer involving electron beam, followed by ion beam was performed on the hole prior to the cross-sectional milling owing to its capability that can minimize the secondary damage introduced to the substrate surface by the deposition of protective layer [5]. In this work, a 500-nm thick Pt layer was firstly deposited at the selected area of the holes using electron beam. As previously reported, the reason for choosing electron beam in the first step deposition is to minimize the damage between substrate and deposited layer interface. The second step of Pt layer deposition was then performed using ion beam with Pt thickness of 1 µm. The thickness of Pt layer during ion beam deposition can be more than those of electron beam as the surface of substrate has been covered by Pt layer from the electron beam deposition. In addition, deposition time using ion beam is faster than those of electron beam due to the nature of each beam sources. The FESEM images of the holes deposited by Pt layer are shown in Figure 3.1 and Figure 3.2. The cross-sectional milling was then performed in the area covered by Pt layer.

![Figure 3.1 FESEM images at 0° tilt of Pt layer deposited by electron beam on (a) circular hole, (b) rectangular hole (Magnification 5K)](image1)

![Figure 3.2 FESEM images at 52° tilt of Pt layer deposited by ion beam on (a) circular hole, (b) rectangular hole after electron beam deposition (Magnification 8K)](image2)

Figure 4.1 shows the cross-sectional FESEM images of circular and rectangular hole structures with Pt protective layer deposited onto the holes. It is found that although the depth of the milled structure can be evaluated in both holes with protective layer, evaluation on the roughness and redeposition is difficult as the milled surface was covered by the Pt layer. Hole structures without protective layer were also milled for cross-sectional analysis, and the FESEM results of the exposed cross-sectional area were shown in Figure 4.2. Although the hole dimension can be evaluated, the quality of milled surface (i.e.
at top, bottom, and sidewall) might be affected due to the cross-sectional milling process. Consequently, it is difficult to analyze the damage that was originally introduced by the first ion milling that was performed to fabricate the holes. However, when comparing these two techniques, the later part of cross-sectional analysis is considered to be more promising compared to the one with protective layer. Therefore, further consideration on the ion beam parameter needs to be done in the future to minimize the secondary effect on the surface roughness in the cross-sectional analysis sample without protective layer.

![Cross-sectional FESEM images of hole with Pt protective layer (Magnification 20 K)](image)

**Figure 4.1** Cross-sectional FESEM images of hole with Pt protective layer (Magnification 20 K)

![Cross-sectional FESEM images of hole without Pt protective layer (Magnification 20 K)](image)

**Figure 4.2** Cross-sectional FESEM images of hole without Pt protective layer (Magnification 20 K)

4. **Summary**

This work was conducted to evaluate the effect of protective layer deposition in cross-sectional analysis of FIB in Ge substrate. A single micro-size circular and rectangular hole structures were fabricated in phosphorus/tin implanted Ge substrate using ion milling of FIB. The cross-sectional milling was then performed in the substrate with/without Pt protective layer. Here it is found that although the depth of the milled area can be evaluated in both cross-sectioned holes with/without protective layer, an evaluation on roughness and redeposition is difficult, specifically in the one with protective layer. While the existence of secondary damage might affect the quality of milled surface during the cross-sectional milling in the sample without protective layer. Further consideration on the ion beam parameter need to be done in the future to minimize the secondary effect on the surface roughness in the sample without protective layer for the cross-sectional analysis using dual beam system.
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