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Interface Damage of Protective Layer in TEM Lamella Preparation for Highly Doped Ge Substrate

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Abstract. This work reports on the TEM sample preparation of highly doped germanium (Ge) sample using focused ion beam (FIB) technique. The method of the deposition of protective layer is varied to observe the damage at the interface of protective layer and specimen surface. It is shown that TEM lamella prepared using the FIB inevitably contains the surface damage induced by the deposition of protective layer. An effective method of controlling the damage at the interface of protective layer and specimen surface is by introducing a two-step deposition technique of protective layer using electron beam and ion beam, with smooth interface can be observed when the imaging was performed for the cross-sectional TEM.

1. Introduction

Continuous advancement in transmission electron microscope (TEM) has pushed the abilities of the electron microscope technology to even higher resolutions enabling researcher to perform imaging of materials at nanoscale level for specimen's chemistry and crystallography. During the operation of TEM, the specimen need to be uniformly thin enough (< 100 nm) for the high energy beam of electrons to be transmitted and interact with the atoms to gain the aforementioned information. This specimen preparation step is very crucial in TEM and method involved in preparing the sample differs depending on the nature of the material and the information required from it [1,2]. Various preparation methods, such as mechanical polishing techniques and light-ion milling, and focused ion beam (FIB) can be used to obtain cross-sectional TEM specimen. Combination of mechanical polishing techniques and light-ion milling will involve two important steps i.e. grinding/polishing of sample using emery paper (silicon carbide) or diamond lappy film with different grit numbers or particle size, follows by light ion milling using ion slicer or precision ion polishing system. Meanwhile, focused ion beam can directly mill the specimen surface by controlling the energy and intensity of ion beam without polishing the sample. Both ways will eventually result in the TEM specimen with the thickness lower than 100 nm. However, optimization of the specimen preparation steps is important in order to gain the required information from the TEM observation. This work reports on the TEM specimen preparation of highly doped germanium (Ge) sample using FIB technique [3–5]. The method of the deposition of protective layer is varied to observe the damage at the interface of protective layer and specimen surface.

2. TEM Lamella Preparation Method using Dual Beam FIB

In this work, a highly doped Ge (100) substrate, with implanted damaged layer ≈ 60 nm was used for preparing a thin slice of the material, called lamella [3–5]. TEM lamella preparation was demonstrated using dual-beam FIBs machine that enables sample preparation using a focused ion beam of gallium (Ga), and surface imaging of scanning electron microscope using an electron beam, both equipped in two independent microscope columns.

TEM lamella preparation is also known as a lift-out method which consists of five steps:

(a) Deposition of the protective material onto specimen surface on the selected area, follows by the milling of the selected area (cutting pattern) using Ga^+ ions to produce a thin slice of lamella
(b) Extraction of the wedge containing lamella
(c) Lift out of lamella using omniprobe needle
(d) Glue of lamella on top of copper (Cu) grid
(e) Thinning (< 100 nm) and cleaning of lamella using low-voltage focused Ga^+ ion-beam

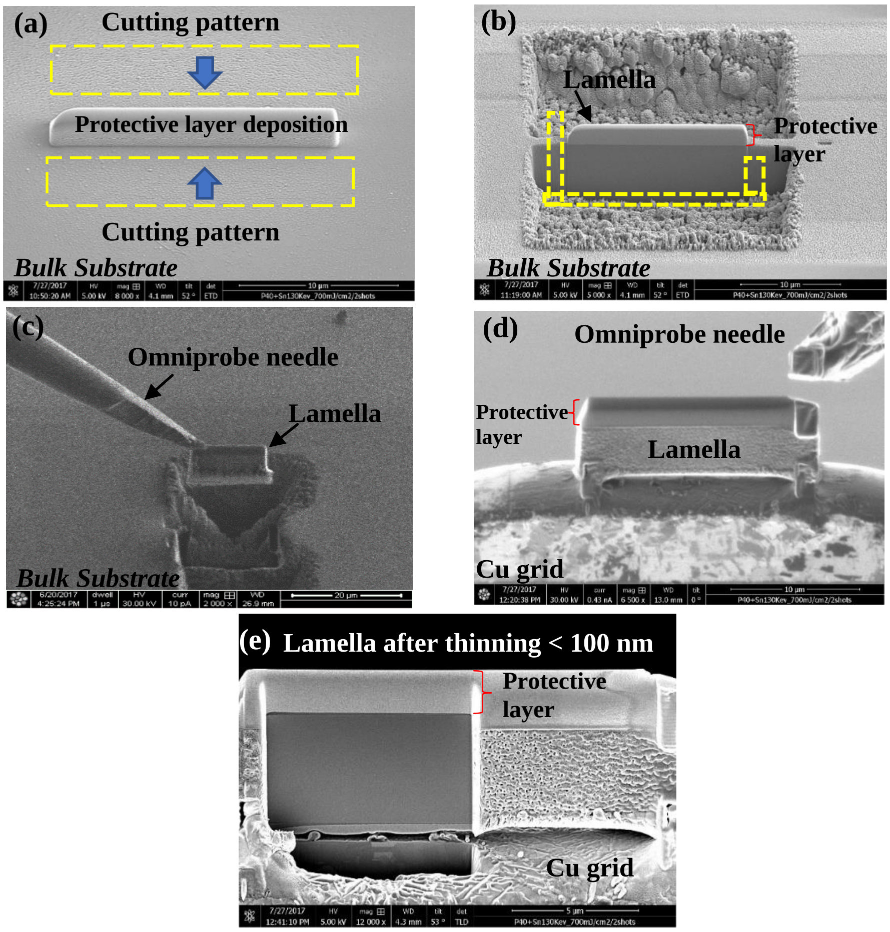

Figure 1. Preparation steps of a FIB TEM lamella

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3. Discussion on the Deposition of Protective Layer

As shown in Fig. 1, the process of FIB lamella preparation starts with deposition of a protective layer on a specific micrometric area on the bulk substrate. This protective layer also functions as a marker to milling specimen for a thin slice of lamella. This protective layer is important in order to provide protection to the top surface of the specimen in the selected lamella area from ion beam induced damage and unwanted milling during the milling and thinning process of lamella. Materials that have been widely used as a protective layer are tungsten (W) and platinum (Pt). Generally, protective layer is deposited by using Ga^{+} ion beam with energy of 30 keV, and thickness of 1 µm. We have prepared a lamella sample with a protective layer of W deposited on the substrate using the parameter in the aforementioned technique. The lamella was then prepared by following the steps as mentioned in Fig. 1. The result of cross-sectional TEM of the lamella at the interface area of protective layer and specimen surface is shown in Fig. 2(a).

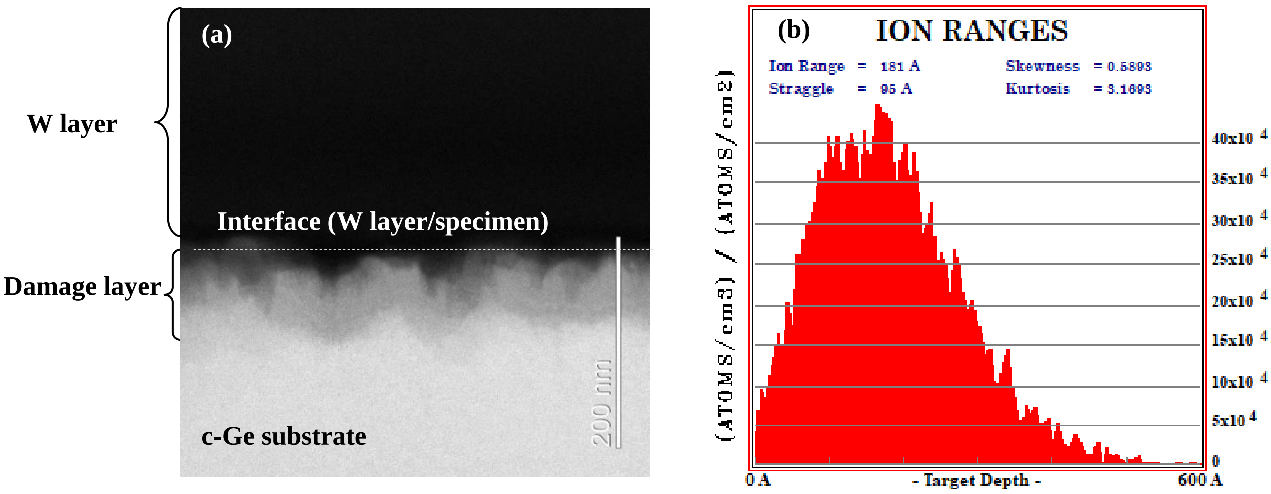

Figure 2. (a) Cross-sectional TEM of lamella prepared by the deposition of W using 30 keV-Ga ion beam (b) Ga ions distribution in Ge substrate at 30-keV ion implantation energy

As can be seen in Fig. 2(a), the deposition of W protective layer using 30-keV ion beam on the surface of highly doped Ge substrate created a rough interface and damage layer with estimated thickness of ≈ 120 nm. This is considered due to the energetics ions beam leads to the inevitable surface damage when the ion is bombarded onto Ge surface. In addition, SRIM-2013 program which calculates the stopping and range of ions into matter using a quantum mechanical treatment of ion-atom collisions [6], shows that Ga ions penetrate deeper into Ge substrate, as illustrated in Fig. 2(b). Fabricated sample is expected to have deeper penetration depth as physical and chemical reactions between the ions and substrate are not taken into account during the calculation [3]. As a result, for the highly doped Ge substrate used in this works, the created interface damage affects the imaging of the original implanted layer which is estimated to be existed ≈ 60 nm near the surface.

In order to control this interface damage, we introduced deposition of protective layer using a low-energy electron beam prior to the ion-beam deposition. We demonstrated this technique by using Pt as the material for the protective layer, with two-step deposition involving 5-keV of electron beam Pt deposition, followed by 30-keV of ion beam Pt deposition with estimated thickness to be ≈ 0.5 µm and ≈ 1 µm, respectively. Figure 3 shows the deposition of Pt using the aforementioned steps in SEM and FIB images. The total thickness for Pt deposition is estimated to be ≈ 1.5 µm.

The lamella was then prepared by following the steps as mentioned in Fig. 1. The cross-sectional TEM result of the lamella prepared using this two-step technique shows smooth interface between the deposited Pt layer (electron beam) and the top surface of Ge substrate (Fig. 4). It is also shown that damages from the milling and thinning of the lamella only affected the top protective layer of Pt deposited by ion beam, while the layer of Pt deposited by electron beam remain and protect the surface of interest for TEM observation. Implanted damage layer of ≈ 60 nm can clearly be seen from the TEM image, proving the effectiveness of this two-step technique.

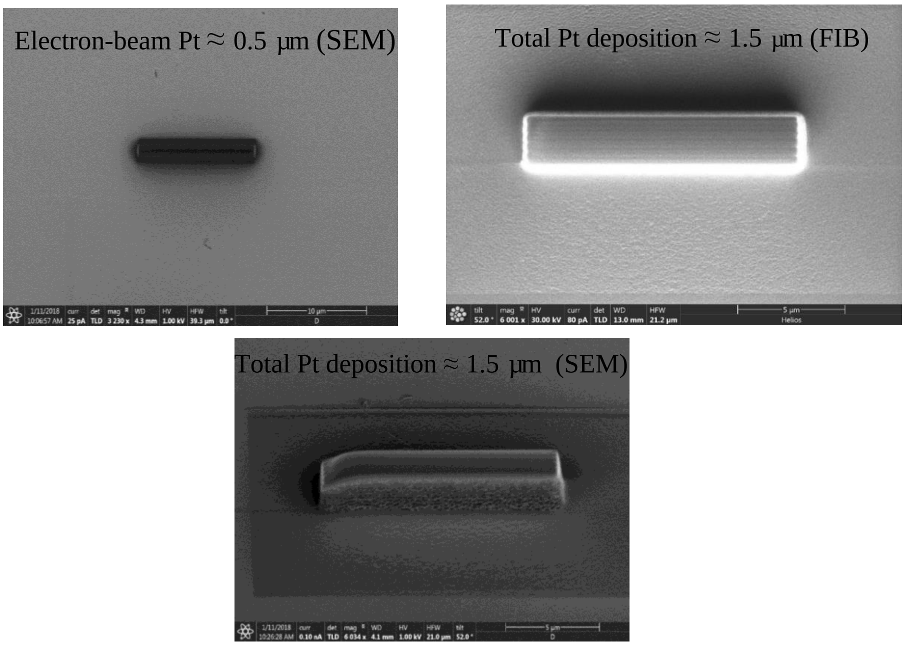

Figure 3. Deposition of Pt protective layer using electron beam followed by ion beam

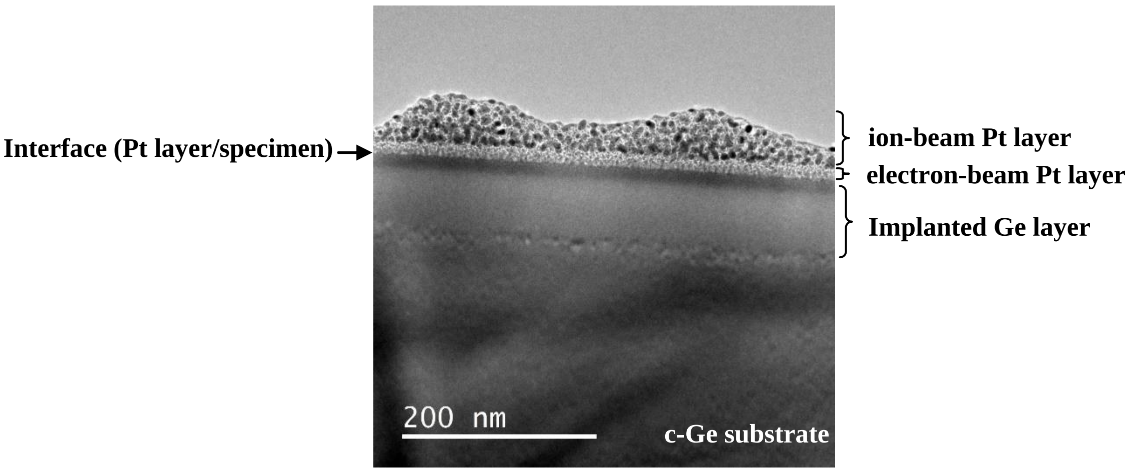

Figure 4. Cross-sectional TEM of lamella prepared by the deposition of Pt using 30 keV-ion beam

4. Conclusion

This work reports on the TEM sample preparation of highly doped germanium (Ge) substrate using FIB technique. Sample preparation steps included are deposition of protective layer, extraction of interest area using milling process, lamella lift-out, glue of lamella on top of copper grid and final thinning process. It is shown that TEM lamella prepared using the FIB inevitably contains the surface

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damage induced by the deposition of protective layer. An effective method of controlling the damage at the interface of protective layer and specimen surface is by introducing a two-step deposition technique of protective layer using electron beam and ion beam. Smooth interface can be observed when the imaging was performed for the cross-sectional TEM. The information gained from this work is important to prepare cross-sectional TEM lamella sample for evaluating specimen such as thin film and shallow pn junction in MOSFET [7 ─ 11].

Acknowledgments
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