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Technical Report

Transmission Electron Microscopy Sample Preparation of $Ge_2Sb_2Te_5$ Nanowire Using Electron Beam

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A simple and novel transmission electron microscopy (TEM) sample preparation method for phase change nanowire is investigated. A $Ge_2Sb_2Te_5$ (GST) nanowire TEM sample was meticulously prepared using nanomanipulator and gas injection system in a field emission scanning electron microscopy for efficient and accurate TEM analysis. The process can minimize the damage during the TEM sample preparation of the nanowires, thus enabling the crystallographic analysis of as-grown GST nanowires without unexpected phase transition caused by e-beam heating.

Key Words: Gas injection system, Nanowire, Transmission electron microscopy sample, Nanomanipulator, Electron beam

INTRODUCTION

$Ge_2Sb_2Te_5$ (GST) is one of the promising phase change materials. The phase of GST can be changed between crystalline and amorphous rapidly, reversibly, and reliably at relatively low temperatures (Raoux, 2009; Raoux & Wuttig, 2010). The phases between crystalline and amorphous produce large differences in electrical and optical properties (Lee et al., 2005; Welnic et al., 2007). Due to its unique characteristics, research has been conducted significantly for various applications such as optical and electrical memories for decades. Especially, it is being used broadly as the main material of phase change random access memory (PRAM) (Horii et al., 2003).

Single crystal GST nanowires (NWs) have been grown by bottom-up process via vapor-liquid-solid (VLS) method using a chemical vapor deposition (CVD) tool (Jung et al., 2006). As-grown GST NWs have hexagonal close packed structure and can be used for phase change applications (Jung et al., 2006; Nam et al., 2012). The devices based on a single crystalline GST NW in defect free condition allow us to examine the phase change mechanism more accurately compared to the conventional GST thin films with pre-existing grain boundaries and fabrication defects. For the crystallographic analysis of the phase change NWs, transmission electron microscopy (TEM) can be generally used.

There are two methods in preparing TEM samples for the crystallographic analysis of NWs. As shown in Fig. 1, one is a "solution dispersion method" and the other is a method using a "focused ion beam (FIB)" (Salkar et al., 1999; Mayer et al., 2007).

The former is the method of picking up diffused NWs in a solution using a mesh-grid. While it provides an advantage of easier preparation and analysis for the characteristics of NWs without incurring any physical changes, it could be difficult to prepare proper TEM samples if the amount of NWs is not enough. Also, the observation of NW orientation is limited to a specific direction in TEM since the cross-sectional shape of GST NW is in the rhombus form (Lee et al., 2007).

On the other hand, the latter is the method of either milling the cross-section of a NW along with a substrate where it is grown (Mayer et al., 2007) or welding a NW on a grid in order to prepare TEM sample with an accelerated ion beam (Peng et al., 2008; Diercks et al., 2009). As only a portion of

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NW is prepared, the method is limited in observing various orientations in the crystal structure. Also, NW exposed to the ion beam accelerated at 30 kV may undergo a phase change during the sample preparation so that it is difficult to understand the unique characteristics of NW.

To overcome the limitations of conventional methods, we suggest a new method of TEM sample preparation for GST NW using a tip and an electron beam (e-beam) deposition in a field emission scanning electron microscopy (FE-SEM) equipped with nanomanipulator and gas injection system (GIS).

METHODS

The GST NWs are grown via the VLS method in a CVD tool at 450°C for 2 hours on a SiO_2 substrate with the pressure of 50 mTorr using Ar and N_2 flow. Au nanoparticles with the diameter of 100 nm are used as metal catalysts (Jung et al., 2006). A selected as-grown NW has a diameter of 90 nm and a length of 4.4 μm, having single crystallinity.

We used Dual Beam FIB equipped with nanomanipulator and GIS to prepare single crystal GST NW sample for TEM analysis. The equipment is manufactured by JEOL (Japan) and is a combination of the FE-SEM part with in-lens Schottky field emission gun and the FIB part using Ga liquid metal source.

We prepared the TEM sample only using the electron beam portion of the Dual Beam FIB to minimize damage to the sample. The specifications for the electron beam of the FE-SEM are accelerated voltage of 0.2 to 30 kV and beam current of 1×10^{-11} to 2×10^{-8} A with a resolution of 1.2 nm at 30 kV.

In this experiment, we used electron beam condition of 5 kV and 3×10^{-10} A for the image condition as summarized in Table 1 in order to find and lift-off a NW.

We used a nanomanipulator (Autoprobe 200.2; Oxford, UK) to transfer the NW to a grid. The nanomanipulator is attached to the Dual Beam FIB and can move to any position while observing the SEM or FIB image. It is possible to move along X, Y, and Z axis at the velocity of 0.1 to 60 μm/sec and to rotate in 360°.

As shown in the "lift-off condition" of Table 1, the sample is lifted off from the substrate by moving the nanomanipulator side to side at the velocity of 2 μm/sec.

The NW is then fixed to a grid using the GIS. As summarized in "bonding condition" of Table 1, the NW was fixed to the grid using the e-beam assisted deposition with the condition of 5 kV and 5×10^{-9} A for 30 seconds.

RESULTS

The process of preparing undamaged TEM sample using electron beam is explained with FE-SEM images as shown in Fig. 2. Fig. 2A is a SEM image of a selected GST NW for the analysis in TEM. Fig. 2B and C are the images showing that the tip of nanomanipulator is inserted and moved to pick up the NW. Because the electron beam is focused on the NW, tip at a distance of 300 μm away from the substrate is out of focus. We can identify the distance between tip and substrate as the tip is being focused.

Fig. 2D is an image showing that the tip adjacent to the substrate moved slowly to contact with NW. As the tip approaches the NW, it could adhere to the tip by electrostatic interaction. If the NW adhering to the tip moves finely, it can be lifted off from substrate. Then, the tip is removed away from stage.

Fig. 2E and F are images that the stage is moved to a grid area and the tip is inserted on the stage. As with Fig. 2B and C, we can identify the distance between tip and grid by how well the tip is focused. Fig. 2G is an image that the NW is fixed on the grid wall using e-beam deposition after being in contact. In this way, we can prepare a TEM sample of NW without incurring any damage.

As shown in Fig. 3, the prepared GST NW TEM sample was

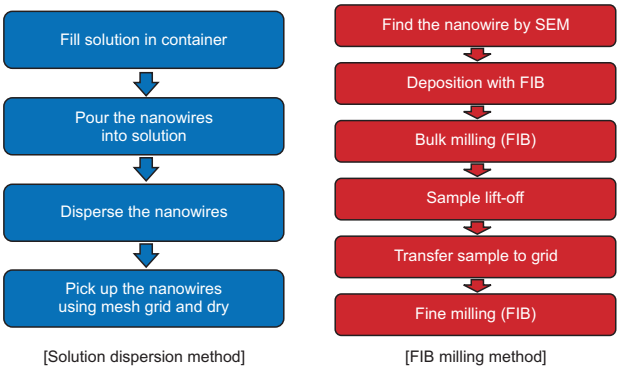

Fig. 1. Typical transmission electron microscopy sample preparation processes. SEM, scanning electron microscopy; FIB, focused ion beam.

Table 1. Transmission electron microscopy sample preparation conditions using electron beam

Item	Condition
Sample diameter	80~100 nm
Equipment	Dual Beam FIB (JIB-4601F; JEOL)
Image condition	Accelerated voltage: 5 kV
	Probe current: 3×10^{-10} A
Lift-off condition	X, Y axis
	Velocity: 2 μm/sec
Bonding condition	Accelerated voltage: 5 kV
(E-beam deposition)	Probe current: 5×10^{-9} A
	Time: 30 sec

FIB, focused ion beam.

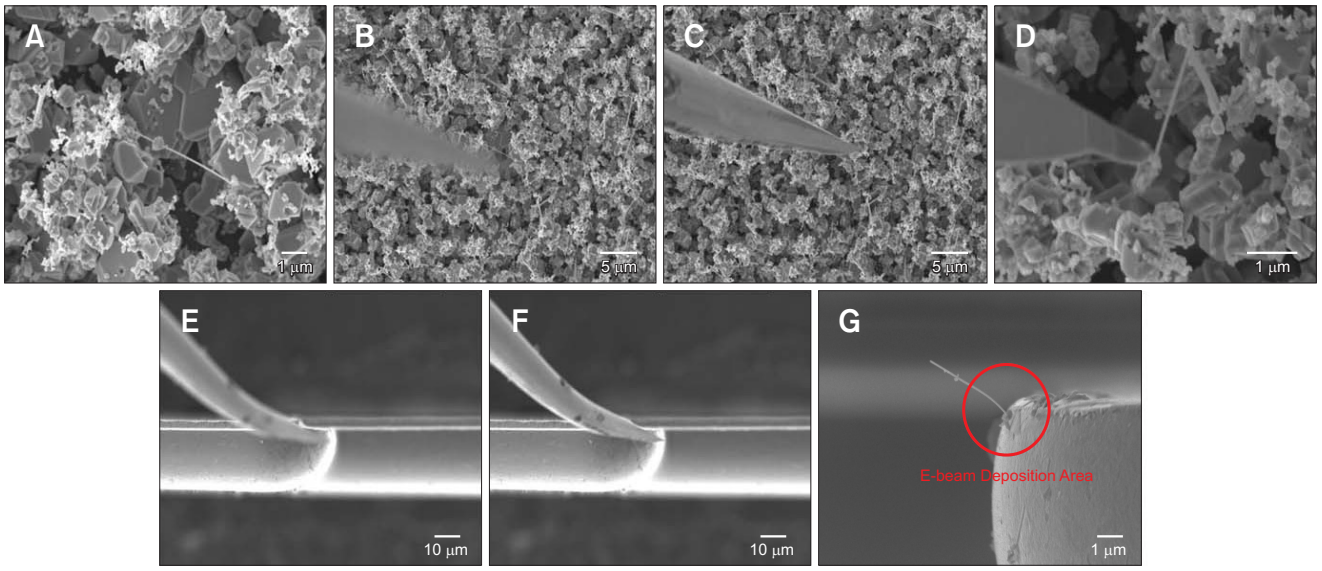

Fig. 2. Transmission electron microscopy sample preparation process using electron beam. Scanning electron microscopy images of a selected $Ge_2Sb_2Te_5$ (GST) nanowire (NW) (×10,000; A), a tip of the nanomanipulator moving to the selected NW (×3,000; B, C), the edge of the tip contacted to the NW (×20,000; D), the tip attached to grid (×1,000; E, F), and the fixed GST NW on the grid wall (×10,000; G).

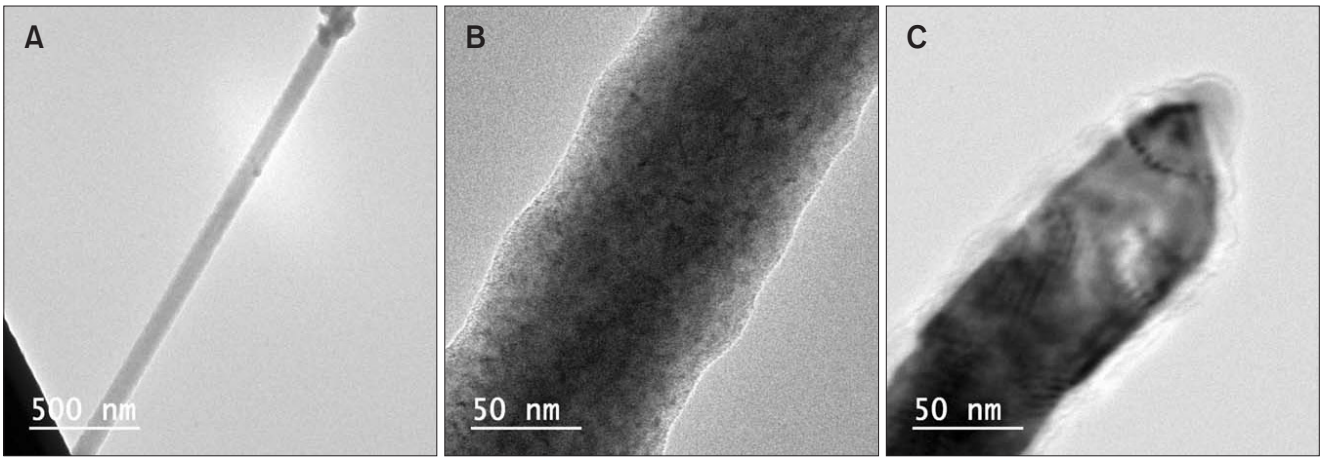

Fig. 3. Transmission electron microscopy (TEM) images of $Ge_2Sb_2Te_5$ (GST) nanowire (NW) prepared by electronbeam. (A) The image being fixed NW on selected area of grid wall. It was possible to find the position of the NW. (B) The image magnifying center of GST NW. It was possible to observe a NW without damage by TEM sample preparation. However, the exposed part of GST NW to e-beam started to melt from the surface because the representative phase change material (GST) has relatively low meting point. (C) The image magnifying edge of GST NW, The end of the NW was not only melted down but also bent and deformed by an electron beam.

analyzed by using a TEM (JEM-ARM200F; JEOL) at 200 kV. Since we fixed NW at the designated position by using SEM, the time for searching the prepared GST NW sample is greatly reduced in comparison to the "solution dispersion method". In addition, the process damage on the sample is minimized compared to the sample produced by using the conventional FIB method.

The as-prepared GST NW has one-dimensional structure as seen in Fig. 3A. As one of the representative phase change materials, GST has relatively low meting point, <650°C. The outer part of the GST NW exposed to e-beam starts to melt from the surface as shown in Fig. 3B due to the heat generated with the electron beam. The melted part of GST NW is brighter than the other parts so it is easily recognizable. This e-beam induced melting leads to the slight deformation in GST NW as seen in Fig. 3C. While atomic resolution analysis of GST NW can be hindered by the rapid melting with the concentrated electron beam at high magnification, we were able to observe the intrinsic property of a phase change NW associated with heating.

CONCLUSIONS

The improved understanding of a phase change material can be approached with the device made of a single crystalline GST NW. Due to the inherent property of the phase change material, GST NW is prone to damage with the conventional preparation process of TEM sample using focused ion beam. By preparing TEM sample of GST NW with our electron beam process, surface damage of the sample has been appreciably reduced.

In addition, using nanomanipulator and e-beam deposition by GIS enabled us to prepare the selected NW as TEM sample for better analysis. This type of TEM sample preparation technique can be applied to a variety of NWs for minimal damage to the sample and improved TEM analysis.

CONFLICT OF INTEREST

No potential conflict of interest relevant to this article was reported.

REFERENCES

Diercks D, Gorman B, Cheung C L, and Wang G (2009) Techniques for consecutive TEM and atom probe tomography analysis of nanowires. *Microsc. Microanal.* **15(S2)**, 254-255.

Horii H, Yi J, Park J, Ha Y, Baek I, Park S, Hwang Y, Lee S, Kim Y, and Lee K (2003) A novel cell technology using N-doped GeSbTe fi lms for phase change RAM. *2003 Symp. VLSI Technol., Dig. Tech. Pap.* 177-178.

Jung Y, Lee S H, Ko D K, and Agarwal R (2006) Synthesis and characterization of Ge2Sb2Te5 nanowires with memory switching effect. *J. Am. Chem. Soc.* **128**, 14026-14027.

Lee B S, Abelson J R, Bishop S G, Kang D H, Cheong B K, and Kim K B (2005) Investigation of the optical and electronic properties of Ge2Sb2Te5 phase change material in its amorphous, cubic, and hexagonal phases. *J. Appl. Phys.* **97**, 093509.

Lee S H, Jung Y, and Agarwal R (2007) Highly scalable non-volatile and ultra-lowpower phase-change nanowire memory. *Nat. Nanotechnol.* **2**, 626-630.

Mayer J, Giannuzzi L A, Kamino T, and Michael J (2007) TEM sample preparation and FIB-induced damage. *MRS Bull.* **32**, 400-407.

Nam S W, Chung H S, Lo Y C, Qi L, Li J, Lu Y, Johnson A T C, Jung Y W, Nukala P, and Agarwal R (2012) Electrical wind force-driven and dislocation-templated amorphization in phase-change nanowires. *Science* **336**, 1561-1566.

Peng Y, Luxmoore I, Forster M, Cullis A, and Inkson B (2008) Nanomanipulation and electrical behaviour of a single gold nanowire using in-situ SEM-FIB-nanomanipulators. *J. Phys.: Conf. Ser.* **126**, 012031.

Raoux S (2009) Phase change materials. *Annu. Rev. Mater. Res.* **39**, 25-48.

Raoux S and Wuttig M (2010) *Phase Change Materials: Science and Applications* (Springer Science & Business Media, New York).

Salkar R, Jeevanandam P, Aruna S, Koltypin Y, and Gedanken A (1999) The sonochemical preparation of amorphous silver nanoparticles. *J. Mater. Chem.* **9**, 1333-1335.

Welnic W, Botti S, Reining L, and Wuttig M (2007) Origin of the optical contrast in phase-change materials. *Phys. Rev. Lett.* **98**, 236403.