

Rolling up Ge microtube from bare-Ge substrate

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ABSTRACT

We show Ge microtubes can be rolled-up from bare-Ge wafer at almost any position, once these films are released from its substrate.

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1. Introduction

Microtubes have attracted considerable attention because of their potential applications in optoelectronics and micro electro mechanical systems (MEMS). In the past, there are two methods to make solid state submicron tubes [1]. Both methods rely on the release of thin layers of the material from a substrate by a selective etching procedure. The position of these tubes is exactly determined by the etching time. By controlling the thickness of the layers, and hence the tube walls, the deposition method can be as precise as a single atomic layer. A typical method is to deposit the film on a selective layer on the substrate. The tubes are rolled up by selective etching of the sacrificial layer. The thin film can be single crystalline or not. And the other method is to grow a strained layer by selective etching. The layer sequence consists a bilayer of two different materials (materials 1 and 2). Material 1 has a larger lattice constant than material 2 [2]. Once the bilayer is released by selective etching, each material tends to acquire its inherent lattice constant. The bilayer bends upwards, finally forming a tube after one complete revolution. Longer etching times result in multiple revolutions. In this study, we propose a new method for making similar solid state micron tubes without the etching procedure. The method uses hydrogen implantation followed by thermal annealing for preparing the solid state microtubes.

2. Materials and methods

To obtain Ge microtubes, the 100 keV H⁺ ions were normally implanted into an n-type Ge wafer with a fluence of $1 \times 10^{17} \text{ cm}^{-2}$. The surface normal of the wafer was identical with (001) crystal axis. The sample was implanted at a room temperature during the implantation process in order to keep the hydrogen induced damage. After implantation, the implanted sample was etched in diluted HCl solution for removing possible surface oxide during implantation or handling. After implantation the Ge microtubes were self-rolled up by 30 min post thermal annealing at 280 °C. Different lengths of Ge microtubes were detached from the Ge substrate. The detached microtubes were transferred to glass or silicon wafer by tweezers for the following analysis. An optical digital camera (DC) and a scanning electron microscope (SEM) were employed to observe the shape of the detached film. The thickness and uniformity of the microtubes were also measured by SEM. The Ge–Ge bonding was confirmed by Raman measurement. Raman spectrum was obtained using 632-nm excitation with 0.1 cm^{-1} resolution.

3. Results and discussion

In order to estimate the hydrogen ion range in the Ge substrate, the SRIM code is used to estimate the depth profile of hydrogen ions implanted in the Ge sample. The dominant mechanism of the crystal slicing is the accumulation of the defects in the sample. SRIM [3] shows the simulated depth and damage profile of 100 keV H⁺ ions in the Ge material. The peaks of the depth and damage profile are located at 0.7 μm, respectively. After the implantation and annealing process, microtubes were detached from the bulk

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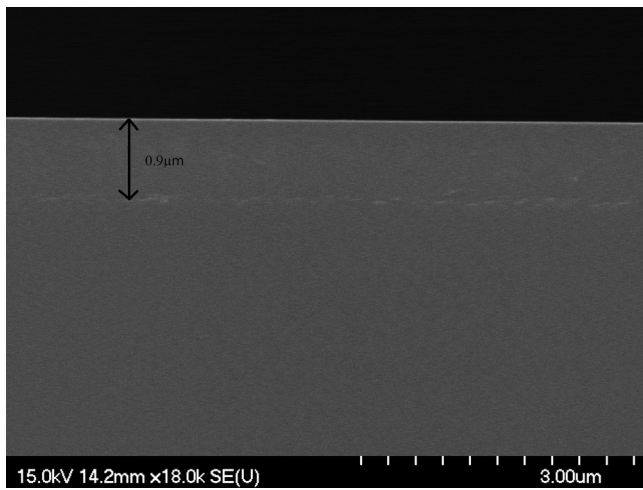


Fig. 1. The sample was implanted by 100 keV H⁺ with $1 \times 10^{17} \text{ cm}^{-2}$ dose. The cross-section SEM image shows the micro-crack below the Ge surface. The depth is about 0.9 μm .

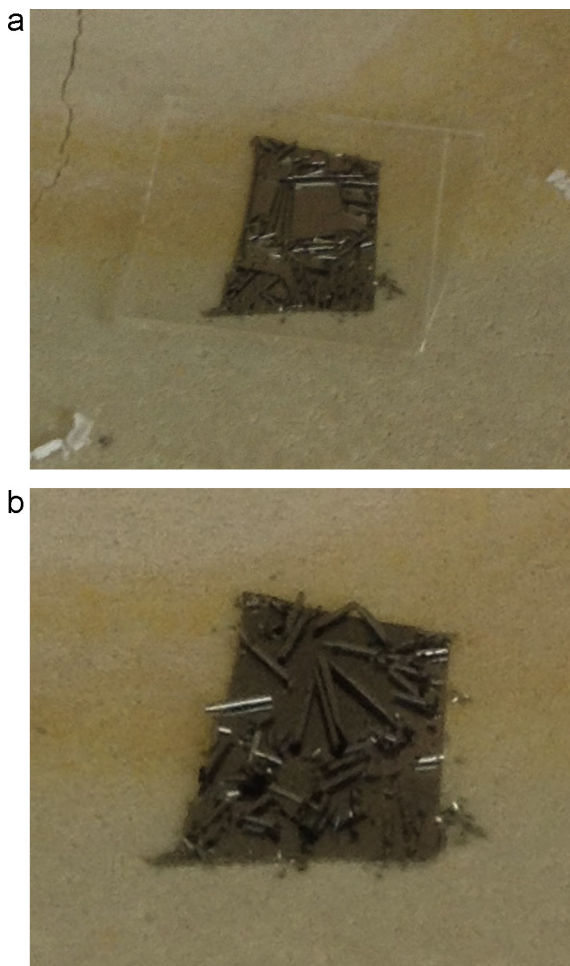


Fig. 2. Digital camera pictures of the rolled-up Ge microtubes. (a) Ge microtubes were released from the Ge substrate by thermal annealing with a glass cover on the Ge substrate that kept the Ge thin film from rolling up. (b) After removing the cover glass, the Ge thin films were self-rolled up by the strain caused by the hydrogen implantation damage.

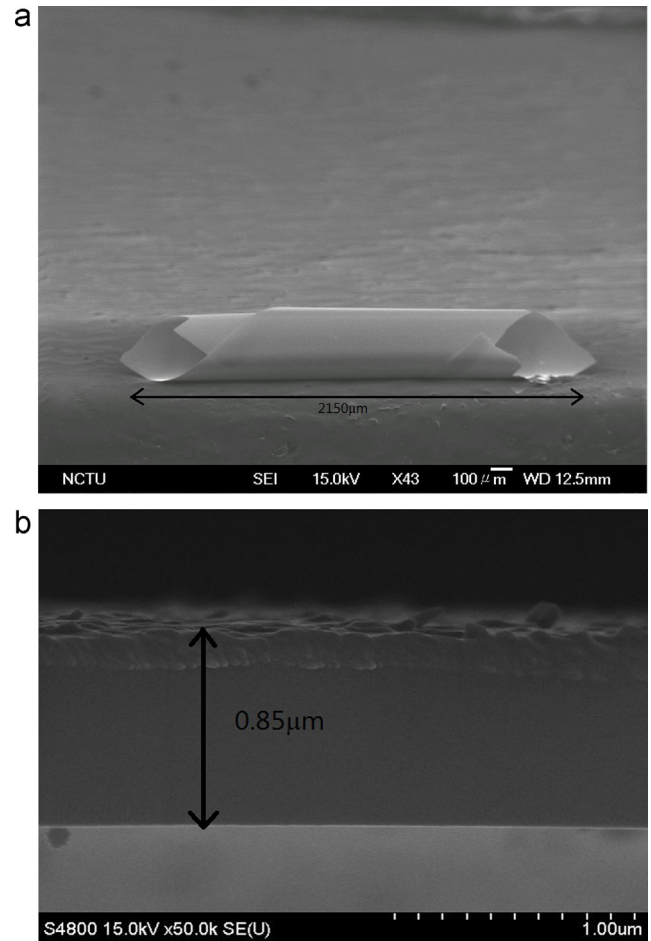


Fig. 3. (a) The SEM image shows the cross-section of the rolled-up Ge microtube. It can be seen that the cross-section is divided into upper and lower layers. The upper layer comes from the splitting of the Ge substrate, and the lower layer comes from the surface layer of Ge substrate. (b) A complete Ge microtube.

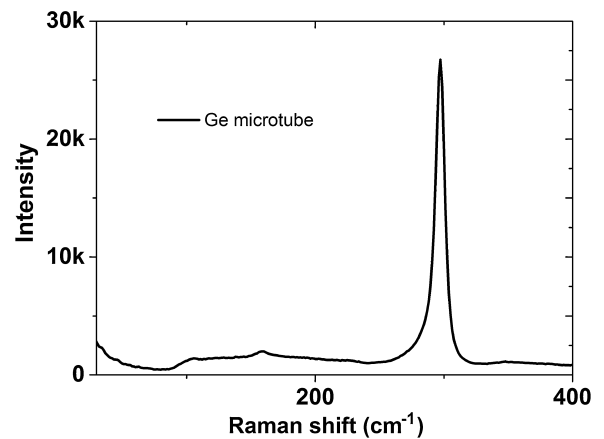


Fig. 4. The Raman scattering experiment of the Ge microtube. The Raman shift peak of the Ge microtube is located at 296.9 cm^{-1} with a FWHM 9.8 cm^{-1} whereas the Raman shift of amorphous Ge is usually located around 275 cm^{-1} .

material. Fig. 1 shows the SEM result, there were obviously cracks accumulating around the depth of $0.9 \mu\text{m}$ below the Ge surface. After applied thermal annealing, the Ge microtubes were rolled up simultaneously. Fig. 2(b) shows a digital camera picture of the self-rolled up Ge microtubes on its substrate. The sample size is around $12 \text{ mm} \times 15 \text{ mm}$. The length of Ge microtubes are from 0.5 mm to 8 mm. In order to observe the orientation of rolled-up Ge microtubes, a transparency glass was put on the sample before annealing

process. From Fig. 2(a), it can be found that there is no specific direction for rolling up Ge microtubes.

Fig. 3 shows SEM result on Ge microtube. From cross-section measurement, the thickness of Ge microtubes were measured. The thickness of Ge microtube is 0.85 μm , and the diameter of Ge microtubes are around 300 μm . In close-up, it can be seen that there exists an outer layer on the Ge microtube. This outer layer is the most damage position during hydrogen implantation. Meanwhile, we also use EDS to check the composition of Ge microtubes. Both inner and outer layers show pure Ge without oxidation. Fig. 4 shows Raman scattering spectrum on the outer layer of Ge microtube. The Raman scattering peak is located at 296.9 cm^{-1} with a 9.8 cm^{-1} FWHM. Since the single Ge Raman shift is around 299 cm^{-1} [4], it implies that the outer layer exerts a tensile strain on this layer [4] and we believe this is the reason that the Ge thin film can be rolled-up on itself. This mechanism is like the bilayer method for rolling up microtubes. Hence, from the result of Raman scattering, it also indicates that Ge–Ge bonding in these microtubes is single crystalline like.

4. Conclusions

In this study, we demonstrated a new method for making free-standing self-rolled up Ge microtubes. Detached Ge microtubes

were successfully obtained by 100 keV H^+ ion implantation with a fluence of $1 \times 10^{17} \text{cm}^{-2}$ at room temperature followed by thermal annealing. Because the processes for rolling up Ge microtubes only uses hydrogen implantation and low temperature annealing, it can be easily integrated into the MEMS device or Ge-based sensor device.

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