Investigation of Ar ion-milling rates for ultrathin single crystals

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초 록: Here we report the Ar-ion milling rates of ultrathin Si and GaAs single crystals. The thickness change is measured using convergent beam electron diffraction (CBED) technique with the help of Bloch wave simulation method. This study suggests the experimental procedures to determine the references for an etching rate to reduce a sample thickness or to remove the damaged sample surface using Ar-ion source.

1. Introduction

Focused ion beam (FIB) is widely used to prepare a transmission electron microscopy (TEM) specimen because the region of interest becomes narrow and specific. FIB system can precisely mill the specific area with a spatial accuracy of within few tens of nanometers. FIB, however, has problems such as Ga implantation and surface damage which affect the original sample structure [1]. In addition, the thickness of TEM specimens prepared by FIB is commonly thicker than Ar-ion milled TEM specimens which makes difficult to obtain high quality of structural and chemical information from electron diffraction (ED) pattern, high resolution (HR) image, atomic resolution scanning-TEM (STEM) image, electron energy loss spectroscopy (EELS), etc.

In contrast with FIB, a typical Ar-ion milling system uses much lower accelerating voltage and shows no implantation effect from Ar-ion. Furthermore, the recent Ar-ion milling systems provide rocking mode or selective sector speed thinning methods to mill TEM specimens uniformly down to few nanometers in thickness. The damage from Ar-ion can be reduced by using the low accelerating voltage of <1 kV. Based on the above, we applied the Ar-ion milling system to the ultrathin single crystals prepared by FIB in order to determine the Ar-ion milling rates of Si and GaAs single crystals.

2. Experimental procedures

For this study, we selected the single crystals of Si and GaAs. The selected specimens are first pre-thinned up to ~300 nm in thickness using FIB. In order to measure the Ar-ion milling rates, the FIB-prepared specimens were Ar-ion milled at 1.5/3 kV with a 6° incident angle for 60 and 30 seconds, respectively. All samples were attached in the center of a Cu-grid as shown in Fig. 1. The Ar-ion beam directions are indicated by the arrows. The Ar-ion milled specimens were then observed in TEM using convergent beam electron diffraction (CBED) to measure the thickness with help of Bloch simulation. For the Bloch simulation, we used the atomic scattering factors of Doyle and Turner [2], and the absorption parameters of Bird and King [3].

Figure 2(a) shows a medium magnification bright field (BF) image of Si single crystal recorded along the zone axis of [110]. The experimental CBED patterns for the Si single crystal were recorded from nine points as indicated in Fig. 2. Figures 2(b) and (c) respectively show the representative experimental CBED pattern and the corresponding simulated CBED pattern. From the simulation result, the sample thickness can be then estimated as 279 nm. We confirmed that the experimental and simulated patterns well match within the thickness range of ±5 nm. This measurement is repeated to calculate the Ar-ion milling rates for Si and GaAs ultrathin single crystals.

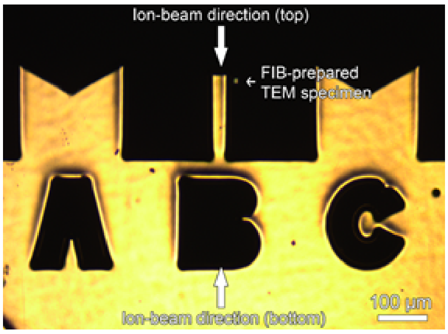

Figure 1. FIB-prepared TEM specimen mounted on a Cu-grid. The beam directions are indicated by two arrows.

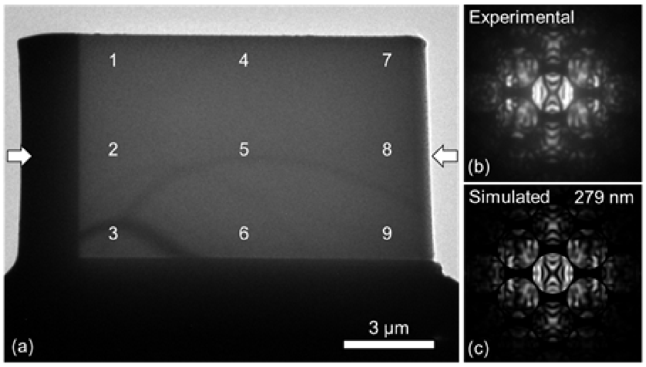

Figure 2. (a) A medium magnification bright field TEM image of Si single crystal prepared by FIB. (b) The experiment and (c) simulated CBED pattern to measure the thickness of TEM specimen.

3. Conclusion

We here measured the Ar-ion milling rates of Si and GaAs single crystals using CBED technique. The Si and GaAs single crystals are respectively Ar-ion milled by 0.58 nm/sec and 0.83 nm/sec at 1.5 kV while the milling rates are approximately doubled up to 1.13 nm/sec and 1.73 nm/sec at 3 kV (Details will be published soon).

References

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