Applied Microscopy
Korean Society of Microscopy

pISSN 2287-5123 · eISSN 2287-4445
http://dx.doi.org/10.9729/AM.2015.45.4.195

Regular Article

Preparation Method of Plan-View Transmission Electron Microscopy Specimen of the Cu Thin-Film Layer on Silicon Substrate Using the Focused Ion Beam with Gas-Assisted Etch

Ji-Soo Kim, Sang-Yeol Nam, Young-Hwan Choi[1], Ju-Cheol Park*

Gyeongbuk Science & Technology Promotion Center, Gumi Electronics & Information Technology Research Institute, Gumi 39171, Korea
[1]Department of Science of Cultural Properties, Chung-Ang University, Seoul 06974, Korea

*Correspondence to:
Park JC,
Tel: +82-54-479-1805
Fax: +82-54-479-1810
E-mail: jcpark13@geri.re.kr

Received November 4, 2015
Revised December 2, 2015
Accepted December 3, 2015

Gas-assisted etching (GAE) with focused ion beam (FIB) was applied to prepare plan-view specimens of Cu thin-layer on a silicon substrate for transmission electron microscopy (TEM). GAE using XeF_2 gas selectively etched the silicon substrate without volume loss of the Cu thin-layer. The plan-view specimen of the Cu thin film prepared by FIB milling with GAE was observed by scanning electron microscopy and C_S-corrected high-resolution TEM to estimate the size and microstructure of the TEM specimen. The GAE with FIB technique overcame various artifacts of conventional FIB milling technique such as bending, shrinking and non-uniform thickness of the TEM specimens. The Cu thin film was uniform in thickness and relatively larger in size despite of the thickness of <200 nm.

Key Words: Focused ion beam, Gas assisted etching, Xenon difluoride, Transmission electron microscopy

INTRODUCTION

Focused ion beam (FIB) has been overwhelmingly used in the semiconductor industry to perform integrated circuit failure analysis, device modification, and a variety of other applications (Kim et al., 2009). Recently, FIB milling method has been developed as a main technique for preparing transmission electron microscopy (TEM) specimen in both cross-sectional and plan-view configuration over the past 10 years by the benefit of dual-beam FIB system (Huang et al., 2005; Yu et al., 2006; Baily et al., 2013). However, during FIB milling process, a lot of artifacts such as contamination, bending, shrinking and volume loss are formed in TEM specimen (Lechner et al., 2012). These artifacts are huge obstacles to make a lamella not only larger in size but also thinner in thickness for high-resolution (HR) microscopy work. Especially these artifacts were prominent when we made a plan-view specimen of thin metal layer by conventional method of one-direction milling. So we applied the method of gas-assisted etching (GAE) using a precursor gas of the XeF_2 during FIB milling for the preparation of plan-view TEM specimen. FIB milling with GAE has many advantages like enhancement of etching rate and reduction of unintended implantation and re-deposition on a side-wall and above all, GAE can selectively etch the silicon-based materials in multi-layer structures (Fu et al., 2005; Komoda et al., 2007) and it is highly efficient method in making TEM specimen of multi-layer of thin-films. In this paper, we will suggest how to prepare the plan-view TEM specimen with selective etching of GAE using XeF_2 gas. XeF_2 is a highly reactive gas with silicon based materials like Si, SiOx and SiNx. So we applied this character of XeF_2 preparing the plan-view TEM specimen with the relatively large area of a thin film of Cu layer on silicon.

This research was financially supported by the Ministry of Education, Science Technology (MEST) and National Research Foundation of Korea (NRF) through the Management of Gyeongbuk R&D Promotion Agency.

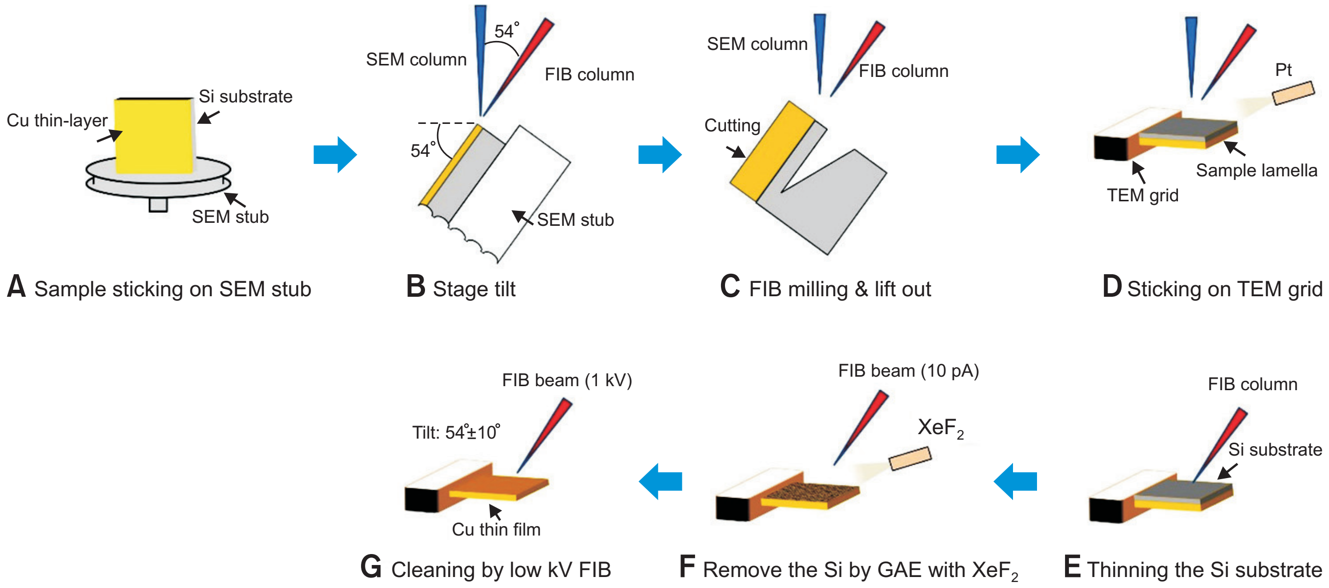

Fig. 1. The schematic diagram of the procedure making a plan-view lamella by using FIB-GAE for Cu thin-film layer on Si substrate. (A) Sample sticking on SEM stub. (B) Stage tilt. (C) FIB milling and lift out. (D) Sticking on TEM grid. (E) Thinning the Si substrate. (F) Remove the Si by GAE with XeF_2. (G) Cleaning by low kV FIB. FIB, focused ion beam; GAE, gas-assisted etching; SEM, scanning electron microscopy; TEM, transmission electron microscopy.

MATERIALS AND METHODS

The material used in this work is a Cu thin-layer which is deposited plasma enhanced chemical vapor deposition on silicon substrate (100). Sample preparation was performed in a FIB instrument (AURIGA; Carl Zeiss, Germany) incorporating a Ga liquid metal ion source and in-situ scanning electron microscopy (SEM) imaging using a thermal field emission source. It has five gas-injection system installed in chamber. Those gases are Pt, C, XeF_2, $MgSO_4 \cdot 7H_2O$ and SiO_2 and there is contained in sealed reservoirs in solid state at room temperature. We used XeF_2 at melting point of 129°C for this study. During operating, the gas was injected into the chamber through a nozzle. The preparation of TEM specimen by the FIB lift-out method was carried out as follows: (1) Cu thin-layer on silicon substrate was stuck perpendicularly to the surface of SEM stub (Fig. 1A). (2) Sample stage was tilted to 54° to adjust in parallel to the angle between Cu thin-layer and FIB column because Carl Zeiss FIB column leaned to 54° from SEM column (Fig. 1B). Rough milling for lamella was processed to remove only the Si substrate at 2 nA of milling current and after milling the Si substrate remained over the 500 nm for easy process of lift-out (Fig. 1C). (3) The cut lamella was lifted out from the milling area of the sample and then stuck on side wall of a post in TEM grid by manipulator (Fig. 1D). A part of the Si substrate of lamella was processed to remove under 100 nm (Fig. 1E). (4) Now, the sample stage was tilted to about 0° for GAE and then the GAE carried out with jetting the XeF_2 gas at the FIB milling current of under 10 pA and scanning mode (Fig. 1F). (5) Both surfaces of lamella were cleaned by FIB at 1 to 2 kV to remove the beam damage and contaminations during made in FIB work and at the moment tilt angle was 54°±8° (Fig. 1G). The process making a TEM specimen was schematically shown in the Fig. 1. The results of prepared specimen was observed the images and diffraction patterns by TEM (C_S-corrected ARM 200CF; JEOL, Japan).

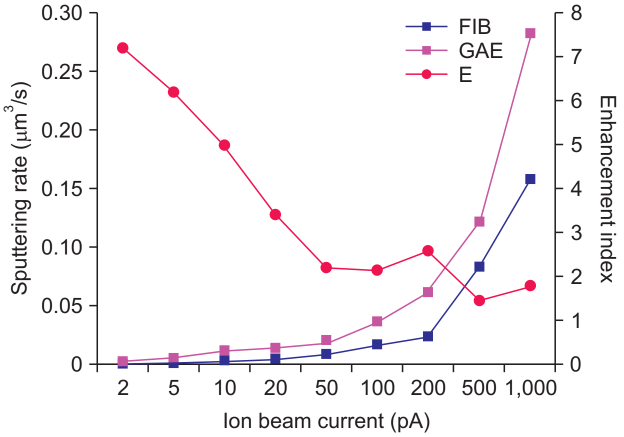

Fig. 2. Effect of the beam current on etching rate with GAE and without GAE for silicon. E is the enhancement index showing etching efficiency of XeF_2 gas. FIB, focused ion beam; GAE, gas-assisted etching.

RESULTS AND DISCUSSION

To evaluate the milling efficiency with GAE and without GAE, we measured the sputtering rates of silicon for both methods. Fig. 2 shows the compared data of the sputtering rate of silicon with GAE and without GAE according to the change of the FIB beam current from 1 to 1,000 pA. As a result, in the case of FIB milling with XeF_2 GAE, the sputtering rate is on average about three times higher than that of only FIB milling for which only physical sputtering operates. Each sputtering rate was determined by dividing the etched volume by the etching time and has unit of μm^3/sec. The width and height length used in the experiments was 5 μm and 5 μm, respectively and etching time was 30 seconds. The conditions were applied to all experiments in the same manner. The etched depth was measured according to change the beam current of Ga^+ ion at the same etching area by using the FIB-SEM. And each sputtering rate was calculated by measuring the etched depth. It is expressed by the formula shown below.

$$\text{Sputtering rate}=\{\text{width }(\mu m)\times\text{height }(\mu m)\times\text{depth }(\mu m)\}/\text{etch time (sec)}$$

And the ratio of the sputtering rate without GAE and sputtering rate with GAE is defined as enhancement index (E) (Fu et al., 2005).

$$E=\text{sputtering rate without GAE}/\text{sputtering rate with GAE}$$

The enhancement index increases as a beam current of FIB decreases, which indicates that at the relatively low beam current of Ga^+ ion, the sputtering by the reaction between XeF_2 gas and silicon is more effective than at the higher beam current of Ga^+ ion.

In addition, FIB with XeF_2 GAE can selectively etch only silicon-based layers in multi-layer sample because the reaction of XeF_2 with specified silicon materials is very selective. As a result, researchers can obtain a plan-view metal layer like Cu from the multi-layer sample due to the very lower reactivity of copper with XeF_2. In the case of this study, the reaction formula is expressed as (Nan Yao, 2007).

$$Ga^+ + XeF_2(g) + Si(g) + Cu(s) \rightarrow Ga^+ + Xe(g) + SiF_2(g) + Cu(s)$$

Fig. 3 shows SEM and TEM images of a plan-view Cu lamella obtained by FIB-GAE with XeF_2. The initial thickness of the Cu layer was measured as about 200 nm by the cross-sectional analysis of FIB. The SEM and bright-field TEM image shows the overview of the prepared lamella. The horizontal and vertical length of size of the lamella is measured as 6.0 μm

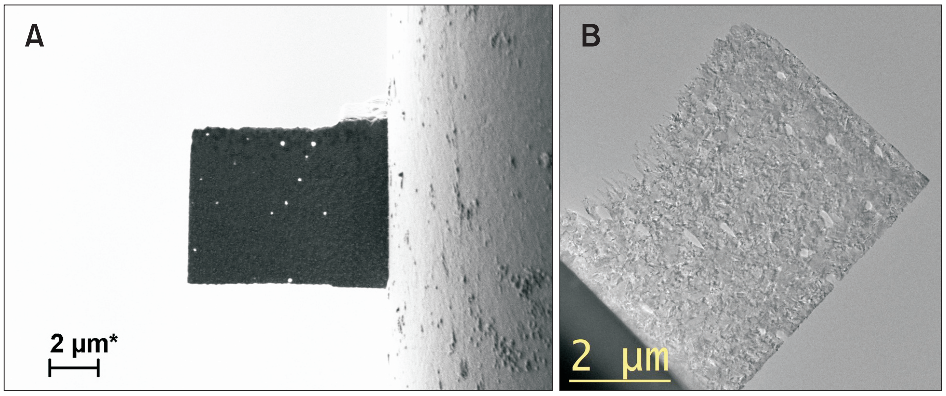

Fig. 3. (A) Scanning electron microscopy image of a plan-view specimen of Cu layer obtained by focused ion beam (FIB) etching assisted with XeF_2 gas. (B) Transmission electron microscopy image after low kV cleaning by only FIB. Notice that the uniform thickness of the lamella.

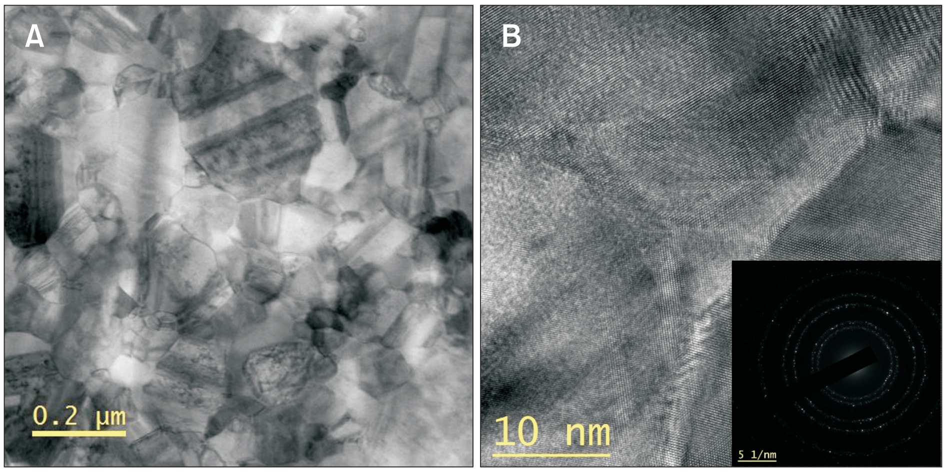

Fig. 4. (A) Plan-view scanning transmission electron microscopy bright-field image of Cu thin-film layer. (B) High-resolution image and diffraction pattern of Cu grain.

and 6.5 μm. It is observed that the lamella has no bending and shrinking artifacts and its thickness was uniform. For precise TEM analysis, it is important to make the lamella as large as possible. The reason of different shape between Fig. 3 is the volume loss of lamella after low kV cleaning in FIB system, cleaning was performed Ga ions at 1 kV but this allowed the optimum thickness of TEM analysis. Low kV cleaning at the FIB system by Ga ions is useful to remove the remained substrate, re-deposition of etched materials and else contaminations besides the thinning the thickness of lamella. Fig. 4 shows the results of TEM observation of the lamella. Fig. 4A is scanning TEM bright-field image and Fig. 4B is the HR-TEM image and diffraction pattern of poly Cu grain. Fig. 4A shows that the Cu layer is composed of polycrystalline grains and many defects such as stacking faults and dislocations and clear grain boundaries are visible. HR-TEM images in Fig. 4B shows the lattice fringes in each grain and clear rings in the diffraction pattern. As a result, it is confirmed that FIB with GAE method can provide the proper plan-view specimen which has a thin and uniform thickness and is wide enough to be used for sophisticated TEM analysis.

CONCLUSIONS

We proposed a new preparation method for a plan-view specimen of TEM lamella by using FIB-GAE technique. The lamellae were made by FIB with XeF_2 gas assisted etching. We applied the selective etching effect to the sample of Cu thin-layer on silicon substrate. GAE with XeF_2 carried out selective etching of only silicon materials in the sample with enhanced etch rate. Silicon substrate was selectively removed by XeF_2 gas and only Cu thin layer remained in the TEM grid. The shape, thickness and uniformity of the prepared TEM sample were evaluated through the HR-TEM image and diffraction pattern by SEM and TEM analysis. Thus, it is suggested that the FIB with GAE method can be a useful method to make plan-view specimens of metal thin-film layers with good quality for TEM analysis.

CONFLICT OF INTEREST

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

REFERENCES

Bailey R, Geurts R, Stokes D J, Jong F D, and Barber H A (2013) Evaluating focused ion beam induced damage in soft materials. *Micron* **50**, 51-56.

Fu X L, Li P G, Jin A Z, Chen L M, Yang H F, and Cui Z (2005) Gas-assisted etching of niobium with focused ion beam. *Microelectronic Engineering* **78-79**, 29-33.

Huang Y, Cockayne D J H, Marsh C, Titchmarsh J M, and Petford-Long A K (2005) Self-organized amorphous material in silicon (001) by focused ion beam (FIB) system. *Applied Surface Science* **252**, 1954-1958.

Kim J S, Lee SY, Lee LS, and Kim J Y (2009) Three dimensional reconstruction of structural defect of thin film transistor device by using dual-beam focused ion beam and scanning electron microscopy. *Korean J. Microscopy* **39**, 349-354.

Komoda H, Moritani C, Takahashi K, Watanabe H, and Tasutake K (2007) Sample tilting technique for preventing electrostatic discharge during high-current FIB gas-assisted etching with XeF2. *Microelectronics Reliability* **47**, 74-81.

Lechner L, Biskupek J, and Kaiser U (2012) Improved focused ion beam target preparation of (S)TEM specimen-a method for obtaining ultrathin lamellae. *Microsc. Microanal.* **18**, 1-6.

Nan Yao (2007) *Focused Ion Beam System* (Cambridge University Press, New York).

Yu J, Liu J, Zhang J, and Wu J (2006) TEM investigation of FIB induced damages in preparation of metal material TEM specimens by FIB. *Materials Letters* **60**, 206-209.