

The Shave-off Depth Profiling by the Nano-Beam SIMS

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The shave-off depth profiling achieves the highly precise depth profiling with nanometer-dimensional depth resolution. This method is a very unique depth profiling for acquiring a depth profile by the shave-off scanning mode (A fast horizontal sweep of an FIB is combined with a very slow vertical sweep). The FIB of dynamic condition shaves completely off the sample from the surface to the depth direction. This method can be advanced in its flexibility and applicability by an introduction of the lift-out method which conventionally used as a sample preparation tool for TEM. Combining the lift-out method with the shave-off depth profiling, the highly precise depth profiling of the selected 3D volume in the bulk sample has been realized with nanometer-dimensional depth resolution. And we also discussed the effect of a protection film in order to keep on sharp cutting edge.

INTRODUCTION

Recent devices with fine and complex multi-structures have been actively developing. For the advances of their devices, it is necessary to obtain the depth information with high depth resolution and wide depth range. Today there are some depth profilers with nano-dimensional resolution. These profilers have different applications and analytical features (Fig.1). For example, Atom Probe (AP) and ultra shallow depth profiling are kinds of depth profilers with nano-dimensional depth resolution [1] [2]. However, these profilers cannot perform highly accurate depth profiling from the surface to the buried bulk structure continuously in the selected local area.

The scanning Secondary Ions Mass Spectrometry (SIMS) combined with Focused Ion Beam (FIB) can perform lateral elemental mapping with nano-dimensional resolution [3]. Utilizing the capability of micro-machining with the FIB, a new depth profiling method has been invented: the shave-off depth profiling [4]. This method is a very

unique depth profiling for acquiring a depth profile by the shave-off scanning mode (A fast horizontal sweep of an FIB is combined with a very slow vertical sweep). The FIB of dynamic conditions shaves completely off the sample from the surface to the depth direction. The shave-off depth profiling can minimize the effects of surface roughening, atomic mixing, and sputtered deposition.

Flexibility and applicability in the shave-off depth profiling was restricted by the shape or size of a sample, because of a large amount of time for sample preparation using only FIB micro-machining process. In this paper, we have advanced the shave-off depth profiling by an introduction of a lift-out method and discuss the effect of a protection film in order to keep on sharp cutting edge. The lift-out method was first developed for TEM sample preparation [5]. By using this method, a piece of the sample can be lifted up from the bulk sample without destroying the structure of the sample of interest and cleaving the substrate.

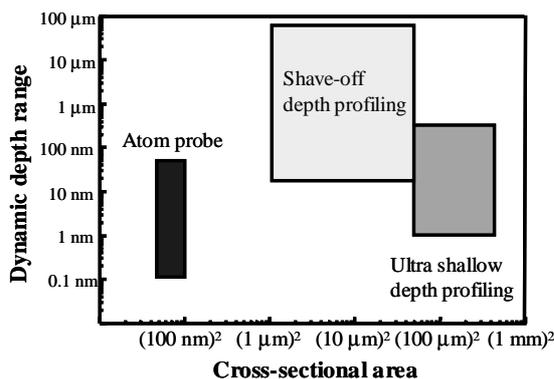


Fig. 1 Analytical feature of typical depth profilers

EXPERIMENTAL METHOD

Sample preparation using the lift-out method

The lift-out method adopted in this study was accomplished using SMI3050 (SIINT) focused ion beam microscope. The lift-out procedure is shown in Fig. 2. The sample is a multi-layer thin film (Al 1 μm / SiO₂ 0.8 μm / Si substrate). First, in order to protect the region of interest from sputtering, the sample surface is covered with 0.5 μm thick carbon coat and the FIB machines the surrounding region of interest, as shown in Fig. 2 (a). Second, the sample is tilted and the bottom of the piece is cut, as shown in Fig. 2 (b). Third, tungsten probe is attached to a piece of the sample by carbon deposition and the remaining side is cut by the FIB, as shown in Fig. 2 (c). Fourth, a piece of the sample is fixed with carbon deposition on the substrate and the extra carbon deposition is cut, as shown in Fig. 2 (d, e).

The shave-off depth profiling

A piece of the sample which was prepared using the lift-out method was analyzed by the shave-off depth profiling with the nano-beam SIMS [6]. The nano-beam SIMS which aimed for nano-dimensional analysis was developed by our group. Primary ions of this apparatus are field-emitted Ga⁺ with 30 keV acceleration energy. The mass analyzer is a modified

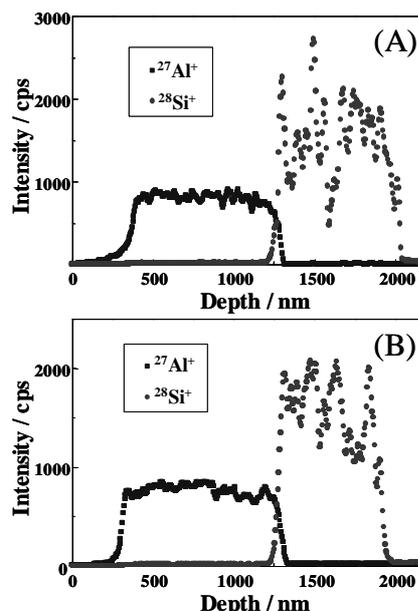


Fig. 3 The shave-off depth profiles
(A) Without protection film
(B) With a protection film

spark source mass spectrometer (JEOL JMS01BM) of a Mattauch-Herzog type geometry. This apparatus realizes a nano-scale ion beam and simultaneous multi-elemental detection. The condition of this shave-off depth analysis was as follows: 6.5 kV sample voltage, 35 pA beam current, (1×6) μm² surface area of a piece of the sample.

A protection film

It is known that the FIB has long tails in the profile of the beam by the cause of spherical aberrations, or transverse thermal velocity effects [7]. M. Nojima et al. showed in details beam profile of nano-beam SIMS [8]. In the shave-off depth profiling, the sample is shaved off by the edge, namely the outer part of the FIB. Therefore, it is concerned that the long tails affect the shape of profile and make depth resolution worse. When the side wall of the sample which is faced to the axis of primary ion beam is covered with a protection film, the sample is

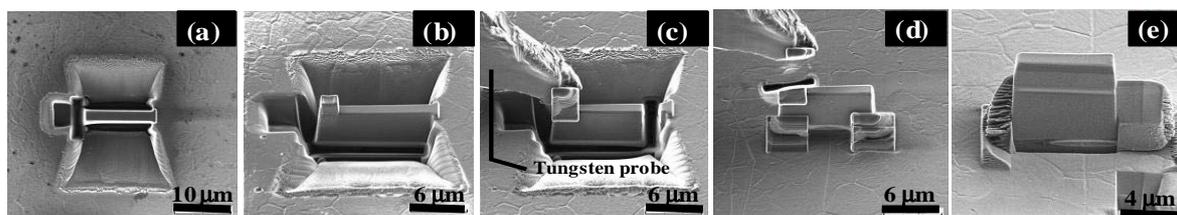


Fig.2 The lift-out procedure

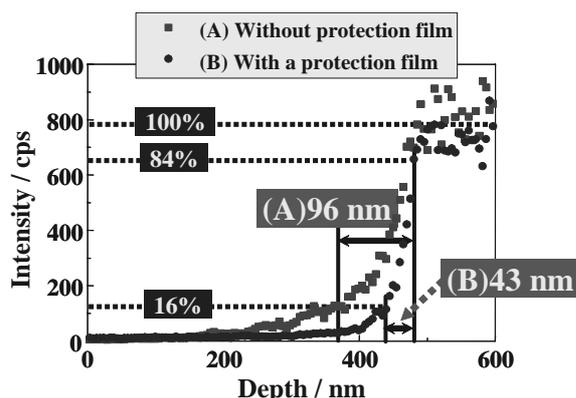


Fig. 4 Comparison of rising edges of $^{27}\text{Al}^+$ signals

partially protected from sputtering by long leading tails of the beam profile of the FIB. We deposited a protection film (0.1 μm carbon deposition film) on the upper side wall of a piece of the sample which was prepared using the lift-out method.

RESULTS AND DISCUSSION

Figure 3 shows the shave-off depth profiles. The upper side wall of the sample of (B) was covered with a protection film of 0.1 μm thick. Surface areas (cross-sectional areas at shave-off analysis) of the samples were 1 $\mu\text{m} \times 6 \mu\text{m}$. Dispersion of data points is mainly due to local charge-up caused by a SiO_2 insulation layer. The depth profile of (B) shows sharper increase and decrease in comparison with (A). Figure 4 shows comparison of rising edges of $^{27}\text{Al}^+$ signals of (A) and (B). We define the depth resolution as the distance between depths with 16 % and 84 % of the secondary ion intensity, respectively. According to this definition, depth resolutions at these two edges were estimated to be 96 nm and 43 nm for (A) and (B), respectively. The observed depth resolution in shave-off depth profiling is also affected by the sample alignment error with the horizontal scanning direction (rotation error) and beam profile and the protection film (discussing in this paper) [9]. Depth profiles (A) and (B) were measured under the same beam condition. Difference in the rotation errors in both measurements was within 0.1 degree, which introduced resolution deterioration of 10 nm at maximum. Although the absolute value of the tilt errors could not be estimated, because tilt angle could

not be adjusted under secondary electron or ion image observation, the maximum difference in tilt angles between two samples was limited by the mechanical precision of the sample stage in the FIB microscope used for lifting-out. If this difference is assumed to be 0.5 degree, the tilt error difference is estimated to be less than 9 nm between these profiles. Observed difference on each resolution between these two profiles were 53 nm, which cannot be explained by the sample alignment error. Therefore, deposition of the protection film is the very effective method in order to improve the depth resolution.

CONCLUSION

We have advanced the shave-off depth profiling by the introduction of the lift-out method. As a result, depth profiling of the selected 3D volume in the bulk sample is obtained by the shave-off depth profiling. A protection film on one side wall of the sample can avoid the influence on the shave-off depth profile from long leading tails in the beam profile of the FIB, resulting in better depth resolution.

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REFERENCES

- [1] M. Schuhmacher, B. Rasser and F. Desse, *J. Vac. Sci. Technol.*, **B 18** (1), 529 (2000).
- [2] D. Blavette, E. Cadel and B. Deconihout, *Mat. Cha.* **44** (1-2), 133 (2000).
- [3] M. Nojima, M. Toi, A. Maekawa, B. Tomiyasu, T. Sakamoto, M. Owari and Y. Nihei, *e-J. Surf. Sci. Nanotech.*, **2**, 131 (2004).
- [4] F. A. Stevie, C. B. Vartuli, L. A. Giannuzzi, T. L. Shofner, S. R. Brown, B. Rossie, F. Hillion, R. H. Mills, M. Antonell, R. B. Irwin and B. M. Purcell, *Surf. Interface Anal.*, **31**, 345 (2001).

- [5] H. Satoh, M. Owari, Y. Nihei, *J. Vac. Sci. Technol.*, **B 6 (3)**, 915 (1988).
- [6] M. Nojima, B. Tomiyasu, Y. Kanda, M. Owari, Y.Nihei, *Appl. Surf. Sci.*, **203/204**, 194 (2003).
- [7] J. W. Ward, R. L. Kubena, M. W. Utlaut, *J. Vac. Sci. Technol.*, **B 6**, 2090 (1988).
- [8] M. Nojima, M. Toi, A. Maekawa, B. Tomiyasu, T. Sakamoto, M. Owari, Y. Nihei, *Appl. Surf. Sci.*, **231-232**, 930 (2004).
- [9] B.Tomiyasu, S. Sakasegawa, T. Toba, M. Owari, Y. Nihei, *HYOUMEN KAGAKU*, **20 (8)**, 523 (1999).