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Technical Report

Ion Beam Alignment Procedures using a Faraday Cup or a Silicon Dioxide Film on Silicon Substrate with Auger Electron Microscope

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In surface analysis such as AES (Auger electron spectroscopy) or XPS (x-ray photoelectron spectroscopy), ion sputtering is generally used in order to remove contaminated layers and to perform an in-depth profiling. It is important to align an ion beam at an analysis area and to estimate sputtering rates prior to an actual measurement. In this report, two methods are introduced how to align the ion beam. (a) Faraday cup method is applicable to quantitatively estimate the ion beam by monitoring current and (b) SiO_2 method is an easy way to visually align the ion beam position. Detailed alignment procedures are promised to be useful for daily analysis workers.

1. Introduction

Electron spectroscopy such as AES (Auger electron spectroscopy) and XPS (x-ray photoelectron spectroscopy) are very useful to evaluate elemental compositions and chemical states in several nm surface regions in depth. They have been widely used in a research and development phase and failure analyses in industry field. Ion sputtering by rare gases is generally adopted to remove a contaminated layer and to measure an in-depth profiling. Here are parameters such like an ion accelerating voltage, an ion current density, an ion species, and an incident angle to a specimen in ion sputtering procedure, and these parameters affect a sputter etching rate, surface roughening, and elemental mixing [1]. Mathieu pointed out surface roughening caused by its current density distribution in the cross-section of an ion beam [2]. It is needed for surface analysis with ion sputtering to flatten the central area of an ion beam rasterizing region in order to avoid morphological effects [3]. It is also important that an analysis area should be located at the central region of an ion beam irradiation area. These configurations are shown in Fig. 1. A sputtering rate should be also known by an analyst as a function of the parameters.

Two main subjects are in the procedure of ion beam alignment; one is that an analysis area is placed within the central region of an ion beam irradiation area and the other is adjustment of an appropriate ion beam current density with varying rasterizing size. When using a Faraday cup, one can qualitatively set an ion beam because of measuring a current for a specified area that corresponds to a hole on a Faraday cup. An accurate

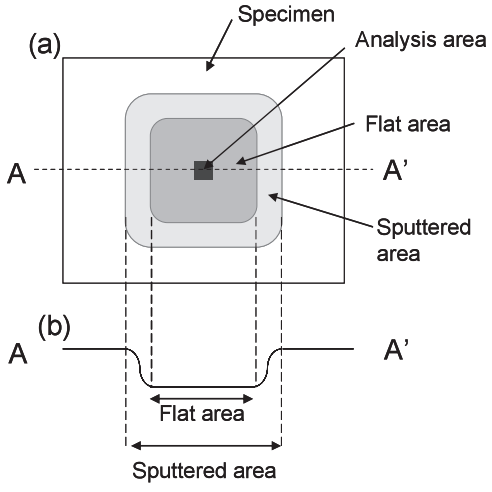

Fig. 1. Configurations of sputtered, flat and analysis area. (a) Top view; (b) Cross-section view of along the line A - A'.

measurement of ion beam current using a Faraday cup was discussed by Seah *et al.* [4] and a measurement technique of an ion current distribution was studied by Inoue *et al.* [5] using a kind of Faraday cup with a sophisticated design. On the other hand, when using a SiO_2 film, one can visually control an ion beam position and a rasterizing condition. In this report we introduce the methods that can be applied even by a beginning instrument operator using a conventional Faraday cup and an easy-available SiO_2 film.

2. Experimental

The instrument used was PHI700 Auger Nanoprobe™ from ULVAC-PHI. The floating type ion gun (model 06-350) was mounted on the instrument and an ion accelerating voltage and a floating voltage are variable in the rage of 1 V to 5000 V and 0 to 500 V, respectively [6]. Figure 2 shows the cross-sectional design (a) and the external view (b). There is a hole of 250 μm in diameter on the top of the Faraday cup holder as shown in Fig. 2(c) on the outer electrode and there is a trapezoidal hole of 5.3 mm in depth in the inner electrode that is electrically insulated from the outer one. Two electric lines are wired from the sample stage in a vacuum to the outside of the analysis chamber and they enable current measurements from the outer electrode and the inner electrode, independently. The current from the latter electrode is the ion beam current through the 250-μm-hole and that from the former one corresponds to the total ion beam current except for the part of 250 μm diameters. The bias voltage of +90 V is applied to the inner electrode when measuring the ion current in order to suppress current increase caused by secondary electrons generated by ions. The current is measured with a Keithley 6485 picoam-

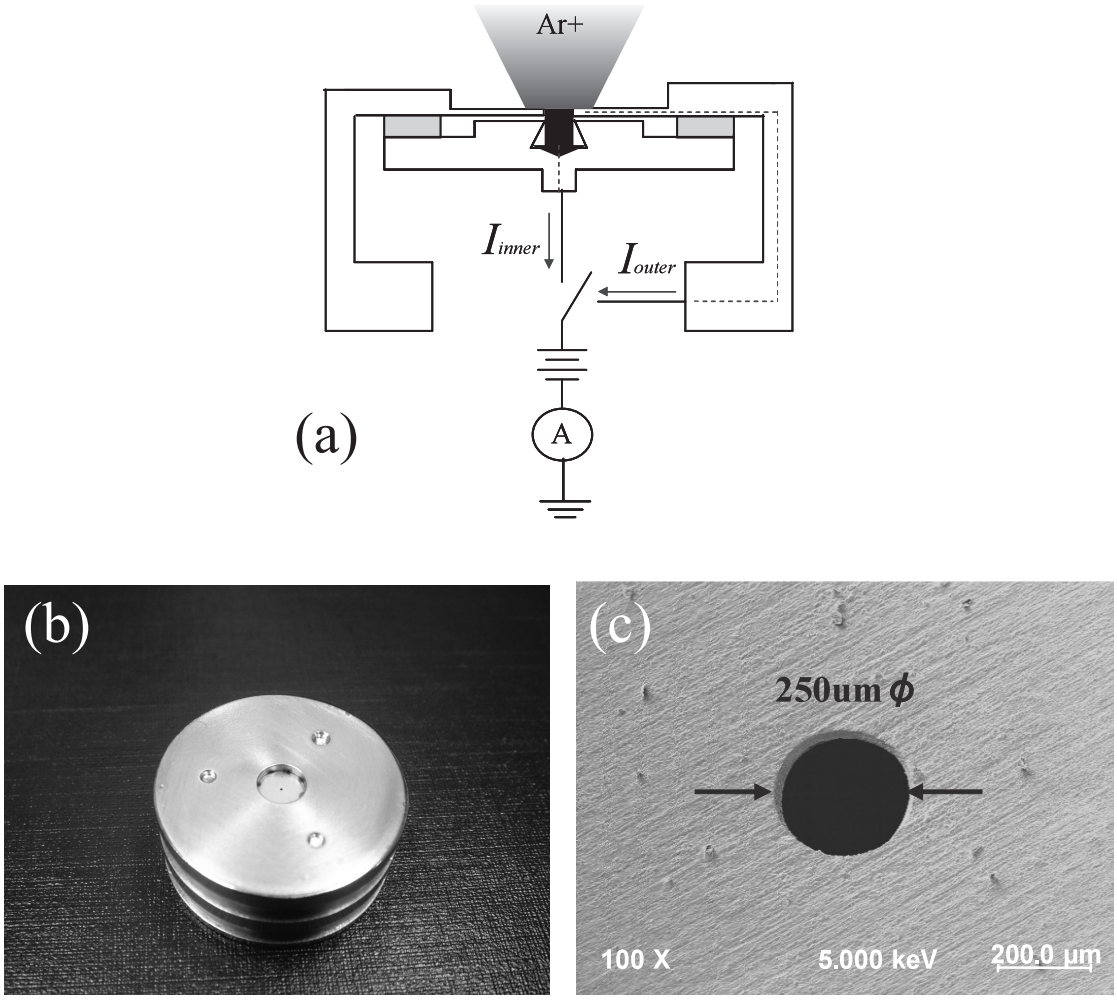

Fig. 2. The appearance and the cross-sectional design of Faraday cup.(a) Cross-section view. When one electric line is switched, the other line is grounded. I*inner* and I*outer* correspond to the current from the inner and outer electrode, respectively. (b) A photo of the Faraday cup. (c) SE image of the center of Faraday cup with a hole of 250 μm in diameter.

meter for the ion beam that is swept at the rate of about 1 kHz along the x-axis and 10 Hz along the y-axis. For the second subject a 105 nm thick thermally oxidized SiO_2 film on Si wafer was applied.

3. Example of in-depth profile using miss-aligned ion beam at a crater edge

The depth profiles of GaAs/AlAs superlattice are shown in Fig. 3 where one [Fig. 3 (a)] of them is the result measured under the conditions of suitable alignment of the ion beam and the analyzed position and the other [Fig. 3(b)] is the result obtained intentionally at the crater edge of rasterizing ion beam. Sputtering was carried out by Ar^+ ions of the accelerating voltage of 1 kV, the incident angle of 45 degrees, the raster area of 1.5 mm × 1.5 mm, and the analysis area of 50 μm × 50 μm. The both depth profiles are almost same in the region of surface. Increasing the depth by ion sputtering, degrading the interface resolution as well as decreasing the amplitude (corresponding to the intensity) of the signal for the miss-aligned case. The time durations from the topmost surface to the substrate were 25 minutes and 39 minutes, respectively. The sputtering rate was about 36% less for the miss-aligned depth profiling obtained at the crater edge than that for well-aligned profiling at the center of the crater.

4. Faraday cup method

Schematic views are shown in Fig. 4 for ion beam alignment using a Faraday cup. As the ion beam is aligned at the hole position of the Faraday cup, the ion beam current is greater when focusing thinner than that when broader, comparing Fig. 4(a) and 4(b). Even the ion beam is perfectly focused, if the position of ion beam is shifted from the faraday cup hole as shown in Fig. 4(c), the ion beam current becomes lower. It is need that (a) the Faraday cup hole position should be at the analyzed position, (b) the ion current should be optimized to get an

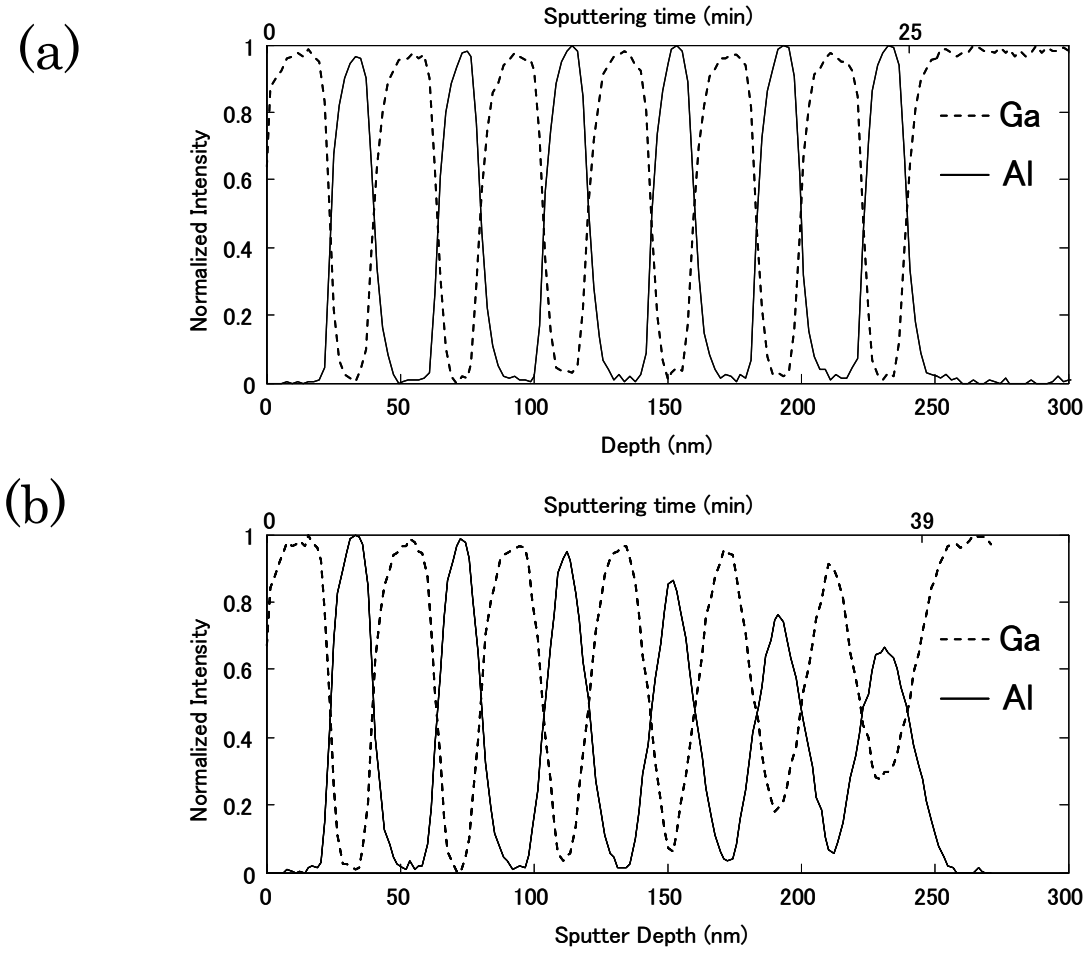

Fig. 3. Comparison of AES depth profiles of GaAs/AlAs superlattice under proper and improper ion beam alignments. (a) under proper ion beam alignment. (b) under improper ion beam alignment.

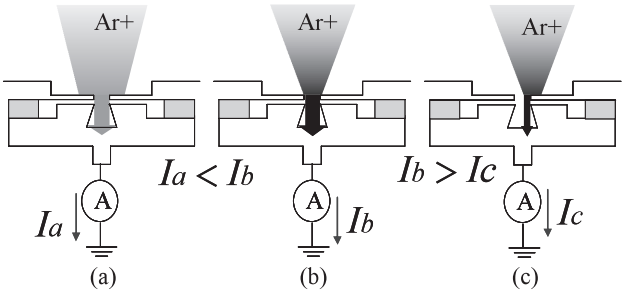

Fig. 4. Schematic view for ion beam alignment using a Faraday cup. I_a, I_b and I_c are the ion currents through the Faraday cup hole in the figures (a) to (c). (a) not just focused ion beam. (b) a focused ion beam and proper irradiation location. (c) a focused ion beam, but improper irradiation location.

appropriate sputtering rate, and (c) the ion beam should be focused to get a highest current at the inner electrode. Doing these alignment procedures, the analyzed point is placed at the central region of the sputter crater when the ion beam is swept and the analysis is performed at the evenly sputtered area. This method is applicable for AES and XPS.

To summarize the alignment procedure using the Faraday cup.

1. to align the Faraday cup hole to the analyzed position using SEI (secondary electron image) or OMI (optical microscope image).
2. to turn off the accelerating voltage of electron beam in order to avoid an affect of primary electron beam to the measured current in the case of AES.
3. to turn off SED (secondary electron detector) multiplier voltage because of the protection of the detector since an current of ions is generally significantly greater than that of electrons.
4. to turn on the ion beam.
5. to note that the ion beam shall not be rasterized.
6. to adjust raster offsets to get a maximum ion current at the Faraday cup.
7. to adjust objective lens parameters to get an maximum ion current at the Faraday cup.
8. to repeat 6 and 7.
9. to adjust condenser lens parameters to get an appropriate ion current at the Faraday cup or the target (corresponding to the outer electrode).
10. to go back to the procedure 8.
11. to repeat 9 and 10.
12. to set raster parameters for actual measurements.
13. to turn off the ion beam.
14. to record or save the ion beam parameters.

The ion beam irradiated onto the specimen surface is varied by focusing or defocusing the ion beam at the objective aperture by the condenser lens for the ion gun used in this study. This is the reason the condenser lens parameters are changed in order to get an appropriate ion current. Figure 5 shows the Faraday cup ion current at the accelerating voltage of 2 kV when changing the con-

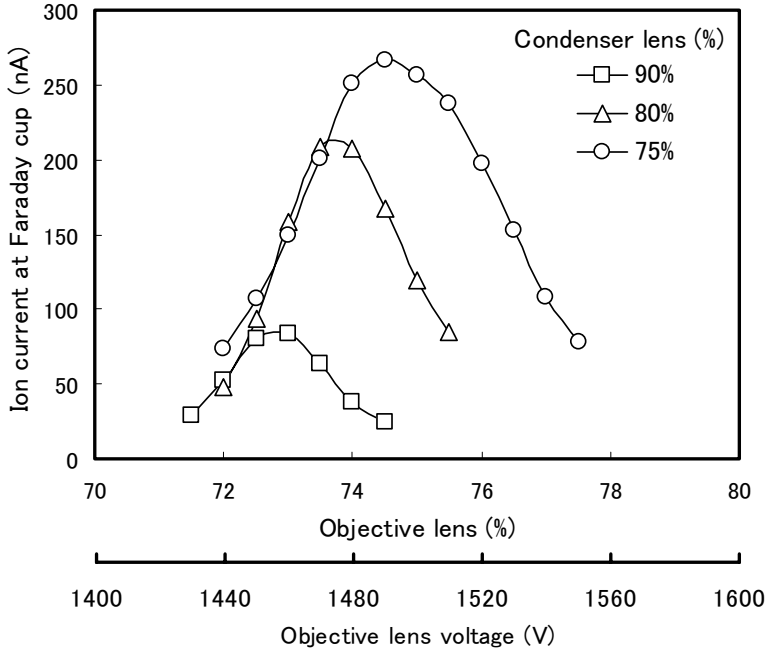

Fig. 5. Faraday cup ion current at the accelerating voltage of 2 kV when changing the condenser lens and the objective lens parameters. These parameters are shown voltage ratio (%) of the acceleration voltage. The values are the percentage by 2000 V.

denser and the objective lens parameters. The maximum position of each curve corresponds to the condition that on ion beam is best focused. The parameter of condenser lens and the objective lens is shown the voltage ratio to the acceleration voltage in percentage. It is found that the optimum value of the objective lens depends on the ion current determined by the condenser lens parameter.

Figure 6 shows the ion currents at the Faraday cup against the raster area, and the estimated sputtering rates that are calculated from the measured sputtering rate at the raster size of 2.25 mm^2. Increasing the raster dimension, the current decreases because ions going through the Faraday cup hole decrease per unit of time. Once the sputtering rate is measured for a reference specimen under the specified condition, one can estimate the sputtering rate since the sputtering rate is proportional to the ion beam current density. It is found that the estimated sputtering rate is in good agreement with the actually measured one for the raster area of 4 mm^2.

5. SiO_2 method

The alignment procedure using SiO_2 film is shown in Fig. 7, where the primary electron beam of the accelerating voltage of 3 kV is co-irradiated with the ion beam. The part of dark contrast corresponds to the region irradiated by ion beam in SEI. This method is not suitable for the cases of thick (large diameter), high current or low voltage ion beam. This is because it is hard to adjust the focus of the ion beam when the ion beam is too thick in the field of view of SE image. Therefore when the

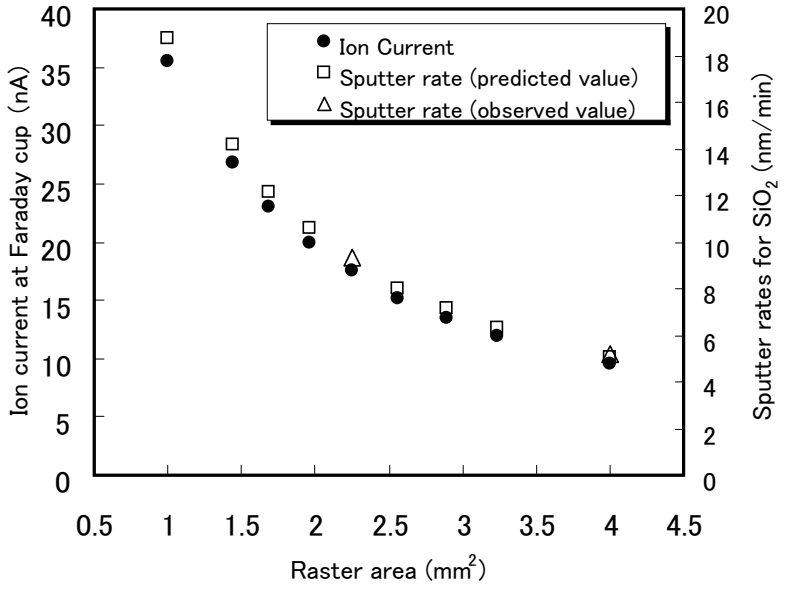

Fig. 6. Example of the ion current depending on raster sizes and sputtering rates for SiO_2 at 1 kV.

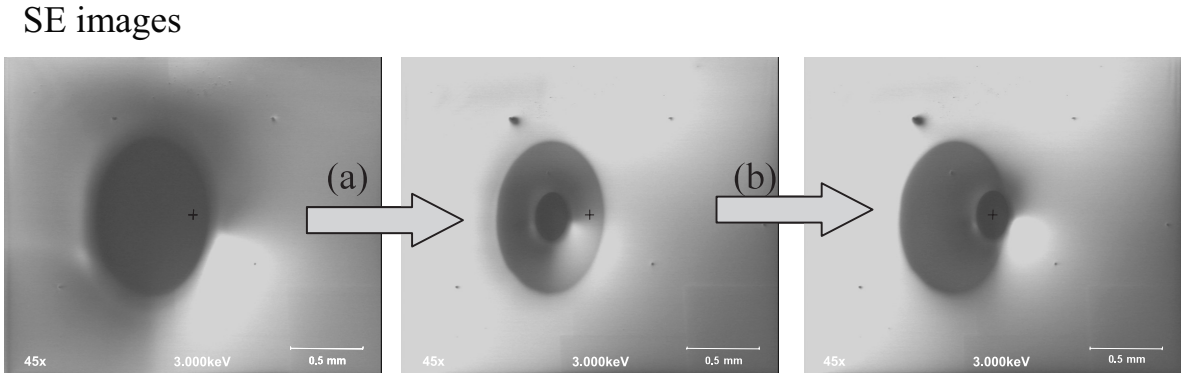

Fig. 7. A part of the alignment procedure using SiO_2 film.

SiO_2 method is applied, it is recommended that the ion beam should be thinner by decreasing the ion current, for instance, by changing the condenser lens parameter from "75%" to "90%" as shown in Fig. 5. Decreasing the ion current, it is easier to recognize the ion beam position and to focus the ion beam. Note that the optimum objective lens condition changes when increasing the ion current. Thus the initial conditions of condenser lens and objective lens parameters should be applied after adjustment of ion beam position for the low current ion beam. Here the ion beam shall be roughly aligned under the initial conditions. Though it is difficult to quantitatively measure the ion current and to exactly focus an ion beam, it is very convenient to adjust the irradiation position of ion beam because it is easy to recognize the ion beam visually. When applying this procedure on the SiO_2 that thickness is known, one can estimate the reference sputtering rate without exchanging the specimen.

To summarize the alignment procedure using the SiO_2 film.

1. to place a SiO_2 film of about 100 nm thick at the analyzed position.
2. to set the electron beam , typically at 3 kV and about 10 nA.
3. to set lower voltage for SED multiplier.
4. to turn on the ion beam.
5. to set the condenser lens parameters at 90 to 100 % of the maximum applied voltage, leading to easy recognition.
6. to set the ion beam raster size as 0 × 0.
7. to adjust SEM conditions to recognize the ion beam irradiation position.
8. to adjust the objective lens parameters to get the ion beam irradiation as thin as possible.
9. to adjust the raster offsets that the ion beam irradiation places at the center of SEI.
10. to set the parameters of condenser lens, objective lens, and raster size for actual use.
11. to turn off the ion beam.

6. Summary

As an example of a miss-alignment of an ion beam, it is shown that depth resolutions are remarkably degraded in a deeper region under improper sputtering conditions. Two alignment procedures are introduced in detail. One is (a) Faraday cup method that is applicable to quantitatively estimate and the other is (b) SiO_2 method is an easy way to visually check the ion beam position and the sputtering rate. We expect this technical report is useful for daily work of AES/XPS users.

7. References

[1] for example, S. Hofmann, *Surf. Interface Anal.* **9**, 3 (1986).
[2] H. J. Mathieu, Chapter 3 in *Thin Film and Depth Profile Analysis*, Springer-Verlang, ed. by H. Oechsner (1984).
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[4] G. D. Ingram and M. P. Seah, *J. Phys. E: Sci. Instrum.* **22**, 242 (1989).
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[6] H. Iwai, H. Namba, T. Morohashi, R. E. Megri, A. Ogata, T. Hoshi, and R. Oiwa, *J. Surf. Anal.* **5**, 161 (1999).

査読コメント

査読者 1．荻原俊弥（NIMS）

ユーザーにとって，実用的に役立つ内容の論文であり，掲載可です.

［査読者 1-1］

「3. Example of in-depth profile using miss-aligned ion beam」の項目で，2 つ質問（コメント）です.

［査読者 1-1-1］

一つは，Fig. 3(b)のデプスプロファイルは，クレーターエッジの部分を測定したものですが，それを miss-aligned ion beam と表現するのは，よいのでしょうか？miss-aligned ion beam とは，スポットイオンビームの調整が悪く，例えば，同心円状にスパッタされなく，いびつな形状のものであり，そのビームを用いてラスターさせた場合と考えます．miss-aligned ion beam では，もっと，深さ分解能が悪いデプスになるように思いますが.

［著者］

読者がビーム調整が悪い場合と混同しないように"at a crater edge"と表題に，節最後の文章に追記いたしました. アンダーライン部が追記した部分です。

表題：

3. Example of in-depth profile using miss-aligned ion beam at a crater edge

修正文章：

The sputtering rate was about 36 % less for the miss-aligned depth profiling obtained at the crater edge than that for well-aligned profiling at the center of the crater.

［査読者 1-1-2］

二つ目は，同じ項目のところで，25 min と 39 min というように，時間で表現されておりますので，その元となるデータも時間で表示があったら，わかりやすいと思います.

［著者］

コメントにしたがって Fig. 3 の上横軸を時間スケールとしました.

査読者 2．井上雅彦（摂南大学）

本技術報告は, AES, XPS での測定で重要となるイオンビームのアライメント手順について初心者にもわかるように具体的に示したものです．分析の現場で有用な情報であり，JSA に掲載する価値があると認めます．このままで掲載可と判断しますが，いくつか小さなミスプリントを見つけましたので修正をお願いします.

［査読者 2-1］

イオンビームをラスターしながら Faraday cup で測定すると，条件によってはビーム電流は交流になってしまうと思われます．Fig. 6 で測定されているイオン電流は，実験で用いられた電流計の時定数が十分大きく，本来変動する電流が平均化されて測定されたものでしょうか？このあたりの条件，状況等を追記された方が良いような気もします.

［著者］

電流の測定には Keithley 6485 ピコアンメーターを使っています．仕様によると，20 nA レンジの rise time（0-2 V）が 8 ms，200 nA レンジでは 500 μs となっています．ラスター距離が大きく電流が 20 nA 以下の場合とラスター距離が小さく電流が 20 nA 以上で時定数は異なっています．ファラデーカップ径が 250 μm，ビーム径はそれ以上であるが，Fig. 6 の x 軸全領域で提起の問題が生じていることになります．実際の測定ではラスター距離，ラスター周期に依存した平均値となっていますが，現時点での推定は難しい状況です．ですので，電流計測に用いたピコアンメーターの機種とイオンビームの走査速度に関して次の文章を「2. Experimental」の最終段落に加えました.

追加文章：

The current is measured with a Keithley 6485 picoammeter for the ion beam that is swept at the rate of about 1 kHz along the x-axis and 10 Hz along the y-axis.