Technical Proposal for Measurement of Sputtered Depth Using a Mesh - Especially for Auger Depth Profiling -

M. Suzuki, M. Mogi, and H. Ando
Center for Materials Developments and Analytical Technology, NTT Advanced Technology Corporation, Morinosato-Wakamiya, Atsugi, Kanagawa 243-0198, Japan

(Received October 5 1998; accepted January 7 1999)

We will propose a very easy method for measuring the ion-sputtered depth using a metallic mesh. This method can be adopted for bulk materials, which do not have any marker indicating the depth as layered systems do, and is applicable to a wide ion sputtered conditions. It is possible to measure the true depth at the actual analyzed position rather than to measure the conventional crater depth. We will also demonstrate the application to measuring the sputtering rate of a GaAs substrate even for a rastered area of 8x8 mm^2. The proposed method will be used for the investigation of the sputtering yields of various bulk materials.

1. Introduction

Depth profiling measurements [1] are widely used in practical surface analysis with AES (Auger electron spectroscopy), XPS (x-ray photoelectron spectroscopy), and SIMS (secondary ion mass spectrometry). Ion sputtering is inevitable because of its substance principal for SIMS analysis. In depth profiling technique, a "sputtered depth" is the most important parameter when discussed on an experimental as well as theoretical level. Although sputtered craters can be directly measured in SIMS, it is not easy to measure the sputtered depths in AES and XPS. This is because the rastered area of an ion beam is usually several square millimeters. In practical surface analysis with AES and XPS, sputtering rates are obtained for the various conditions of the operated apparatus in the laboratory using SiO_2 films [2], Ta_2O_5 films [3], and so on. One of the necessary conditions for these standard materials is the well-defined film thickness.

In daily/practical analytical work, analysts measure the time to completely sputter the film with a known thickness in AES or XPS measurements: thus, obtaining the sputtering rate. Film materials with thicknesses of which are known are limited, whether the thickness is certified or not. Then we have to adopt the sputtering rate for analyzed materials as that of materials with known thicknesses; this is even though we do not know whether the adopted sputtering rate is near or far from the actual sputtering rate of the analyzed material. The converted sputtered rate should actually be used for the analyzed material, referring the database of sputtering yields [4]. The database shows a large difference in sputtering yields depending on materials cited. Unfortunately, it is difficult to confirm whether the values shown in the database are near the true values for the analyzed materials. Furthermore, the method to obtain the sputtering rates using the films with the known thicknesses is limited to the layered material systems.

In order to experimentally obtain/estimate the sputtering rates for the materials that are not layered systems, even if an area of several square millimeters is sputtered, we will propose a method using a metallic mesh. It overcomes the difficulty of measuring the sputtering rate of bulk materials and for analyzed materials. An additional advantage is to be able to do an AES measurement for high-resistance materials without peak shifts caused by charging effects when measuring the specimen surface with a part of the metallic mesh instantaneously. In this report we will demonstrate the technical procedure and examples of the results.

2. Technical Procedure of Measuring Sputtered Depths with a Mesh and Example

It is very easy to prepare the specimen as shown in Fig. 1. Here we used a silicon substrate as an example of the specimen in this report. The mesh is put

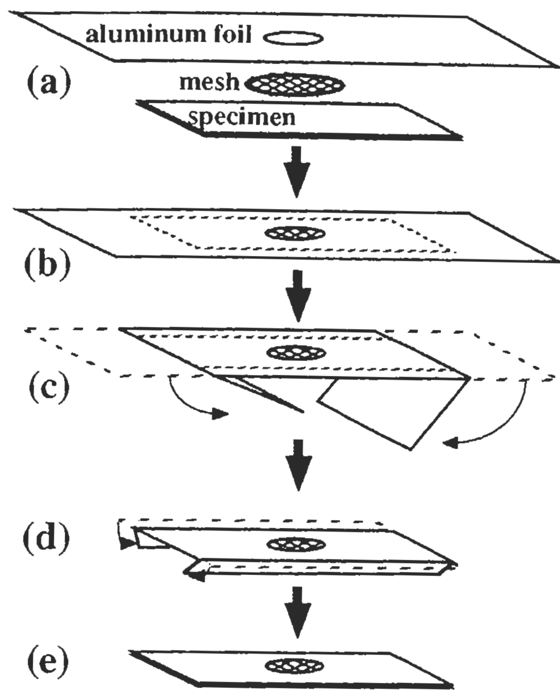

Fig. 1
Setting procedure of a specimen, a metallic mesh, and an aluminum foil with a hole. (a) The prepared things are the mesh and the aluminum foil with a hole. (b) The mesh and the aluminum foil are put one on top of the other on the specimen by aligning the mesh and the hole. (c), (d), (e) The aluminum foil is folded down over the mesh and the specimen.

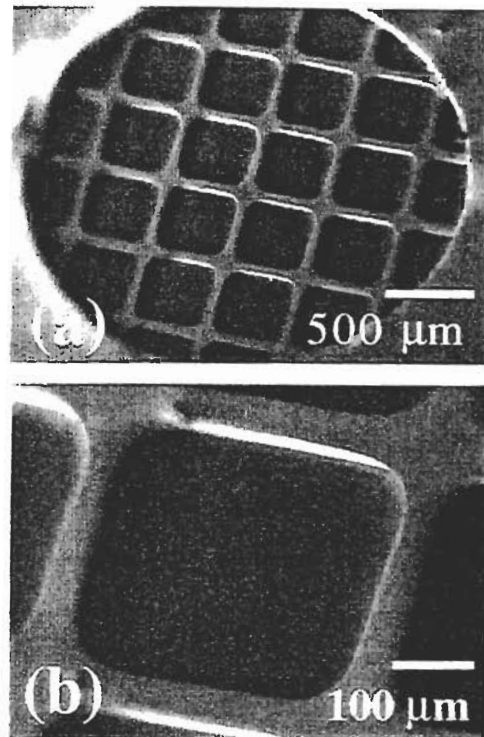

Fig. 2
SEM images taken by a SAM instrument before sputtering. (a) In the low magnitude image, the mesh is seen through the hole of the aluminum foil, and the specimen surface can be analyzed without any disturbance. (b) In the higher magnitude image, we can select area to be analyzed through the hole of the mesh lattice.

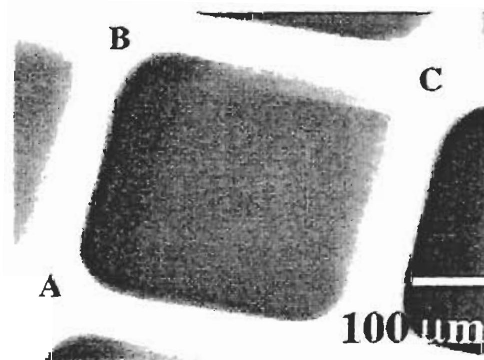

Fig. 3
SEM image with the mesh after sputtering. It is easy to find the mesh and the shadowed pattern caused by ion sputtering.

between the specimen and the aluminum foil. The suitable mesh is made from copper because of the softness. We use a mesh with a lattice spacing of several hundred micrometers, considering the ion beam diameter, sputtered area, and the capability of the used stylus profiler. The wrapping material for the specimen and mesh is aluminum foil because of its flexibility. Another material should be used if the aluminum reacts with the specimen materials. The hole diameter of the aluminum foil is about 2 mm. In this preparation procedure, it is important that the mesh and the hole of the aluminum foil are aligned well. Moreover, it is necessary that the analyzed area of the specimen must appear through the mesh window; this is confirmed with an optical microscope etc. The aluminum foil should be wrapped and fitted with the mesh and the specimen, caring to make good electric and mechanical contact between them.

Fig. 2 shows the SEM (secondary electron microscope) images of the specimen (silicon substrate in this example) surface with the mesh and the aluminum foil before sputtering. When the analyzed area is specified, an operator may locate the position in a low magnitude image like Fig. 2(a). In daily SAM (scanning Auger electron microscopy) analytical work, the

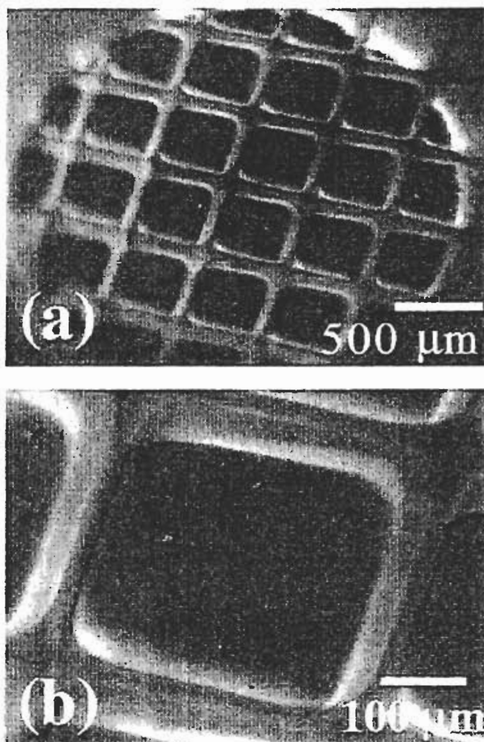

Fig. 4
SEM images after sputtering and removal of the mesh, though this step is not required in usual analysis procedure. (a) In the low magnitude image, the shadowed pattern by the mesh is seen clearly. (b) In the higher magnitude image, there are non-sputtered patterns, normally-sputtered areas, and the sloping area from the former towards the latter. The small features in the sputtered area may have been formed by small particle dusts on the surface before sputtering.

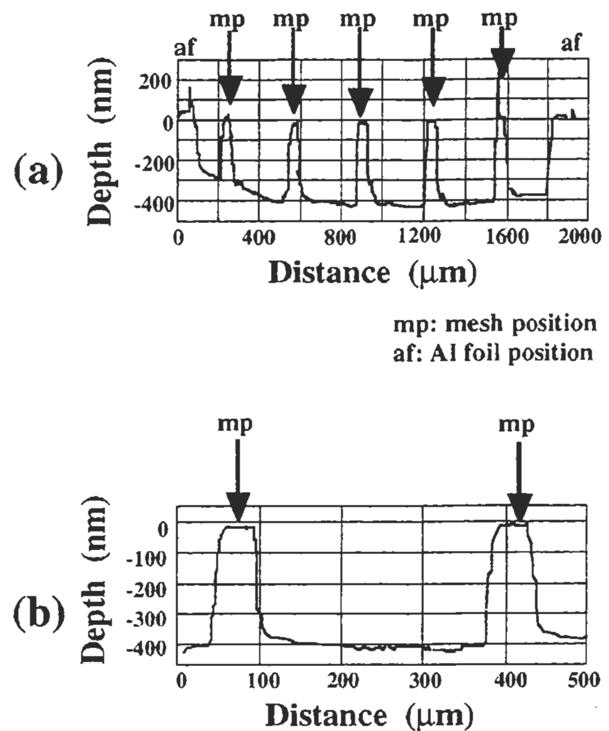

Fig. 5
Stylus profiler images after sputtering. (a) In the low magnitude profile, there are non-sputtered areas on the both sides shadowed by the aluminum foil and the protrusions formed by the mesh lattice. (b) From the higher magnitude profile, the sputtered depth of the actual analyzed area can be estimated by determining the matrix address corresponding to the mesh lattice. In the figures, "mp" and "af" denote the shadow area resulting from the mesh and the aluminum foil, respectively.

analysis is carried out in a higher magnitude SEM image where any part of the mesh does not appear, though there is the mesh in the higher magnitude SEM image of Fig. 2(b). Fig. 3 shows the same magnitude image as Fig. 2(b) after sputtering. The dark area along the right side of the lattice points, AB, is the shadow area for the secondary electron detector. The bright area along the lower side of the points BC is the sloping area, corresponding to the side wall of the sputtered zone, which was not sputtered due to the mask of the mesh.

Fig. 4 shows the SEM image after sputtering and removal of the mesh in air, although the process of this SEM observation is not usually needed for daily analysis. The low magnitude SEM image (Fig. 4(a)) clearly shows the mesh pattern on the specimen surface. In the higher magnitude SEM image (Fig. 4(b)), it is found that the bottom of the sputtered zone is overall very flat and that several small particles are on the bottom face. The same kinds of particle are frequently seen for various materials. We consider that the sputtered zone is contaminated by the particles prior to sputtering and thus, they are affected the topography after sputtering.

Fig. 5 shows the cross-sectional profiles obtained by a commercial stylus profiler (SLOAN, 3030ST). It is clearly seen that the sputtered shape corresponds to the aluminum foil and the mesh pattern. In the low magnitude profile, it is possible to measure the whole length along the diameter of the aluminum foil hole. Thus we can select the sputtered zone in which the actual analysis was performed, resulting in the true sputtered depth. This is true especially for SAM analysis, as shown in Fig. 5(b). Here the flat region is about

250 μm wide and it is very easy to measure the sputtered depth.

Now we will show an example where the mesh method is adopted for a bulk material which, unlike layered systems, does not have any marker indicating its layer thickness. Fig. 6 shows the sputtering rate of GaAs substrate against the rastered distance of the ion beam using the SAM instrument. When the mesh is not used, we may be able to estimate the sputtering rate from the sputtered depth for the rastered distance of 1 to 2 mm. It is very difficult, however, to measure the sputtered depth for the long rastered distances of 5 to 8 mm. We can, however, measure depths accurately by the mesh method presented here even for wide rastered regions. For a silicon substrate, the depth can be measured with the error of ± 7 nm at 400 nm in depth, corresponding 3.5% as shown in Fig. 5(b). An additional benefit of the mesh method in the AES analysis of a high resistivity material is that we can avoid the charging effect by measuring the specimen surface with a part of the metallic mesh. An AES spectrum includes the characteristic peaks from the mesh material, though we can obtain AES peaks coming from the specimen without energy shifts. Here we do not show the result for preventing the charging effect. It will be reported elsewhere.

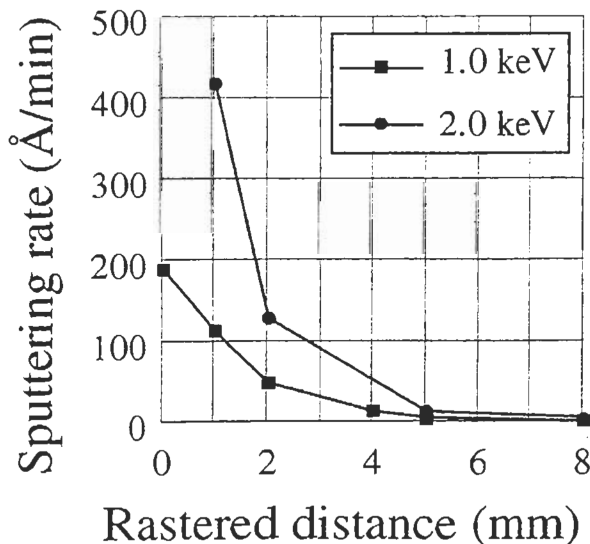

Fig. 6
The sputtering rate of GaAs bulk against the ion beam rastered distance. The rastered area corresponds to the squared values of the distances. The energies of an Ar^+ ion beam are 1 keV and 2 keV.

3. Summarizing Remarks

We have proposed a very easy method using a metallic mesh to measure sputtered depth. This method can be adopted for bulk materials that do not have any marker indicating the depth and is applicable to very wide sputtered conditions. We also demonstrated the application result to the measurement of the sputtering rate of a GaAs substrate. It is possible to estimate the sputtering rate even for a rastered area of 8x8 mm^2. The proposed method will be used in the investigation of the sputtering yields of various bulk materials. This method is also applicable in AES measurements for high resistivity materials without peak enrgy shifts doe to charging effects.

Acknowledgments
The authors are very grateful to Ms. M. Masuda and Mr. H. Okamoto in NTT Advanced Technology Corp., and Drs. Y. Homma and T. Hayashi in NTT Laboratories for their development of this technique and useful discussions.

References

[1] for example, Practical Surface Analysis Vol. 1 Chapter 4, Ed. by D. Briggs and M. P. Seah, Wiley 1990 (Second Edition); Surface Analysis - The Principal Techniques -, Ed. by J. C. Vickerman, Wiley 1997; Thin Film and Depth Profile Analysis, Ed. by H. Oechsner, Springer-Verlag 1984.
[2] SRM-2531-2536 from NIST, USA.
[3] No. S7B83 from NPL, UK.
[4] for example, Appendix 4.4.3 in Practical Surface Analysis Vol. 2, Ed. by D. Briggs and M. P. Seah, Wiley 1990 (Second Edition).