Influences of Measurement Conditions on Etching Rate of GaAs/AlAs Superlattice in Auger Electron Spectroscopy Sputter Depth Profiling

T. Nagatomi,* T. Bungo, and Y. Takai
Department of Material and Life Science, Graduate School of Engineering, Osaka University
Suita, Osaka 565-0871, Japan
*nagatomi@mls.eng.osaka-u.ac.jp

(Received: January 16, 2009; Accepted: February 6, 2009)

Influences of measurement conditions such as the base pressure of the analysis chamber, the acquisition time of Auger electron spectroscopy (AES) and the sputtering time in one period, on AES sputter depth profiling of a GaAs/AlAs superlattice reference material were investigated by irradiating 750 eV Ar^+ ions at the incident angle of 50°. The results revealed that the ratio of the etching rate of AlAs to that of GaAs is strongly influenced by the base pressure of the analysis chamber. When the base pressure is ~2×10^{-9} Torr, the ratio of the etching rate is almost constant at ~0.9 and not affected by measurement conditions. In contrast, when the base pressure is ~8×10^{-9} Torr, the ratio of the etching rate is strongly affected by measurement conditions. The shorter the sputtering time in one period is, the smaller the ratio of the etching rate is. The longer acquisition time of AES is also considered to introduce the difference in the ratio of the etching rate. The influences on the etching rate caused by the measurement conditions might be attributed to the oxygen adsorption on the AlAs surface. In addition, in spite of the strong dependence of the ratio of the etching rate on the measurement condition at the base pressure of ~8×10^{-9} Torr, the depth resolution does not depend on the measurement condition. The present results strongly suggest that careful attention for the optimization of the measurement condition of sputter depth profiling using the GaAs/AlAs superlattice reference material is required since the etching rate is an important factor to quantitatively understand the depth scale of depth profiles.

1. Introduction

The subnanometer depth resolution is required for the characterization of the in-depth distribution of the concentration of the layered structure and the shallow doped layer of semiconductor devices. Sputter depth profiling using surface chemical analysis techniques such as Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), and secondary ion mass spectrometry (SIMS), with the help of sputter etching is one of the most widely used techniques for in-depth profiling. Since the primary energy of ions commonly used for sputter etching in depth profiling is more than a couple of keV, irradiation of such energetic ions induces the atomic mixing in the surface region and results in the deterioration of the depth resolution.

From the point of view of the interaction of ions with the solid surface, the application of low-energy ions of a few hundreds eV to sputter depth profiling is one of the most promising approaches for achieving the higher depth resolution in routine sputter depth profiling analyses. A lot of studies have revealed that the application of low-energy ions is effective for achieving the higher depth resolution [1]. However, sputtering phenomena induced by the low-energy ion irradiation have not been well understood. In order to accommodate the high-depth resolution in-depth profiling in the routine analyses, more comprehensive understanding of low-energy ion sputtering is essential. We have been involved in the investigation of low-energy ion sputtering and found several sputtering phenomena occurred only in low-energy region [2-4].

In the present paper, we reported results obtained by the investigation of influences of measurement conditions in sputter depth profiling of a GaAs/AlAs superlattice reference material on the etching rate, which is one of the important factors for accurate understanding of depth profiles.

2. Experiment

All the experiments were performed using a scanning Auger microscopy (JAMP-3, JEOL). The apparatus was equipped with an electron gun, a cylindrical mirror analyzer (CMA), a compact ultrahigh vacuum floating-type low-energy ion gun

(UHV-FLIG) [5-7]. Primary ions used for sputter etching were 750 eV Ar^+ ions. The AES measurement was performed using 10 keV electrons. The incident angles of ions and electrons are 50° and 45°, respectively.

The sample used in the present study was a GaAs/AlAs superlattice reference material utilized to optimization of the condition of sputter depth profiling using AES, XPS, and SIMS [8]. In order to convert the sputtering time of obtained depth profiles to the depth was performed by the profile analysis using the mixing-roughness-information depth (MRI) model [9-11]. Details of the experimental setup and the MRI analysis are described elsewhere [12-14].

3. Results and discussion

Figure 1 shows the Al-LVV and Ga-LMM depth profiles obtained by irradiation of 750 eV Ar^+ ions. The base pressure was ~2×10^{-9} Torr. The thickness of the atomic mixing zone, w, and the surface roughness, σ, deduced by the MRI analysis are 1.9 and 0.9 nm, respectively, corresponding to the depth resolution of 3.3 nm for the Al-LVV profile at the leading edge of the AlAs layer under the 16-84 criterion [15]. These values are in good agreement with those reported previously [3,4]. It is clearly seen that the times required to remove the GaAs and AlAs layers are different. The etching rate of AlAs to that of GaAs, R, is obtained to be 0.87.

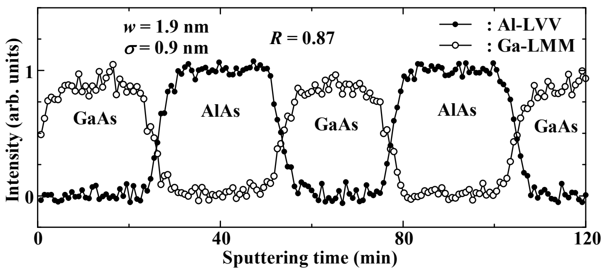

Fig. 1 Al-LVV and Ga-LMM depth profiles of the GaAs/AlAs superlattice obtained by sputter etching using 750 eV Ar^+ ions. The base pressure of the analysis chamber was ~2×10^{-9} Torr. The intensity is normalized so that the intensity in the plateau region of the Al-LVV profile in AlAs is 1. The ion irradiation time in each period was 45 s.

Figure 2 shows the depth profiles similar to that shown in Fig. 1 at the base pressure of 8×10^{-9} Torr, which is 4 times higher than that for Fig. 1. The ratio of the etching rate is obtained to be R=0.72, indicating that the ratio of the etching rate is significantly different by ~20% between Figs. 1 and 2. However, the thickness of the mixing zone, w, and the surface roughness, σ, are the same as those in Fig. 1, indicating that the depth resolution was not changed and only the ratio of the etching rate was affected. Note that the ratio of the etching rate, R, does not depend on measurement conditions at all when the base pressure was ~2×10^{-9} Torr.

Figure 3 shows the O-KLL spectra obtained during depth profiling shown in Fig. 2(a). Before depth profiling, oxygen contaminations exist on the 1st GaAs surface. After staring sputter depth profiling, oxygen contaminations on the 1st GaAs layer are sputtered off and no oxygen atom exists on the surface during sputter etching of the 1st GaAs layer. However, the adsorption of oxygen is confirmed during sputter etching of the 2nd AlAs layer.

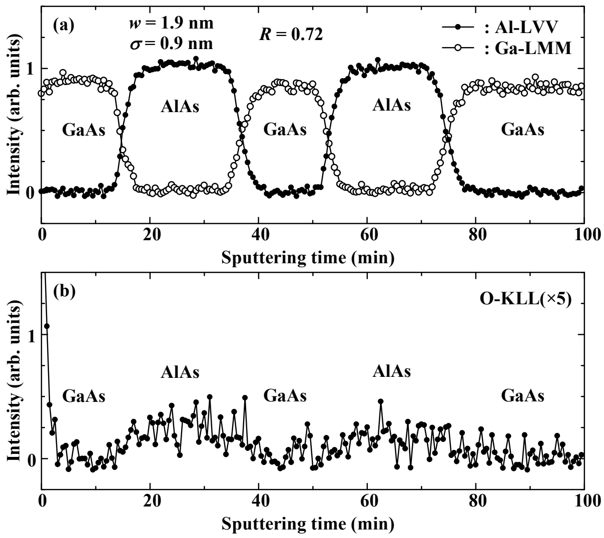

Fig. 2 (a) Al-LVV and Ga-LMM and (b) O-KLL depth profiles of the GaAs/AlAs superlattice obtained with 750 eV Ar^+ ions. The base pressure of the analysis chamber was ~8×10^{-9} Torr. The intensity is normalized so that the intensity in the plateau region of the Al-LVV profile in AlAs is 1. The intensity of the O-KLL profile in (b) is multiplied by a factor of 5. The ion irradiation time in each period was 30 s.

Figure 2(b) depicts the O-KLL depth profile simultaneously obtained with Fig. 2(a). It is clearly observed that oxygen exists only during sputter etching of the AlAs layer, suggesting that the oxygen adsorption may influence the etching rate.

In order to investigate the correlation between the oxygen adsorption and the ratio of the etching rate, sputter depth profiling of the GaAs/AlAs superlattice reference material was performed under almost the same conditions as those of Figs. 2 and 3. The resultant sputter depth profiles are shown in Fig.

4. It is found that the thickness of the atomic mixing zone, w, and the surface roughness, σ, are identical in all profiles. The adsorption of oxygen during sputter etching of the AlAs layer is confirmed, and the ratio of the etching rate of AlAs to that of GaAs is different from that shown in Fig. 1.

For more detailed investigation, the correlation between the ratio of the etching rate of AlAs to that of GaAs, R, and the intensity of the O-KLL peak detected during sputter etching of the AlAs layer is shown in Fig. 5. All the ratio of the etching rate, R, obtained for Figs. 2 and 4, where the base pressure of the analysis chamber was ~8×10^{-9} Torr, are below that in Fig. 1, the base pressure of which was ~2×10^{-9} Torr. However, no systematic correlation between the ratio of the etching rate and the O-KLL peak intensity was confirmed.

Considering the correlation between the ion irradiation time for one period with the ratio of the etching rate, it is found that the longer the ion irradiation time for one period is, the larger the ratio of the etching rate is. This tendency is clearly confirmed in Fig. 6. In addition, the time required for the measurement of one set of AES spectra was ~60 s for Fig. 1, where the Al-LVV, Al-KLL and Ga-LMM peaks were measured, and ~135 s for Figs. 2 and 4, where O-KLL, As-MVV and As-LMM peaks were measured as well.

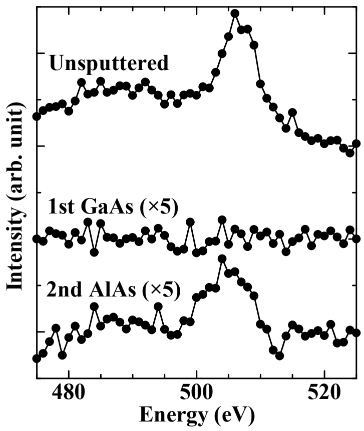

Fig. 3 O-KLL spectra obtained during sputter depth profiling shown in Fig. 2. The upper, middle and bottom spectra are those obtained before starting sputter depth profiling, during sputter etching of the 1st GaAs and 2nd AlAs layers, respectively. The intensities of the spectra for the 1st GaAs and 2nd AlAs layers are multiplied by a factor of 5.

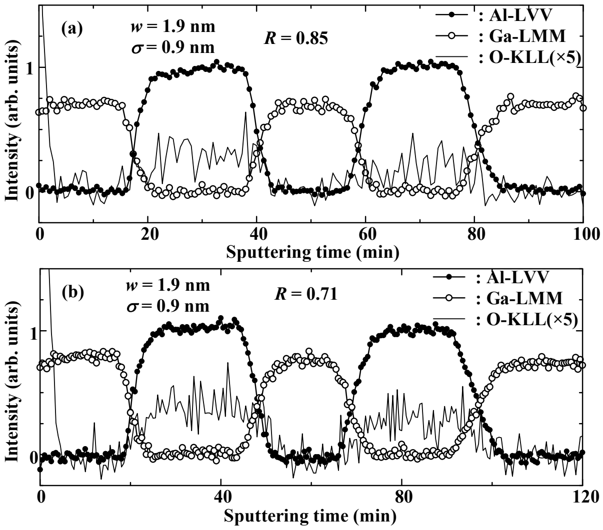

Fig. 4 Al-LVV, Ga-LMM, and O-KLL depth profiles of the GaAs/AlAs superlattice reference material obtained with 750 eV Ar^+ ion irradiation. (a) and (b) are the profiles obtained by irradiating ions for 40 and 30 s in each period. The base pressure of the analysis chamber was ~8×10^{-9} Torr. The intensity of the O-KLL profile is multiplied by a factor of 5.

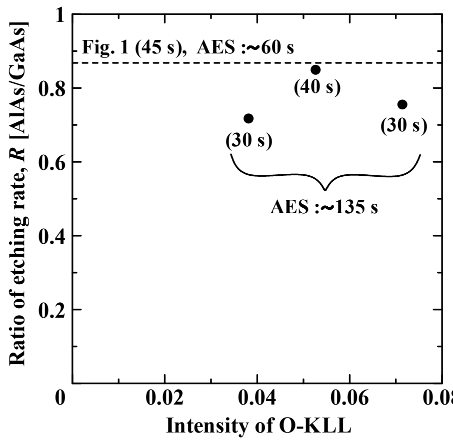

Fig. 5 Ratio of the etching rate of AlAs to that of GaAs as a function of the intensity of the O-KLL peak detected near the middle of the plateau of the 2nd AlAs layer. The time shown in a parenthesis indicates the time of ion irradiation in each period. The time denoted with AES is the acquisition time required for the measurement of one set of AES spectra. The horizontal broken line denoted as Fig. 1 represents the ratio of the etching rate obtained under the condition of the base pressure of 2×10^{-9} Torr shown in Fig. 1, where O-KLL spectra were not measured to reduce the measurement time.

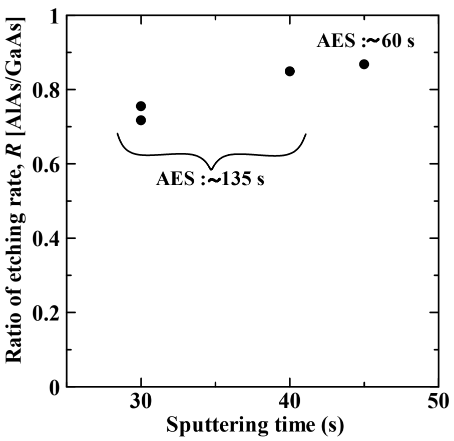

Fig. 6 Ratio of the etching rate of AlAs to that of GaAs, *R*, as a function of the ion irradiation time in one period. The time denoted by AES is that required for the measurement of one set of AES spectra.

The base pressure of the analysis chamber for measurements of Figs. 2 and 4 is ~4 times higher than that of Fig. 1. The thickness of the sputtered layer by each period of ion irradiation of ~30 s is approximately 0.5 nm corresponding to ~1.5 atomic layer. Taking into account the fact that the difference in the etching rate was not observed for different measurement conditions when the base pressure was $\sim 2\times10^{-9}$ Torr, it is suggested that the ratio of the etching rate is affected by the ion irradiation time in one period and the acquisition time for the measurement of one set of AES spectra, i.e., the oxygen adsorption.

4. Summary

Influences of measurement conditions such as the base pressure of the analysis chamber, the acquisition time of AES and the sputtering time in one period, on AES sputter depth profiling of the GaAs/AlAs superlattice reference material were investigated. The obtained results are summarized as follows:

(i) When the base pressure of the analysis chamber is $\sim 2\times10^{-9}$ Torr, depth profiles are not affected by the measurement conditions. The ratio of the etching rate of AlAs to that of GaAs is constant at ~0.9 under irradiation of 750 eV Ar^{+} ions at the incident angle of 50°.

(ii) If the base pressure is $\sim 8\times10^{-9}$ Torr, the ratio of the etching rate of AlAs to that of GaAs becomes smaller for the shorter sputtering time in one period.

(iii) The acquisition time of AES in one period is also considered to affect the etching rate. The longer the acquisition time is, the smaller the ratio of the etching rate is.

(iv) The oxygen adsorption on the AlAs surface is confirmed, indicating that the influences of the measurement condition on the etching rate is due to the oxygen adsorption on the AlAs surface.

The present results strongly suggest that careful attention for the optimization of the measurement conditions of sputter depth profiling using the GaAs/AlAs superlattice reference material is required since the etching rate is an important factor to quantitatively understand the depth scale of depth profiles.

5. References

[1] For instance, M. G. Dowsett, N. S. Smith, R. Bridgeland, D. Richards, A. C. Lovejoy, P. Pedrick. In Proceedings of *Tenth International Conference on Secondary Ion Mass Spectrometry (SIMS-X)*, A. Benninghoven, B. Hagenhoff, H. W. Werner (eds) (John Wiley, Chichester, 1997) 367.

[2]Y. Mizuhara, T. Bungo, T. Nagatomi, and Y. Takai, *Surf. Interface Anal.* **37**, 343 (2005).

[3] T. Bungo, T. Nagatomi, and Y. Takai, *Surf. Interface Anal.* **38**, 1598 (2006).

[4] T. Nagatomi, T. Bungo, and Y. Takai, *Surf. Interface Anal.* (*submitted*).

[5] T. Matsutani, K. Iwamoto, T. Nagatomi, Y. Kimura, Y. Takai, R. Shimizu, R. Aihara, and Y. Sakuma, *J. Vac. Soc. Jpn.* **43**, 1126 (2000).

[6] Y. Mizuhara, J. Kato, T. Nagatomi, Y. Takai, T. Aoyama, A. Yoshimoto, M. Inoue, and R. Shimizu, *Surf. Interface Anal.* **35**, 382 (2003).

[7] Y. Mizuhara, T. Bungo, T. Nagatomi, Y. Takai, S. Suzuki, K. Kikuchi, T. Sato, and K. Uta, *Surf. Interface Anal.* **37**, 171 (2005).

[8] ISO-14606. 2000(E); *Surface Chemical Analysis – Sputter Depth Profiling – Optimization Using Layered Systems as Reference Materials.* ISO: Geneva.

[9] S. Hoffman, *Surf. Interface Anal.* **21**, 673 (1994).

[10] S. Hofmann, *Rep. Prog. Phys.* **61**, 827 (1998) .

[11] T. Bungo, Y. Mizuhara, T. Nagatomi, and Y. Takai, *Jpn. J. Appl. Phys.* **42**, 7580 (2003).

[12] Y. Mizuhara, J. Kato, T. Nagatomi, Y. Takai, and M. Inoue, *J. Appl. Phys.* **92**, 6128 (2002).

[13] Y. Mizuhara, J. Kato, T. Nagatomi, Y. Takai, and M. Inoue, *Jpn. J. Appl. Phys.* **42**, L709 (2003).

[14] T. Bungo, Y. Mizuhara, T. Nagatomi, and Y. Takai, *Jpn. J. Appl. Phys.* **42**, 7580 (2003).

[15] S. Hofmann, *Appl. Surf. Sci.* **70/71**, 9 (1993).