Development and Applications of Multiple Delta-Layer Reference Materials for Semiconductor Analysis

K. J. Kim[1*], D. W. Moon[1], P. Chi[2] and D. Simons[2]

[1]*Nano Surface Group, Korea Research Institute of Standards and Science, P.O.Box 102, Yusong, Daejon 305-600, Korea*

[2]*Surface and Microanalysis Science Division, National Institute of Standards and Technology, Gaithersburg, MD 20899-8371, USA*

[*]kjkim@kriss.re.kr

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The rapid progress in the semiconductor industry requires more precise in-depth profiling analysis of doping elements in narrower and shallower regions. Secondary ion mass spectrometry (SIMS) is the most popular technique for determining the depth distribution of doping elements within 20 nm shallow junctions. Multiple delta-layers (MDL) were prepared by ion beam sputter deposition to enhance the analysis capability of SIMS depth profiling. These MDLs can be used to evaluate SIMS depth resolution, to calibrate the depth scale and to monitor sputtering uniformity. Another possible application is as a consistency check of the calibration of stylus profilometers for measurement of sputter depth. Craters with 3 different depths were formed by sputtering with a Cs ion beam. The crater depths measured by a stylus profilometer were compared with the certified thickness of the reference material measured by a high resolution TEM.

INTRODUCTION

For the development of next-generation semiconductor devices, the international technology roadmap for semiconductors (ITRS) defines the requirement to measure 1 nm gate oxide thickness and the distribution of doping elements within a 20 nm shallow junction depth [1]. SIMS is one of the most useful techniques for the in-depth analysis of minor impurities, because of its high sensitivity and detection capability of all elements including hydrogen. However, determining the SIMS depth scale is complicated by the change of sputter rate in the near-surface during ion beam sputtering. The evaluation of depth resolution in the shallow depth region is very important in sputter depth profiling of shallow junctions.

International standards for useful in-depth analysis have been developed by the International Organization for Standardization (ISO) [2]. ISO-14606 describes the optimization of depth profiling parameters to achieve optimum depth resolution using layered systems [3]. ISO-20341 specifies a method to evaluate the depth resolution parameters using multiple delta-layers [4]. ISO/TR-15969 summarizes the measurements of crater depth by mechanical stylus or optical interferometry [5].

For precise in-depth profiling, well-defined certified reference materials (CRM) are required to calibrate the depth scale and evaluate the depth resolution. Several multilayer standard reference materials have been developed by national measurement institutes. Polycrystalline Ni/Cr multilayers [6], AlAs/GaAs superlattices [7,8] and Ta_2O_5/Ta multilayers [9] have been developed for depth profiling. A marker type multilayer SRM consisting of 7 Cr layers separated by very thin Cr_2O_3 layers was developed [10]. However, these SRMs are inadequate for the analysis of shallow junctions. Recently, boron delta-layers [11] were developed to calibrate the depth scale of the shallow junctions.

In this paper, we introduce the development of multiple delta-layer reference materials for shallow junction analysis and some useful applications of them.

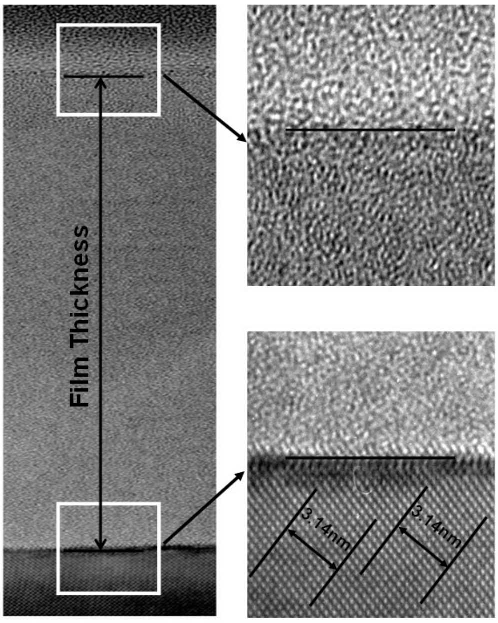

Fig. 1. Certification method of film thickness by HR-TEM based on Si lattice constant.

FABRICATION OF MULTIPLE DELTA-LAYERS

Multiple delta-layer films were grown by ion beam sputter deposition (IBSD) [12]. The target material was sputtered by a 1 keV Ar^+ ion beam and deposited on 150 mm diameter Si wafers. The deposition chamber was connected to a surface analysis system and therefore the original chemical state, composition and impurity levels could be analyzed by *in-situ* x-ray photoelectron spectroscopy (XPS). Multilayer films can be grown by alternating deposition of two target materials using a rotatable target holder. One side was a pure Si wafer target for the growth of Si layers. The other side was a Si wafer target attached with a small metal foil for the growth of metal-doped Si delta-layers. The film thickness was controlled by variation of the growth time.

The growth rate was calibrated by high resolution transmission electron microscope (HR-TEM) measurement of a thin film grown in a known time. The thickness of the standard specimen was measured by HR-TEM, where the distance between the crystal planes (0.31356 nm) can be used as an internal standard for the measurement of the film thickness as shown in Fig. 1. To establish the calibration factor for a TEM image, the separation of 50 Si lattice planes was measured 5 times and the average was set equal to 15.68 nm. The substrate was rotated at a speed of 30 r/min to improve the homogeneity. The thin film grown on a silicon wafer was divided into 10 mm X 10 mm specimens.

The specimens taken from near the center and edge of the wafer are not used as standard specimens. The thickness and dopant concentration of the delta-layers were optimized to minimize the matrix effect. Figure 2 shows SIMS depth profiles of typical multiple delta-layers for SIMS depth profiling. B- and As-doped delta-layers can be used for positive and negative SIMS, respectively.

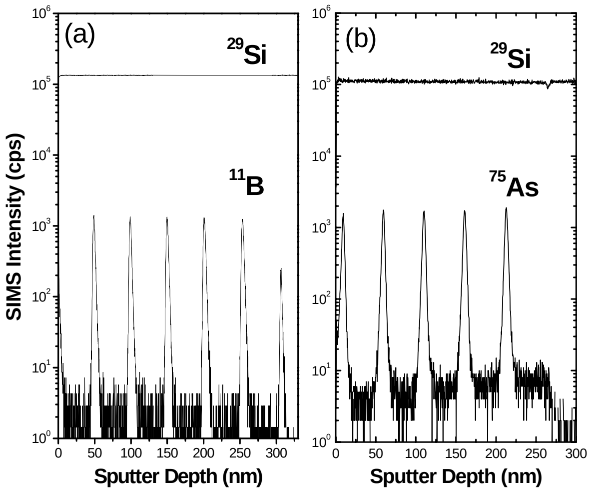

Fig. 2. SIMS depth profiles of typical multiple delta-layers with (a) B and (b) As-doped Si marker layers.

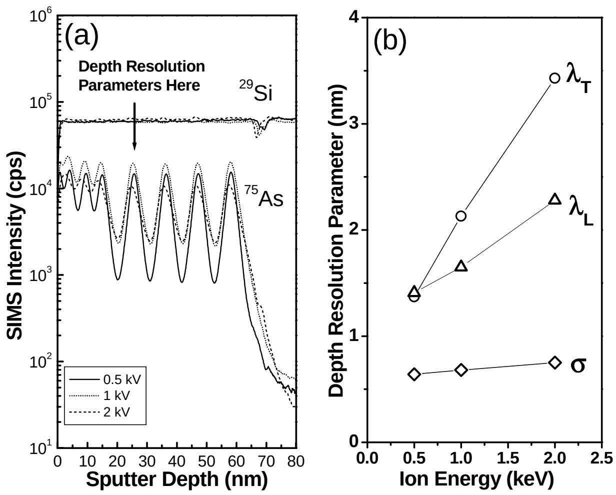

Fig. 3. SIMS depth profiles of As-doped Si multiple delta-layer with various ion energies (a) and depth resolution parameters (b).

APPLICATIONS OF MULTIPLE DELTA-LAYER IN SIMS

Optimization of SIMS parameters

Improvement of depth resolution is one of the most important issues of SIMS depth profiling because it is an essential indicator of SIMS analysis capability. SIMS operating parameters must be optimized to get the best depth resolution. Generally, a decrease of primary ion energy, an increase of incidence angle and use of cluster ions are recommended to improve SIMS depth resolution. ISO-14606 describes methods to optimize the experimental conditions using layered systems as reference materials. Polycrystalline Ni/Cr multilayers, AlAs/GaAs superlattices and Ta_2O_5/Ta multilayers are recommended as useful layered systems. Multiple delta-layers can also be used for the improvement of depth resolution. Figure 3 shows a significant improvement of depth resolution by decrease of the Cs ion energy from 2 keV to 500 eV. The surface As layers are clearly separated by the 500 eV ion beam but are poorly separated by the 2 keV ion beam. An increased sputtering rate in the transient region was also observed [12].

Evaluation of Depth Resolution Parameters

Evaluation of depth resolution is required to confirm the SIMS capability for in-depth analysis in the shallow depth region. The 84%-16% definition is not adequate for the evaluation of depth resolution in SIMS depth profiling where the matrix change at a sharp interface leads to severe interface artifacts [13]. To avoid these interface artifacts, multilayers separated with very thin marker layers can be used [14,15]. ISO-20341 specifies the method for estimation of depth resolution parameters. For the use of this standard, a SIMS profile of a delta- layer shall be described using an exponential rising edge, a Gaussian-like rounded top and an exponential trailing edge. Assuming the SIMS profile of a delta-layer to be a convolution of the two exponentials, $f_L(z)$ in Equation (1) and $f_T(z)$ in Equation (2) with a Gaussian as $g(z)$ in Equation (3), three parameters are required: the leading edge decay length (λ_L), the trailing edge decay length (λ_T), and the Gaussian broadening (σ).

$$f_L(z) = A\exp\left[\frac{z-z_0}{\lambda_L}\right] \quad , \; z < z_0 \qquad (1)$$

$$f_T(z) = A\exp\left[\frac{-(z-z_0)}{\lambda_T}\right] \quad , \; z > z_0 \qquad (2)$$

$$g(z) = \frac{B}{\sqrt{2\pi\sigma}}\exp\left[\frac{-(z-z_0)^2}{2\sigma^2}\right] \quad , \qquad (3)$$

where z is the depth (nm) and z_0 is the apparent peak depth (nm). λ_L, λ_T and σ can be derived from deconvolution using non-linear curve fitting software which is available elsewhere [4,14].

ISO-20341 requires multiple delta-layers that satisfy the following criteria. The matrix of sputtered surface layers shall not change during SIMS depth profiling so that no changes occur in any SIMS matrix effects or in the erosion rate during depth profiling. The surface and the delta-layers shall be flat and parallel to each other. The thickness of the doped delta-layers shall be sufficiently thin. The spacing between adjacent delta-layers shall be large enough so that the secondary ion intensity at the valley between layers is less than 1% of the peak intensity. Figure 4 (a) shows a SIMS depth profile of a B-doped Si multiple delta-layer using a 500 eV O_2^+ ion beam. Depth resolution parameters slightly increase with the increase of sputter depth (b). The trailing edge decay length is much larger than the leading edge decay length.

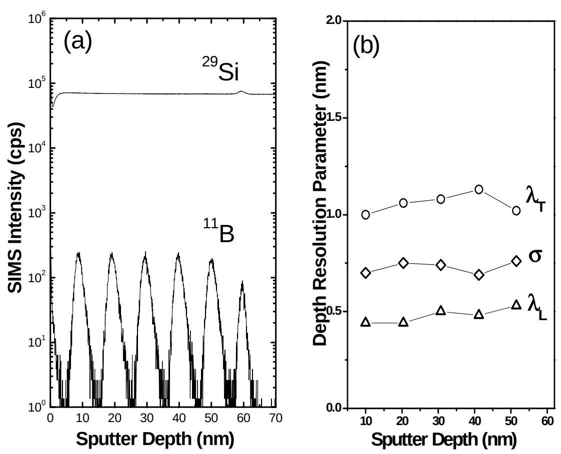

Fig. 4. SIMS depth profiles of a B-doped Si multiple delta-layer (a) and depth resolution parameters (b).

Calibration of Sputtered Depth

Accurate calibration of sputtered depth is very important because it is a basis for the accurate depth determination of shallow junctions [12]. The depth scale is generally determined by the measurement of crater depth generated after SIMS profiling analysis [16,17]. ISO/TR-15969 discusses the reliable measurement of sputtered depth [5]. The crater depth is defined as the average distance between the original surface and the region of a crater bottom from which the measured signal is derived. The crater depth can be measured by mechanical stylus profilometry or optical interferometry. Reference materials with interfaces as depth markers can also be used for the calibration of the depth scale.

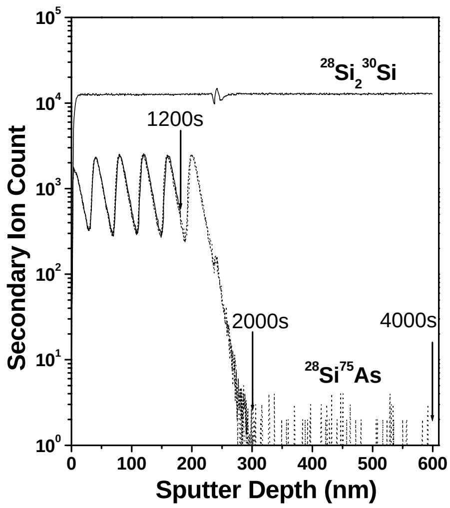

Figure 5. Overlay of 3 SIMS depth profiles of a GaAs-doped Si multiple delta-layer with different crater depths.

Table 1. Crater depth measurements by stylus profilometry

Measurement Date	Crater Depth (nm)		
	180 nm	300 nm	600 nm
2-Aug-2004	184.25	307.64	609.38
2-Sep-2004	184.33	307.04	609.63
22-Sep-2004	183.87	307.11	609.78
Average	184.15	307.26	609.60
ASD (nm)*	2.07	1.85	2.25

*ASD: average of measurements of standard deviation on 3 dates.

In this study, the sputtered depth derived from a multiple delta-layer with a certified thickness was compared with the sputtered depth measured by stylus profilometry. To do this, three craters with different depths were generated by sputtering of a multiple delta-layer CRM (KRISS CRM 103-04-102) with a 10 keV Cs^+ beam. The certified total thickness of the CRM film layer is 249.0 ± 0.5 nm and it consists of 6 Si layers on a Si(100) wafer separated by 5 GaAs-doped Si delta-layers of 0.5 nm thickness. Figure 5 shows 3 SIMS profiles after sputtering of 1200s, 2000s and 4000s. The sputtered depths of the three craters measured by stylus profilometry are 184.15nm, 307.26nm and 609.60 nm with average standard deviations over 3 measurement dates of 2.07nm, 1.85nm and 2.25nm, respectively. Each crater was measured 12 times on each day. The depth scale was calibrated by a calibration sample with a certified step height of 915.6 nm and a standard uncertainty of 0.3%. Table 1 shows the long-term repeatability of crater depths measured just after, 30 and 50 days after the SIMS profiling. The variations of measured crater depth were very small over this time.

The correct position of the interface must be defined precisely for the proper calibration of the depth scale using multilayered systems. One definition of interface location is where the peak intensity decreases to half of the maximum intensity. The thickness to the fifth delta-layer measured by this definition using the profilometry values (212.6 nm) is somewhat larger than that estimated from the HR-TEM measurement (207.6nm). This discrepancy is due to peak broadening in the profile and can be diminished by using lower ion energy or cluster ions for sputtering.

CONCLUSIONS

Ion beam sputter deposition was used to prepare several multiple delta-layer reference samples for analysis by SIMS depth profiling. These MDLs can be used to improve and evaluate SIMS depth resolution as defined in ISO 14606 and 20341. The film thickness measured by HR-TEM can be used as a reference to calibrate the SIMS depth scale. The slight discrepancy between the layer depths determined by profilometry and by HR-TEM indicates the importance of defining the correct position of an interface from the measured profile of a delta layer.

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