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Routine depth profiling of ultra shallow junctions by secondary ion mass spectrometry

Paul K. Chu [a,*], Victor K.F. Chia [b], Stephen P. Smith [b], Charles W. Magee [c]

[a] *Department of Physics and Materials Science, City University of Hong Kong, 88 Tat Chee Avenue, Kowloon, Hong Kong*
[b] *Charles Evans and Associates, 301 Chesapeake Drive, Redwood City, CA 94063, USA*
[c] *Evans East, 666 Plainsboro Road, Suite 1236, Plainsboro, NJ 08536, USA*

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Abstract

Low energy ion implantation is used to form sub-100 nm shallow junctions in ULSI fabrication. In order to completely characterize these junctions, instrumental depth resolution must be optimized and the transient ion yield changes in the near surface region must be minimized. Depth profiling of shallow junctions has previously been demonstrated in a research environment, but the analysis protocols are quite complicated. We have developed a method to routinely profile ultra-shallow junctions by combining oxygen flooding and high depth resolution profiling. The surface secondary ion mass spectrometry technique which normally requires a magnetic sector instrument gets rid of the surface transient whereas the best depth resolution is more readily achieved using a quadrupole instrument. Two analyses are performed and the data are combined to accurately determine the depth distribution of ultra shallow dopants in silicon. Using optimized analytical conditions, the depth resolution is shown to be better than 0.7 nm. The analytical procedures described in this paper are being employed for the routine and commercial characterization of shallow dopants in silicon. © 1998 Elsevier Science S.A.

Keywords: Routine depth profiling; Secondary ion mass spectrometry; Shallow junctions

1. Introduction

The continuing efforts to increase device operating speed and to reduce power consumption is pushing integrated circuit design rules to the deep sub-micrometer regime. Shallow junctions formed by low energy ion implantation and rapid thermal annealing can be less than 20 nm deep. Since the in-depth distributions of these shallow dopants are critical to device performance, it is crucial to develop new analytical procedures to cope with the new challenges. Furthermore, in order for the methodology to be useful to the microelectronics industry, it must be simple and reliable because the industry cannot tolerate inconsistencies.

Secondary ion mass spectrometry (SIMS) is one of the most sensitive and widely used techniques to determine dopant distributions, for example, ion implants [1,2]. Unfortunately, in spite of its success in a research or analytical lab, SIMS is frequently regarded to be difficult and SIMS data are hard to interpret and even misleading according to engineers in the microelectronics industry. As the dimensions of integrated circuits become smaller, SIMS is becoming the only technique for many applications and more IC companies are inclined to putting SIMS instruments in a "fab environment". It is thus imperative that new analytical protocols be developed to transform SIMS into an easy-to-use and reliable technique, as most process engineers are not experts on materials characterization and analytical instrumentation. In fact, the ideal scenario is that recipes are generated and operators can follow them to obtain consistent and reliable results. One of the important areas needing SIMS in a fab is the depth profiling of ultra-shallow dopants. Using traditional SIMS procedures, the typical depth resolution is limited by ion mixing to about 10 nm. Depth resolution below 1 nm has been demonstrated in a research environment, and readers are encouraged to peruse the papers cited in Ref. [3] (pp. 1.2–2). In spite of the success, the complexity of the experimental protocols and the need for the operators to understand the fundamentals of the technique has hitherto hampered a wider application of SIMS to the depth profiling of ultra shallow junctions in microelectronics companies. To com-

* Corresponding author. Fax: +852 2788 7830; e-mail: appkchu@cityu.edu.hk

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plicate matters, ion yield changes in the top several nanometres of the samples due to ion beam equilibration during reactive ion sputtering (oxygen or cesium) make quantitative determination of dopant concentrations in this near–surface region inaccurate. The use of an oxygen leakage in conjunction with oxygen ion bombardment has been shown to be an effective way to get rid of this surface transient and ion yield variation [4]. We have modified the oxygen leak method to make it a routine technique (Surface–SIMS) to quantitatively measure surface dopants in a commercial environment [5]. By combining Surface–SIMS and an analytical procedure designed for high depth resolution, it is possible to quantitatively measure ultra-shallow dopants with depths less than 10 nm.

Depth resolution can be improved by using a lower bombardment energy, a more glancing incident angle, or a heavier primary ion. However, instrumental constraints, such as the coupling of the impact angle with the polarity of the primary/secondary ions in magnetic-sector instruments, make independent alteration of these parameters difficult [3]. On the contrary, in spite of the lower sensitivity and mass resolving power, some of these limitations can be circumvented in quadrupole SIMS instruments which, in principle, can deliver better depth resolution. In this paper, we will describe the analytical procedures developed for the routine and commercial depth profiling of shallow dopants by combining low energy sputtering and Surface–SIMS.

2. Experimental

The SIMS measurements were performed using CAMECA IMS-4f magnetic-sector ion microanalyzers and Physical Electronics 6600 quadrupole SIMS instruments. The oxygen leak experiments (Surface–SIMS) were conducted using CAMECA instruments. The standard operating conditions are: 3 keV O_2^+ net bombarding energy, 61° primary beam incident angle (from normal), 125 μm^2 primary beam raster, 20 nA primary ion current, ~20 μm beam size, 30 μ field aperture, 150 μm contrast diaphragm, 150 μm imaged field, and 2000–4000 mass resolving power. The dynamic transfer optics was not used because enhanced sensitivity was not needed in our experiments. For profiles obtained using the PHI quadrupole SIMS instruments, either 1 to 3 keV O_2^+ or 1 keV Cs^+ was employed.

The depth scales of the profiles were calibrated by measuring the crater depths using a Tencor P-10 profilometer with a precision of 0.2%. The accuracy of the Tencor P-10 is referenced to the Digital Instruments Nanoscope III atomic force microscope. The absolute accuracy of the Nanoscope III was confirmed by measuring a 5 nm muscovite mica standard.

3. Results and discussion

There are differences between the two common types of SIMS instrumentation used for depth profiling (dynamic SIMS). Table 1 highlights some of the major differences between magnetic-sector (CAMECA IMS-4f) and quadrupole based (PHI-6600) SIMS instruments. It is important to realize that the impact energy and angle can be independently varied in quadrupole instruments. In contrast, the effective incident angle in the CAMECA IMS-4f is dependent on the primary beam energy as well as the primary/secondary polarity (Fig. 1). The effective incident angle is given by the following relationship:

$$\sin\theta' = \sin\theta/\{1-E_s/E_p\}^{1/2}$$

where θ' is the effective impact angle, θ is the nominal angle of incidence (30° from normal), E_s is the sample voltage which is usually set at 4500 V, and E_p is the primary voltage. This inter-dependence limits the depth resolution. The situation is worse when the primary and secondary ion polarities are opposite, for example, when negative secondary ions are extracted while using a Cs^+ primary beam. The depth resolution limits achievable on a CAMECA magnetic sector instrument are displayed in Table 2 which summarizes the round-robin results of 25 independent laboratories worldwide [6]. It should be noted that the depth resolutions shown in

Table 1
Comparison of magnetic sector and quadrupole SIMS

Quadrupole	Magnetic sector
low transmission	high transmission
low mass resolution	high mass resolution
ion microprobe imaging	ion microprobe or ion microscope imaging
electron beam imaging	not available
electronic gating	aperture and electronic gating
0 to 100 V sample bias	4500 V sample bias
variable sample angle	fixed sample angle
primary beam energy and impact angle individually controlled	fixed relationship between primary beam energy and incident angle
incident angle 0° to 90°	incident angle 21° to 65°
primary energy 1 to 8 keV	primary energy 2 to 22 keV
odd shaped samples	flat samples

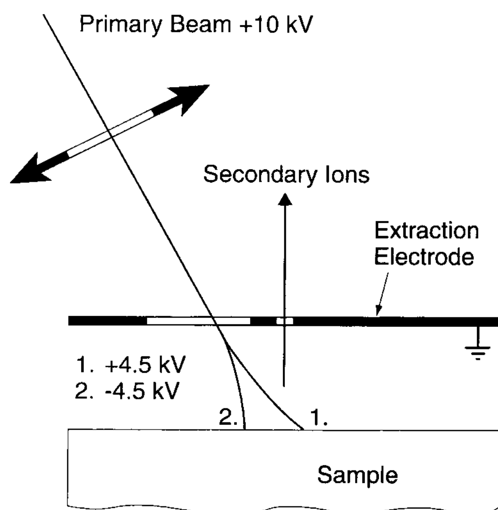

Fig. 1. Primary ion beam trajectories in the CAMECA IMS-4f for positive and negative sample voltages.

Table 2
Average round-robin results of depth resolution achieved on a CAMECA magnetic-sector instrument. A −4500 V sample voltage is assumed for Cs^+ bombardment and +4500 V for O_2^+ sputtering

Primary ion species	Net impact energy (keV)	Depth resolution (nm)
Cs^+	14.5	6
Cs^+	5.5	4
Cs^+	3	2.5
O_2^+	8	3–5
O_2^+	3	2

Table 2 are not the best found in the literature, but they indicate the values which can realistically and routinely be accomplished in an analytical laboratory or a commercial environment. It can be seen that even by bombarding with a 3 kV net O_2^+ primary beam and monitoring positive secondary ions, the best depth resolution is only 2 nm. In this respect, quadrupole-based instrumentation has an advantage because the net impact energy and angle can be individually adjusted, and the best depth resolution is more easily achieved on a quadrupole SIMS instrument.

The other major and relevant difference between these two types of instruments is the mass resolution. As hydrocarbon and water containing species are quite abundant on the surface and in the native oxide of silicon wafers, high mass resolution conditions are required to separate hydrocarbon and other interfering species from the elemental peaks. Hence, magnetic-sector instruments are better suited to profile the near-surface region.

Measuring shallow dopant profiles thus challenges two aspects of SIMS analysis: depth resolution and near-surface ion yield variation. In order to illustrate the problems encountered in traditional SIMS depth profiling, Fig. 2 shows the B and Si profiles acquired from a BF_2 ion implant using three net primary ion energies: 6, 3, and 1.5 keV. The analyses

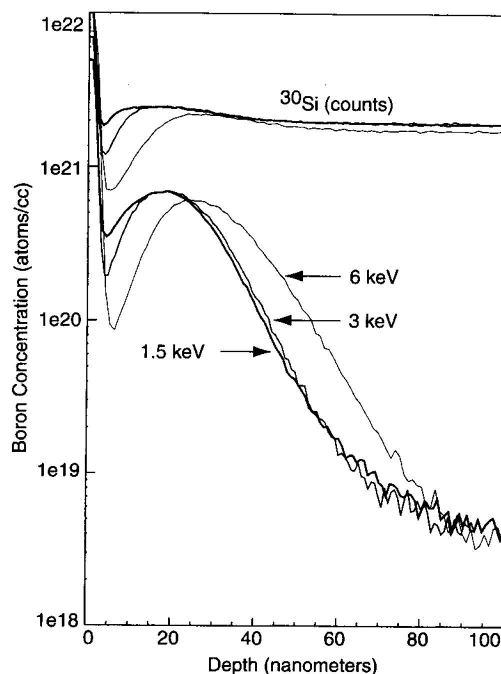

Fig. 2. SIMS profiles of B and Si in a 2×10^{15}, 20 keV BF_2 implant as a function of depth.

were carried out using a fixed bombardment angle on a quadrupole instrument. As the dominant mechanisms for the loss of depth resolution are recoil mixing or knock-on as well as cascade mixing, the primary ion impact energy plays an important role. Fig. 2 clearly illustrates that the profile acquired using 6 keV primary energy is inaccurate. Ion yield variation in the near surface region is also demonstrated in Fig. 2 (illustrated by the non-flat silicon profiles in the top 3 nm region). The initial high ion counts reflect high ion yields in the native oxide. Counts are then low as implanted primary oxygen ions gradually build up in the sample. The thickness of this transient is a function of the primary ion impact energy and is about 12 nm for 3 keV O_2^+. Although this effect is smaller at lower primary ion impact energy, accurate quantification in this transient region can be difficult. Methods that have been tried with limited success to correct for this artifact include point-by-point ratioing of the dopant profile to the matrix silicon ion profile, and deposition of an additional amorphous or polycrystalline silicon layer on the sample to physically shift the transient region away from the surface of interest [7,8].

Surface–SIMS analysis utilizing an O_2^+ primary ion beam while simultaneously flooding the sample surface with O_2 (O-flood or O-leak) provides a more direct and accurate technique for minimizing the transient changes. As shown in Fig. 3, in the presence of a sufficiently high oxygen gas pressure, the silicon surface is saturated with adsorbed oxygen and the secondary ions are emitted from a fully oxidized surface. The ion yields are independent of the amount of primary oxygen implanted into the sample during the sputtering process. We have modified the oxygen leak technique and introduced a rigorous analytical protocol called Surface–

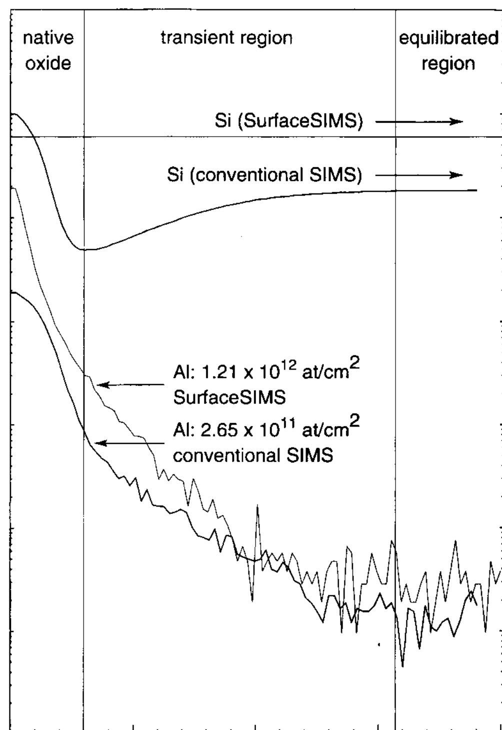

Fig. 3. Comparison of conventional SIMS and Surface–SIMS. The flat silicon profile verifies that the surface transient region is eliminated in Surface–SIMS.

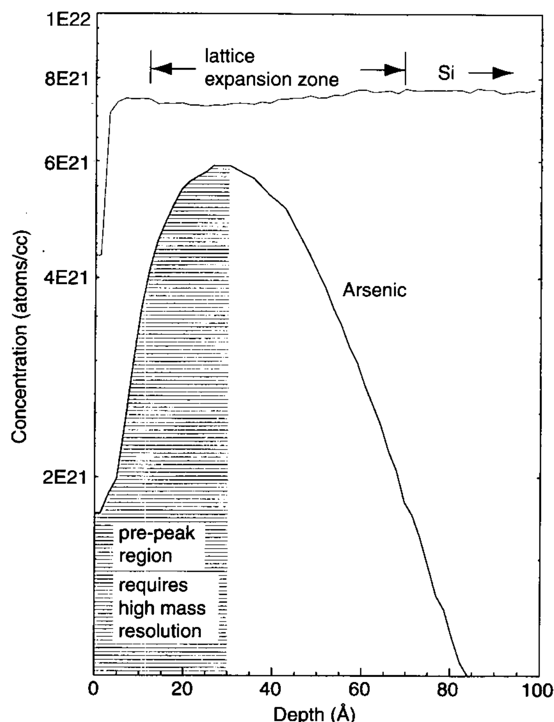

Fig. 4. Shallow arsenic profile showing the near-surface region which requires Surface–SIMS for accurate information.

SIMS as described in the experimental section. Recent research has confirmed the accuracy of Surface–SIMS measurements at depths less than 5 nm from the sample surface [9]. The long-term reproducibility of this Surface–SIMS measurement in a commercial environment has also been addressed [5].

In order to accurately measure a shallow dopant profile, two separate regions must therefore be considered. Using an arsenic profile as an illustration (Fig. 4), the pre-peak region which is about 30 nm from the surface requires Surface–SIMS analysis. Since mass interferences in the native oxide layer are quite abundant in this region, high mass resolution must be implemented, thereby necessitating the use of magnetic-sector instruments (Fig. 5). Even though positive secondary ions are monitored in Surface–SIMS analysis, it is worth mentioning that the phosphorus detection limit shown in Fig. 5 is actually quite good ($\leq 8\times10^{15}$ atoms cm^{-3}) due to the strong ion yield enhancement afforded by the oxygen flood. Also for illustration, Fig. 6 depicts the post-peak region where depth resolution is the primary issue. Three different bombarding angles are shown here to illustrate that better depth resolution is accomplished using a more glancing incidence (75°). Because the primary ion beam impact angle and the primary ion beam energy/secondary ion polarity are decoupled in quadrupole SIMS instruments, very low net bombarding energy (< 2 keV) and high glancing angle incidence are possible.

A question remains about the validity of the quadrupole SIMS data at depths less than 3 nm. Although not shown in

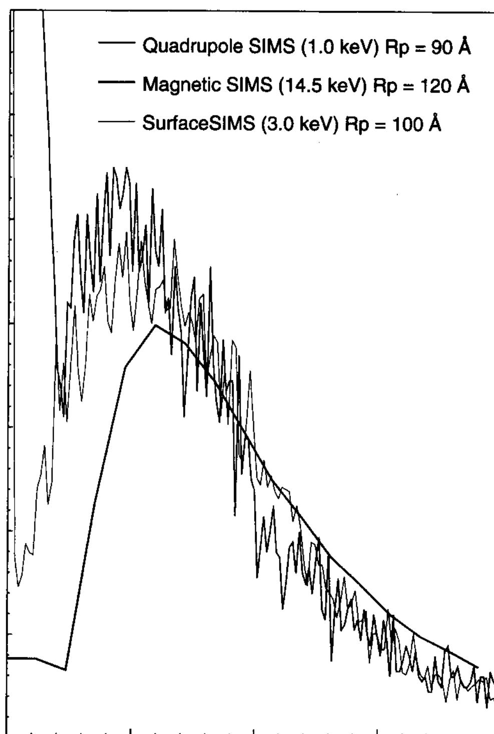

Fig. 5. SIMS profiles of P in a 3×10^{13} 10 keV implant as a function of depth. Note the high ion signal at the start of the quadrupole SIMS profile due to unresolved SiH mass interference and the manifestation of the surface transient region in the conventional magnetic-SIMS profile (low surface phosphorus signal).

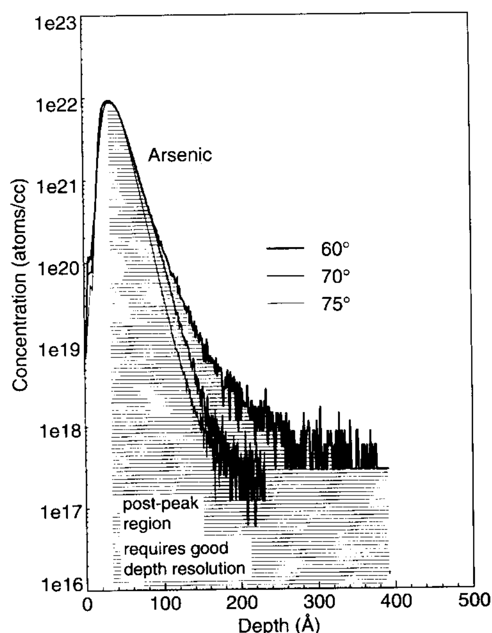

Fig. 6. Arsenic profiles acquired using a quadrupole SIMS instrument and different primary beam impact angles. Better resolution is achieved at a more glancing incidence.

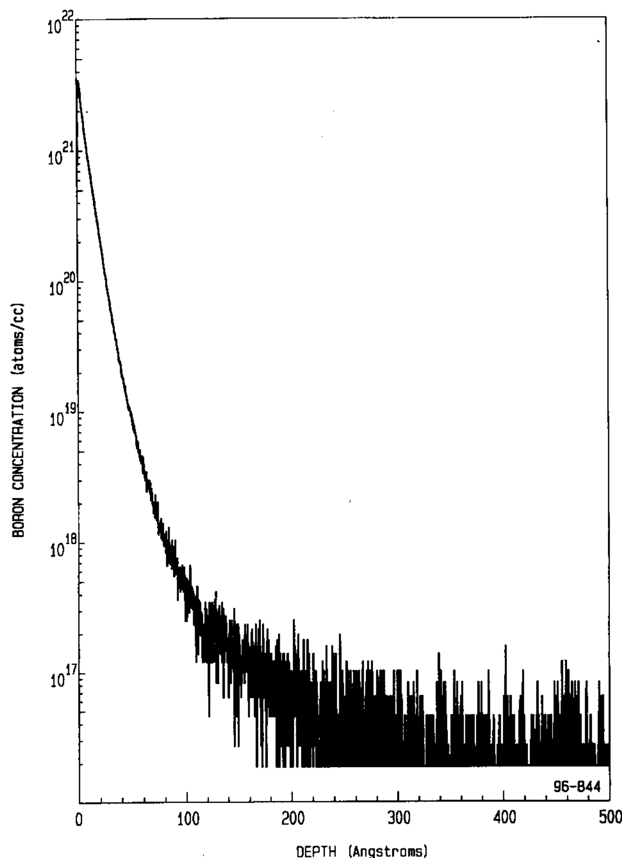

Fig. 7. B depth profile acquired from a 500 eV BF_2 implant by quadrupole SIMS (1 keV O_2^+, 75° incidence). The $1/e$ decay length is calculated to be 0.7 nm.

Fig. 6, it is found that, using Cs primary ion bombardment, the Si matrix ion signal is initially low (a factor of 2 or more), and does not become constant until a depth of roughly 5 to 10 nm. The arsenic data are typically point-by-point normalized to the Si matrix signal over this interval, even though the accuracy of this normalization remains unproven. Therefore, in order to completely characterize this shallow arsenic implant, both Surface–SIMS using a high mass resolution magnetic-sector instrument and high depth resolution depth profiling employing a quadrupole instrument are necessary.

In order to test our analytical procedures, a 500 eV BF_2 implant into pre-amorphized silicon has been chosen for this study because there are practically no mass interferences at mass 11, the nominal mass of boron. Hence, Surface–SIMS can be practiced on a quadrupole-based instrument. In this sample, the net implantation energy for boron is 500 eV × 11/49 = 125 eV. Fig. 7 shows the boron profile acquired using 1 keV O_2^+ (500 eV net energy for O^+), oxygen leak, and 75° incidence (from normal). The calculated $1/e$ decay length is 0.7 nm. As the real implanted boron distribution is not a delta function, the depth resolution achieved in this case is better than 0.7 nm. The shoulder at low concentration ($< 10^{18}$ atoms cm^{-3}) is due to a beam focusing problem at low primary ion energy and will be eliminated in next-generation quadrupole instrumentation.

We have combined Surface–SIMS and high depth resolution profiling to the determination of ultra-shallow dopants in silicon in a commercial environment. The Surface–SIMS conditions are well documented and easily implementable to an IC fab. Operating a quadrupole SIMS instrument using 1 keV oxygen or cesium primary ions at a 75° incident angle is also very straight-forward as most controls on such instruments are electronic and tend to be stable in everyday operation. The final depth profile is an overlay of the two sets of data, with Surface–SIMS showing the top 5 to 10 nm and high depth resolution quadrupole SIMS profiling revealing the rest of the profile. The requirement is that the fab must have two pieces of SIMS instrumentation, a magnetic instrument to perform Surface–SIMS analysis in the top 5 to 10 nm and a quadrupole SIMS instrument. However, as IC junctions are approaching 10 nm or less, SIMS appears to be the most reliable technique offering the required depth resolution and sensitivity. In this era of billion-dollar fabs, it is certainly a worthy price to pay.

4. Conclusions

To characterize ultra low energy ion implants or shallow dopant distributions, it is necessary to eliminate the surface transient artifact using the Surface–SIMS technique. High depth resolution is also important and can be achieved by using low sputtering energy (~1 keV) and high glancing angle. Quadrupole SIMS instruments can currently provide the best depth resolution for SIMS profiling, but in certain cases is limited by unresolved molecular ion interferences or by incompletely characterized transient equilibration effects.

Only Surface–SIMS appears to be unaffected by ion yield enhancements in the near-surface region. A combination of SIMS techniques is required for full characterization of ultra low energy ion implants. In cases where mass interferences are not an issue, for example, when profiling boron, the best depth resolution achieved on a quadrupole SIMS instrument is better than 0.7 nm. SIMS therefore remains the best and most reliable technique to profile extremely shallow dopant profiles.

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