Sputter Rate Change and Surface Roughening During Oblique and Normal Incidence O_2^+ Bombardment Of Silicon, With and Without Oxygen Flooding

Charles W. Magee[1], Gary R. Mount[2], Stephen P. Smith[2], Brad Herner[3] and Hans-J. Gossmann[3]
1. Evans East, Plainsboro, NJ 08536
2. Charles Evans and Associates, Redwood City, CA 94063
3. Bell Laboratories, Lucent Technologies, Murray Hill, NJ 07974

Abstract - A sample of low-temperature epitaxial Si grown with five B delta-doped layers 5.4nm apart has been profiled using secondary ion mass spectrometry under a variety of O_2 bombardment conditions. Energies from 400eV to 1.5keV were used with angles of incidence from 0° to 70°. Analyses were performed using oxygen flooding of the sample surface during analysis, as well as without using oxygen flooding. The apparent spacing between the B delta layers was used to determine the magnitude and extent of increased sputtering rate at the beginning of an analysis. The width of the B peaks was used to evaluate depth resolution and to measure the effect of surface roughening of the profiles. It was found that sputtering with 500eV O_2 at an angle of 50° while flooding with oxygen gave no measurable error in sputtering rate and resulted in no unexpected shift towards the surface of the B delta layers. These analysis conditions also resulted in depth resolution which was as good as that obtained using 400ev O_2 bombardment at 0° incidence. This 0° method of analysis, however, resulted in a 1.1nm shift of the topmost B delta layer towards the surface, and the 0° method had a sputter rate only 1/5 that of the 50° method of analysis.

I. Introduction

The recent need for measuring depth profiles for ultra-low energy ion implants in Si [1] has pushed the technique of secondary ion mass spectrometry (SIMS) into unprecedented degrees of high depth resolution and depth accuracy. This application requires accurate analyses within the top 1 to 10nm of the sample surface where the "surface transient" affects secondary ion yields. Flooding the Si surface with oxygen during analysis is an accepted method for reducing the magnitude of the surface transient effect [2]. However, a recent study by Wittmaack and Corcoran [3] suggests that flooding with oxygen when using 1.9keV oblique incidence O_2 bombardment results in an unavoidable change in sputtering rate for the first portion of the depth profile which artificially shifts features toward the surface by at least 4.5nm compared to profiles obtained without O flooding. In another recent study, Jiang and Alkemade [4], substantiated the claims of Wittmaack and Corcoran, in that Jiang and Alkemade also found a differential shift of the profile towards the surface. They used 1keV O_2 bombardment instead of 1.9keV. The weakness of the Jiang and Alkemade experiment lay in the rather wide spacing of the delta layers (11.9nm) as well as the fact that different energies and angles of primary ion incidence were not tried. They could only find that the depth of sputter rate change extended less than 11.7nm down from the Si surface.

This present study is an extension of the Jiang and Alkemade work. It involves using a similar MBE sample with internal marker layers but with only 5.4nm spacing. We use these markers to determine, unambiguously, enhancements in initial sputter rates and the depths to which the enhanced initial sputter rates extend. Most importantly, we have investigated a variety of energy and angle-of-incidence combinations and have found several sets of conditions of oblique incidence with oxygen bombardment which show negligible artificial shifts toward the surface.

II. Experimental

Boron markers layers were grown in Si by low temperature molecular beam epitaxy [5] on Si(100) at a temperature of 250°C. Each spike was produced by deposition of elemental B to a coverage of $4x10^{12}/cm^2$, followed by Si growth at a rate of 0.02nm/sec to a thickness of 5.4nm. The cycle was repeated five times, hence the shallowest spike is only 5.4nm beneath the surface. The Si evaporation rate was calibrated during the buffer layer growth by measuring the period of RHEED intensity oscillations. The accuracy is better than 1%. The drift in evaporation rate is = 1% per 100nm and is negligible in the present structure. X-ray photoelectron spectrometry (XPS) measurements of the native oxide revealed its thickness to be approximately 0.4nm. There is an enhanced sputter rate associated with sputtering through the surface oxide, but this phenomenon is not discussed in this work because the native oxide is so thin.

The SIMS experiments were performed on quadruple-based secondary ion mass spectrometers (PHI 6650, as well as PHI 6600), both modified for routine use of an oxygen flood. The pressure during O-flooding was approximately $2x10^{-6}$ torr. Primary ion beam energies from 0.4keV to 1.5keV were used, and angles of incidence were varied between 0° and 70° with respect to the surface normal, although only a subset of all of these results is shown here. It is important to note that the grounded sample and the low extraction field above the sample surface of a quadrupole-based SIMS instrument makes the angle of incidence independent of the primary ion energy. Thus, our experiments could be performed where both of these variables can be controlled and known accurately.

III. Results and Discussion

Fig. 1 shows the B profile from the five B delta layer sample analyzed using 1.0keV O_2 bombardment at an angle of incidence of 60° without oxygen flooding (pressure during analysis: 1E-9 torr). The sputtering rate for this analysis (and for virtually all of the other analyses) is determined by assuming that the spacing between the deepest three delta layers is exactly 3 x 5.4nm, or 16.2nm. As was found previously [4], a constant sputtering rate was obtained without O flooding as deduced from the equal spacing between all five of the B deltas. Jiang and Alkemade [4] found, however, that 1keV O_2 bombardment at 60° with O flooding resulted in an increase in the sputter rate somewhere within the top 11.7nm of the profile. Fig. 2 shows the B profile from our sample taken under these same conditions. Our results exactly confirm the Jiang and Alkemade results. Their first Ge delta (11.7nm deep) was measured 2.6nm too shallow. Our second B delta (10.8nm deep) is measured 2.5nm too shallow. The closer spacing of the deltas in our sample, however, show that 2/3 of the error in the depth of a delta 10.8nm deep comes in the first 5.4nm. Some of the spacing error is due to increased sputter rate through the surface oxide while using oxygen flooding.

The oxide on our sample was only 0.4nm thick and was expected to contribute a maximum of 0.2nm error in the first delta layer. Our data also show that there is no change in sputter rate after sputtering 10.8nm.

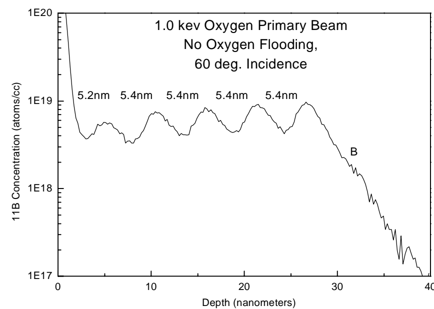

Fig. 1. B profile of the five-delta layer sample. Analysis conditions: 1keV O_2 bombardment at 60°, no oxygen flooding.

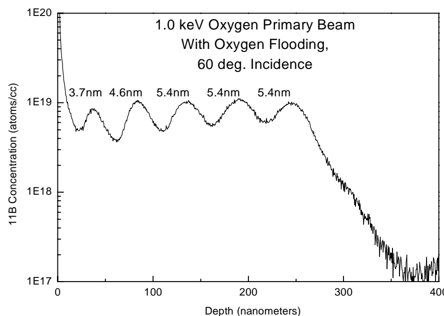

Fig 2. B profile of the five-delta layer sample. Analysis conditions: 1keV O_2 bombardment at 60°, with oxygen flooding.

Jiang and Alkemade failed to explore different angles of incidence and energies, however. Fig. 3 shows the B profile taken using 1keV O_2 bombardment at 50° with oxygen flooding. The spacing of the delta layers shows that only a very small increase in sputtering rate is detected in the top 5.4nm of the profile. The top B delta is measured only 0.8nm too shallow. The equal spacing between all subsequent deltas shows that the sputtering rate has stabilized after sputtering 5.4nm or less. This shift towards the surface of only 0.8nm is much less than that reported by Wittmaack and Corcoran who report a shift of 4.5nm. We have found that the shift only increases to 1.1nm when using 1.5keV O_2 at 60° with oxygen flooding.

The B peaks in the profile of Fig. 3 measured at 50º are also sharper than when measured at 60°. This is undoubtedly due to a reduction in the ripple formation from sputtering which was shown by Jiang and Alkemade to exist when sputtering at 60°. The reason for the change in ripple formation is still unclear despite its many studies [6-9].

In an effort to achieve higher depth resolution, we felt that primary ion energies of less than 1keV would be necessary for profiling the very shallowest profiles. We thus performed a series of studies using 500eV O_2 bombardment. Figures 4a and 4b show the B profiles obtained on our five delta sample using 500eV O_2 bombardment with an angle of incidence of 60°, (a) without oxygen flooding and (b) with oxygen flooding. The profile obtained without O-flooding, 4a, shows roughly even spacing of the top three B delta layers. However, after sputtering deeper than this, severe surface roughening sets in and the depth resolution degrades rapidly and the sputtering rate decreases resulting in apparent spacing of greater than 5.4nm. This is as predicted in references 7 and 8.

The profile taken with oxygen flooding shows a remarkable increase in depth resolution for the deeper B peaks which we have shown previously [10] to be due to a decrease in the surface roughening as measured by atomic force microscopy (AFM). Note, however, that the 60° angle of incidence still

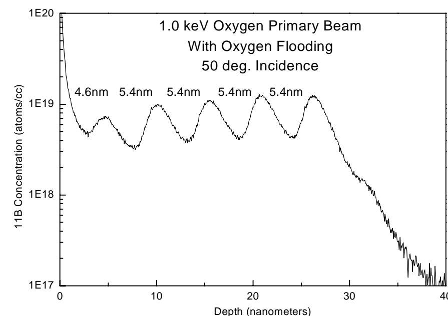

Fig 3. B profile of the five-delta layer sample. Analysis conditions: 1keV O_2 bombardment at 50°, with oxygen flooding.

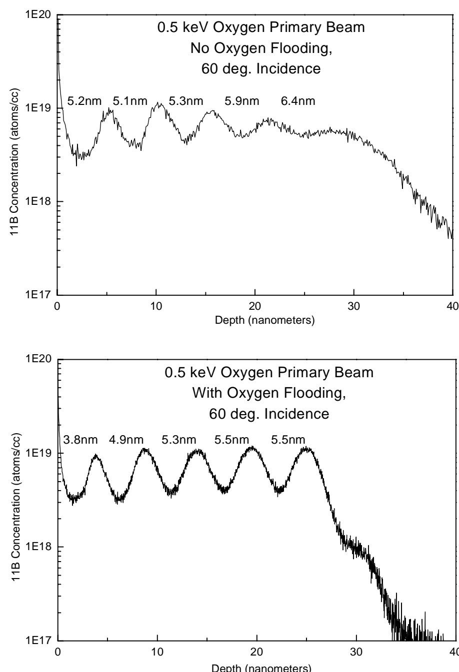

Fig 4. B profile of the five-delta layer sample. Analysis conditions: (a) 0.5keV O_2 bombardment at 60°, no oxygen flooding, (b) 0.5keV O_2 bombardment at 60°, with oxygen flooding.

results in the same decrease in delta spacing (increase in sputter rate) in the first two delta layers that was measured for 1keV bombardment. Some significant degree of surface roughening is still detected by AFM , as well. This is pointed out by several authors [11, 12] as being the reason why oxygen flooding with oblique incidence oxygen bombardment cannot be used for shallow junction profiling. The results in Fig. 5 show that this judgment is, in fact, incorrect, and was due to the authors' incomplete study of the incidence angle and energy parameter space over which the analyses can be made.

The profiles in Figs 5a and 5b show the B profiles again taken using 500eV O_2 bombardment, but the angle of incidence has been reduced to 50°. Figure 5a shows the results when O-flooding is not used. Unlike the 60° case, moderate depth resolution is maintained throughout the entire analysis, although AFM analysis shows considerable surface roughness has developed in the sputtered crater [10]. Note also that, for the first time when *not* using O-flooding, there is a 1.7nm shift toward the surface (delta spacing too small) of the topmost B delta.

The profile is changed dramatically by the addition of O-flooding under these bombardment conditions. Figure 5b shows that the B peaks are now extremely sharp which indicates that surface roughening has been reduced to an insignificant level. This has been confirmed by AFM analysis [10]. The depth resolution is now sufficiently good to resolve the small B peak at a depth of 33nm which was, heretofore, only detected as a shoulder on the falling B profile of the last delta (Fig. 5a). Perhaps more importantly, the spacing between *all* of the B delta layers is 5.4nm to within experimental error and accounting for increased sputter rate in the 0.4nm oxide, which shows that no unexpected increase in sputter rate exists at the beginning of the profile, and that no sputter roughening induced "differential shift" is present. In fact, as shown in Fig. 6, a profile acquired on our five B delta sample using 400eV O_2 bombardment and 0° incidence without O-flooding, as is recommended in references [3, 11, and 12], is actually *worse* than the profile shown in Fig. 5b, because the non O-flood, 0° analysis has a differential shift towards the surface of 1.1nm. The depth resolutions of the two analyses are comparable.

The unique nature of the sample used in this study makes it possible to re-evaluate the errors due to oxygen flooding which have been reported previously on less well known samples. The study described in [3] shows a "differential shift" of at least 4.5nm when oxygen flooding is used when sputtering with 1.9keV O_2^+ at an angle of 52°, as compared to an analysis made under the same sputtering conditions without oxygen flooding. Not only did [3] show a shift in the oxygen-flooded profile of 4.5nm toward the surface due to a sputtering rate change in the flooded profile, but the slopes of two profile were different over a depth approaching 20nm which suggested that the change in sputter rate persisted over such a depth. Compared to the non-flooded profile, the oxygen-flooded profile also appeared to have better depth resolution. The authors attributed this apparent improvement entirely to the artifact of the sputtering rate change. Fig. 7

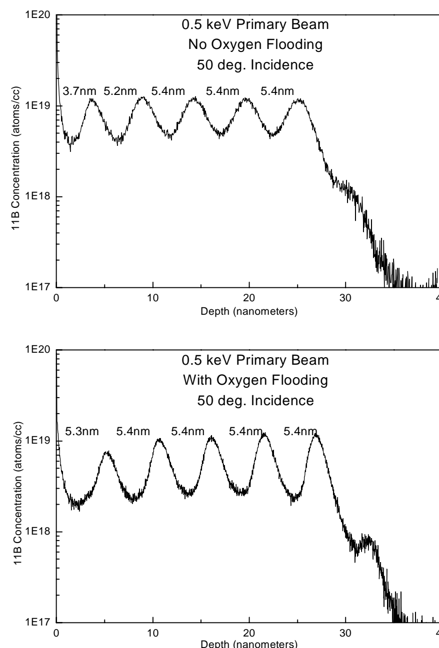

Fig 5. B profile of the five-delta layer sample. Analysis conditions: (a) 0.5keV O_2 bombardment at 50°, no oxygen flooding, (b) 0.5keV O_2 bombardment at 50°, with oxygen flooding

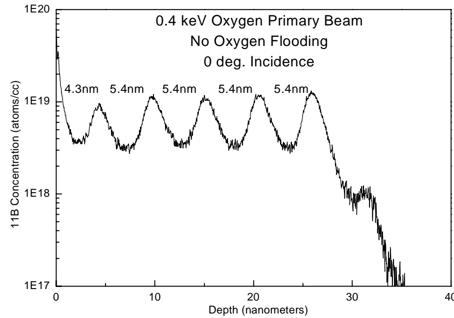

Fig 6. B profile of the five-delta layer sample. Analysis conditions: 0.4keV O_2 bombardment at 0°, no oxygen flooding.

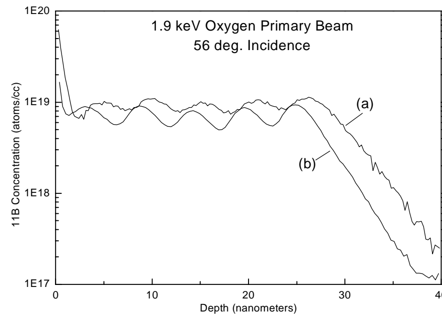

Fig. 7. B profiles of the five-delta layer sample taken using the sputtering conditions noted in [3]: 1.9keV O_2 bombardment, 56° with respect to the surface normal. (a) no oxygen flooding; (b) with oxygen flooding.

shows B profiles taken on our 5.4nm B delta layer sample using the same sputtering conditions as noted in [3]. The results show unambiguously that the above-noted assertions made in [3] are incorrect.

The data in Fig. 7 show that the differential shift toward the surface of the oxygen flooded profile is only 1.6nm, not the 4.5nm noted in [3]. What is more, all of the shift occurs within the topmost 5.4nm of the sample, not 20nm as suggested in [3]. The oxygen-flooded profile also shows an improvement in the depth resolution as shown in the improvement in the sharpness of the B delta layers, and by the more steeply falling profile after the last delta layer is sputtered through. The fact that the spacing *between* the delta layers is the same for both the oxygen flooded and the non-flooded analyses shows that the improvement in depth resolution is *real,* and not due to a sputter rate change as stated in [3].

The difference between our data and those shown in [3] may be due to the difficulty in knowing the precise angle of incidence of the primary ion beam in a magnetic sector instrument such as that used in [3]. The authors of [3] cite the previous work which was used to calculate their angle of incidence, but the exact path of the ion beam through the high extraction field above the sample surface may result in some errors when using the cited equation. Our work here shows that a relatively small change in angle of incidence of only 10° can have an enormous effect on the resultant profiles, so it is reasonable to hypothesize that the angle of incidence noted in [3] may not have been correct.

IV. Conclusion

This study on a very simple sample has shown definitively that oblique incidence O_2 bombardment with oxygen flooding can be used to obtain accurate, high depth resolution shallow junction profiles in Si, but care must be exercised in choosing the experimental conditions. Not all sputtering conditions will result in error-free profiles. Surface roughening during sputtering plays a major role in determining the ultimate outcome of any given analysis, and relatively small changes in sputtering conditions and lead to large differences in results. The need to study exhaustively the entire parameter space of the analysis cannot be overemphasized. Without doing so, one can make inaccurate conclusions.

References

[1] Proceedings of the Fourth International Workshop on the Measurement and Characterization of Ultra-Shallow Doping Profiles in Semiconductors, J. Vac. Sci. Technol. B 16(1), 1998, p. 259.

[2] W. Vandervorst and F.R. Shepherd, J. Vac. Sci. Technol. A 5(3), 1987, p.313.

[3] K. Wittmaack and S.F. Corcoran, J. Vac. Sci. Technol. B 16(1), 1998, p. 272.

[4] Z.X. Jiang and P.F.A. Alkemade, Secondary Ion Mass Spectrometry, SIMS IX, eds. G. Gillen, R. Lareau, J. Bennett and F. Stevie, John Wiley and Sons, 1997, p. 431.

[5] H.-J. Gossmann and E.F. Schubert, Crit. Rev. in Solid State and Mater. Sci. 18, 1993, p.1.

[6] F.A. Stevie, P.M. Kahora, D.S. Simons and P.Chi, J. Vac. Sci. Technol. A 6(1), 1988, p. 76.

[7] W. Wittmaack, J. Vac. Sci. Technol. A 8(3), 1990, p. 2246.

[8] K. Elst, W. Vandervorst, J. Alay, J. Snauwaert and L. Hellemans, J. Vac. Sci Technol. B 11(6), 1993, p. 1968.

[9] J.J. Vajo, R.E. Doty and E.-H. Cirlin, J. Vac. Sci. Technol. A 14(5), 1996, p. 2709.

[10] G. Mount, S. Smith, C. Hitzman, V.K.F. Chia, C. Magee and H.-J. Gossmann, Proceedings of the 1998 Int. Conf. On Characterization and Metrology for ULSI Technol., March 23-27, 1998, Gaithersburg, MD., paper TH-6.

[11] M.G. Dowsett, G.A. Cooke and D.P. Chu, Proceedings of the 1998 Int. Conf. On Characterization and Metrology for ULSI Technol., March 23-27, 1998, Gaithersburg, MD., paper TH-9.

[12] K. Wittmaack, Proceedings of the 1998 Int. Conf. On Characterization and Metrology for ULSI Technol., March 23-27, 1998, Gaithersburg, MD., paper TH-39.