DEPTH DISTRIBUTIONS OF LOW ENERGY DEUTERIUM IMPLANTED INTO SILICON AS DETERMINED BY SIMS

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ABSTRACT

Secondary ion mass spectrometry (SIMS) has been used to determine depth profiles of deuterium implanted into single crystal silicon targets at energies between 0.1 and 5 keV. The atomic mixing inherent in the sputtering process, which directly affects depth resolution, has been reduced by using a bombarding particle of low energy and high Z impacting the sample at a large angle relative to the surface normal (3 keV, Cs^+, impacting at 60°). Using this procedure, depth resolution of 20 Å at a depth of 800 Å has been obtained in depth profiling of Ta_2O_5 on Ta. Mean projected range and straggling of the implant profiles are in good agreement with calculations when irradiations are performed at 11° from the normal to the (100) plane to prevent channeling. The saturation density of trapped deuterium has also been determined to be 1.4×10^{22} D/cm^3.

1. INTRODUCTION

It has been proposed[1,2] that by measuring hydrogen and deuterium depth profiles in materials exposed to tokamak discharges, the flux and energy of hydrogen isotopes hitting the vacuum vessel wall and limiters of the plasma machine could be deduced. Of critical importance is a knowledge of ranges for H and D at appropriate energies in the material exposed to the discharge. This paper presents secondary ion mass spectrometry (SIMS) measurements of deuterium depth profiles in silicon for the energy range 0.1 to 5 keV, and results of numerical calculations of mean projected range and straggle for D implanted into Si at energies between 20 eV and 10 keV.

A possible problem exists with the SIMS analysis which is related to the atomic mixing process inherent in sputtering. At the energies of interest in the tokamak plasma edge the range for D in Si may be as short as tens of Angstroms. To obtain accurate depth profiles of species with such small ranges, the atomic mixing process must be confined to a depth less than the range of the implanted ions. This work includes an examination of the sputtering process with special emphasis on how to minimize the thickness of the surface layer damaged by the primary ion beam.

2. BASIC SPUTTERING CONSIDERATIONS

When a keV ion beam collides with a solid surface, the vast majority of bombarding ions penetrate the solid and transfer their energy to the lattice in a series of collision cascades. The energetic recoil atoms initiate secondary and tertiary collision cascades, some of which produce sputtering. The parameter most critical to good depth resolution is the primary ion penetration depth, as measured perpendicular to the sample surface.

The mean projected range of the primary ion is directly dependent on the energy of the particle. The higher the energy of the primary ion, the thicker will be the layer of lattice disorder. The effect of energy on ion beam induced mixing has been studied using Rutherford backscattering spectrometry (RBS)[3] and has been shown to affect the depth resolution of SIMS depth profiles (4). For this reason, the normal 5 keV primary ion energy was lowered to 3 keV. The primary ion beam could not be focussed well below this energy.

The mean projected range of the primary particle will also be dependent on its atomic number (Z). This is illustrated by using LSS theory to calculate values of the mean projected ranges in silicon for oxygen (Z = 8), argon (Z = 18), and antimony (Z = 51) at 10 keV. The respective ranges are 225 Å, 122 A° and 84 Å. Therefore, we decided to use a surface ionization cesium (Z = 55) ion source which had been developed for the RCA SIMS instrument.[5]

Furthermore, the thickness of the damaged surface layer can be lessened if the primary ions impact the sample at a large angle relative to the surface normal. For the SIMS instrument used in this work[6] the angle of incidence is 60^{o} which should result in a damaged layer a factor of two thinner than the mean projected range of a normally incident ion. Under the experimental conditions used for this work--3 keV Cs^{+} impacting at 60^{o}-- the damaged surface layer in Si is expected to be about 18 Å. By contrast we note that many ion microprobes use 20 keV oxygen ions impacting at 0^{o} which results in a damaged layer of >400 Å in Si.

The instrumental degradation of depth resolution from crater edge effects was reduced in this work through the use of ion beam rastering with signal-gating, and stigmatic secondary ion optics. Under these conditions, the depth resolution obtainable should be of the order of the thickness of the damaged surface layer. To test this, a depth profile was made of a Ta_2O_5 film anodically grown on a β-Ta film which had been deposited on a polished glass substrate. The tantalum oxide film thickness was determined by ellipsometry to be 770 Å. A portion of the $^{18}O^-$ depth profile from 700 Å to 800 Å is shown in Fig. 1. The 2σ (84% to 16%) falloff of the oxygen signal gives a depth resolution d, of 20 Å. The depth resolution in silicon is expected to be ~20% worse due to the fact that the mean projected range of the bombarding ions is larger in Si than in Ta_2O_5, but the example does still show how under these conditions of ion bombardment the atomic mixing process can be reduced and good depth resolution obtained.

3. EXPERIMENT AND RESULTS

Single crystal silicon samples were irradiated with monoenergetic deuterium ions from a hot cathode, low energy spread ion source.(7) The beam of deuterium ions was mass-analyzed by a modified Wien filter.(8) Fluences were measured without secondary electron suppression. The background pressure during irradiation was 10^{-7} torr D_2 and 10^{-8} torr of CO, CH_4, and H_2O.

Single-crystal silicon was used as a sample for all irradiations because of its high purity, low hydrogen content, high trapping efficiences, smooth surface, and because calibrated primary standards(9) of H and D in Si were available. These primary standards were used to calibrate secondary standards of H and D implanted into Si at RCA. The depth scales on all profiles were determined by measuring (with a profilometer) the depth of the crater sputtered during the analysis of the implanted standard. Sputtering rates using the Cs^+ ion source are stable to ± 2%/day. Several instrumental features are necessary for profiling H and D in solids and have been described previously.[10] Since under cesium bombardment negative secondary ions predominate, the species monitored in this study was $^2D^-$.

The range R measured for several low dose implants at normal incidence was deeper than predicted by the theory described in section 4. This discrepancy can be explained if channeling of the incoming D ions to greater depths occurs. To check this hypothesis, (100) silicon was implanted with D^+ ions at angles of incidence of 0^o to 11^o. The resultant D profiles for a 2.5 keV implant are shown in Fig. 2. The 0^o implant distribution lies deeper than that of the 11^o sample by ≈ 150 Å and has a full-width at half-maximum approximately 100 Å larger than the profile of the 11^o sample. For a 5 keV D^+ implant the difference in range between the 0^o and 11^o irradiations was even larger (420 Å). Differences in H range in Si due to channeling have been seen previously[11] at higher energy.

A series of D implants at 11^o was made at the following energies: 100, 250, 500, 1250, 2500, and 5000 eV. The fluences were kept sufficiently low to insure that saturation would not occur. Several of the SIMS depth profiles are shown in Fig. 3. The straggle (defined in this work as the half-width at half-maximum) and range for all implants were measured and are shown in Fig. 4. The depth profiles of 100 eV and 250 eV implants did not show as clearly defined peaks as did the higher energy implants. We expect that this is due to the larger straggle-to-range ratio at low energy. The large error bars on the low range side of the low energy implants is in part due to the definition of range which is discussed in Ref. 2. The depth profiles in Figures 2 and 3 are not symmetrical Gaussians. We note this because Gaussian profiles have been used in Ref. 2.

The depth profiles all show a finite deuterium concentration at the surface. For this to occur, one of two conditions must be met. Either there must be an activation energy required to escape the crystal, or trapping must occur at defects rather than simply at the end of the range. These considerations lead us to propose that these deuterium profiles, at least in part, must represent damage profiles in addition to end-of-range profiles.

It has been proposed[2,12] that at high fluence, silicon will saturate with hydrogen to a certain depth (dependent on incident energy) and will have a flat profile from the surface into this depth, but will then show a decreasing hydrogen concentration as the depth increases. This behavior has been observed at higher bombarding energies.[13] Saturation levels form the basis of one method[12] of determining energies of hydrogenic species at the tokamak plasma edge. While it is not the purpose of this paper to elaborate on that method, the saturation phenomena is of sufficient interest that we present the first high resolution data here. Two silicon samples were implanted with 4 keV D_2^+ to fluences of approximately 1×10^{16} and 1×10^{18} at/cm^2. Deuterium depth profiles of the two samples are shown in Fig. 5. One can see how the profile of the high-dose sample is saturated at approximately 1.4×10^{22} at/cm^3 to a depth of 700 Å, the mean projected range of the low-dose sample. This maximum concentration is in good agreement with predictions[2].

4. CALCULATIONS OF RANGE AND STRAGGLING

Thompson et al.[14] have compared disorder distributions predicted by Monte Carlo calculations with those observed experimentally for H^+ incident on silicon at energies between 10 keV and 80 keV. From these comparisons they have concluded that in this energy range the electronic contribution to the stopping power can be represented at $kE^{0.5}$ where k has a value 1.55 times that predicted by Lindhard theory.[15] In the present work we have used this electronic contribution to dE/dx, the Lindhard version of the Thomas-Fermi elastic scattering cross section,[15] and the methods described in Reference 16 to calculate the average projected range, R, and its straggling

(HWHM) S, for 20 eV to 10 keV D^+ incident on amorphous (or randomly oriented) silicon. The results of these calculations are shown as the lines labeled R and S, respectively, in Fig. 4. The results agree well with the experiment.

5. SUMMARY AND CONCLUSIONS

To obtain sufficiently good depth resolution to measure the small deuterium ion ranges encountered in this study, SIMS techniques were improved to reduce the thickness of the surface layer damaged by the primary ion beam to $\lesssim$ 25 Å. This was accomplished by bombarding with 3 keV Cs^+ ions at a 60° angle of incidence.

With these improvements depth profiles measurements were made for 0.1 to 5 keV D implantations into Si. The agreement with calculations gives encouragement that the calculated ranges at lower energy are also accurate. The good depth resolution will enable more accurate comparisons of depth and damage profiles with theories and will allow the measurement of hydrogen energies in tokamaks over the range 0.1 to 10 keV with an energy resolution of ~ 10%.

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FIGURE CAPTIONS

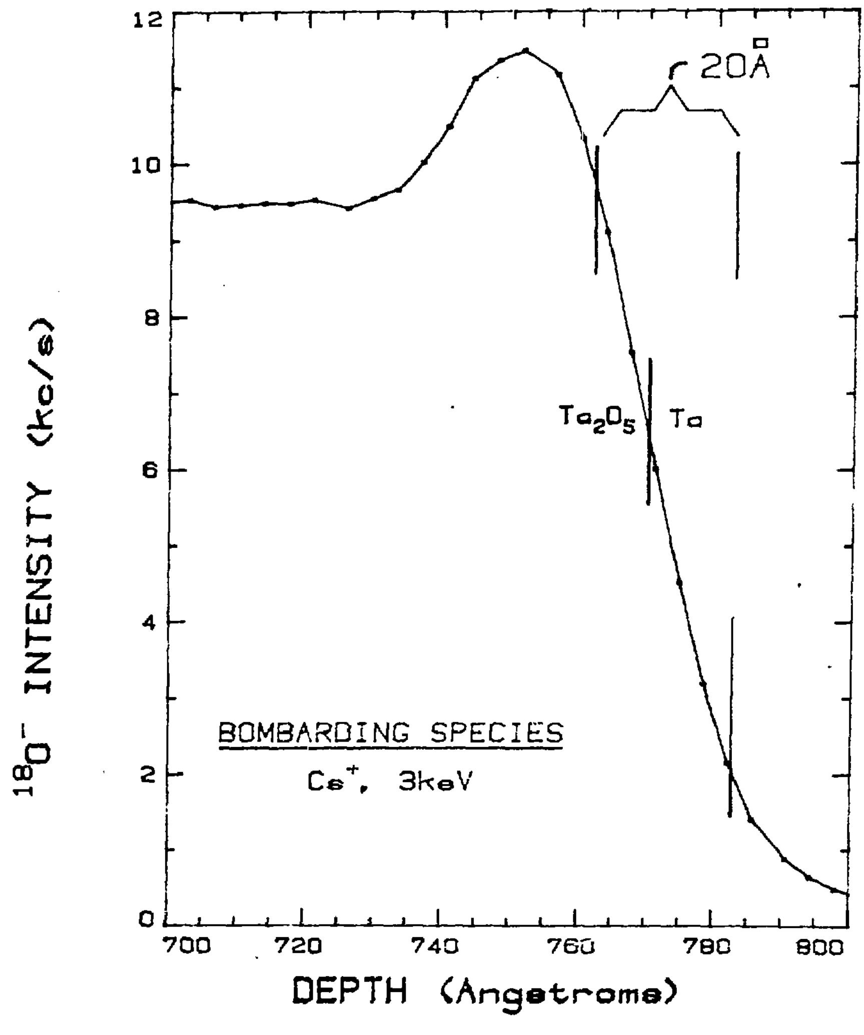
Fig. 1. Portion of a $^{18}O^-$ depth profile of a 770 Å Ta_2O_5 film on Ta used to evaluate depth resolution. The 2σ (84% to 18%) fall-off of the oxygen signal is 20 Å. The rise in the $^{18}O^-$ signal at 740 Å is probably due to sputtering rate change just prior to the interface causing a rise in the implanted Cs concentration, thus increasing the sensitivity for oxygen. The naturally occurring ^{18}O was monitored because the count rate for ^{16}O was too high to be measured reliably.

Fig. 2. Deuterium depth profiles of two (100) silicon samples ion implanted with 2.5 keV D at 0^o and 11^o off the surface normal.

Fig. 3. Depth profiles of D implanted into Si at 750, 2500 and 5000 eV.

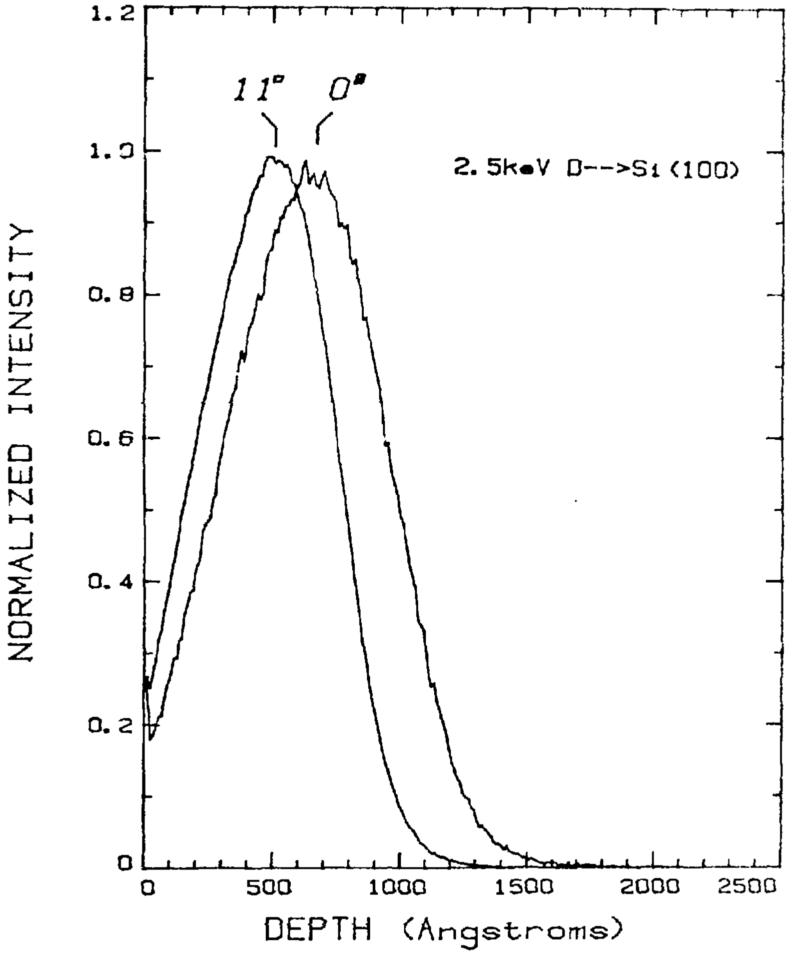
Fig. 4. Calculated mean projected range, R, and straggling, S, for deuterium incident on silicon. S is defined as the half width at half height. Data points are from this work: □ range; + straggling. For energies of 500 eV and above, the uncertainty in the SIMS measurements are approximated by the size of the data points.

Fig. 5. Depth profiles of two (100) silicon samples ion implanted with 2 keV D to fluences of: a) 10^{18} at/cm^2; b) 10^{16} at/cm^2. The sputtering rate, hence the depth scale, may be slightly different in the saturated portion of profile a) from that in the unsaturated portion.



12
10
8
6
4
2
0
700
720
740
760
780
800
DEPTH (Angstroms)
18O- INTENSITY (kc/s)
20Å
Ta2O5
Ta
BOMBARDING SPECIES
Cs+, 3keV

Figure 1



NORMALIZED INTENSITY
DEPTH (Angstroms)
2.5keV D-->Si (100)
11°
0°
1.2
1.0
0.8
0.6
0.4
0.2
0
0
500
1000
1500
2000
2500

Figure 2

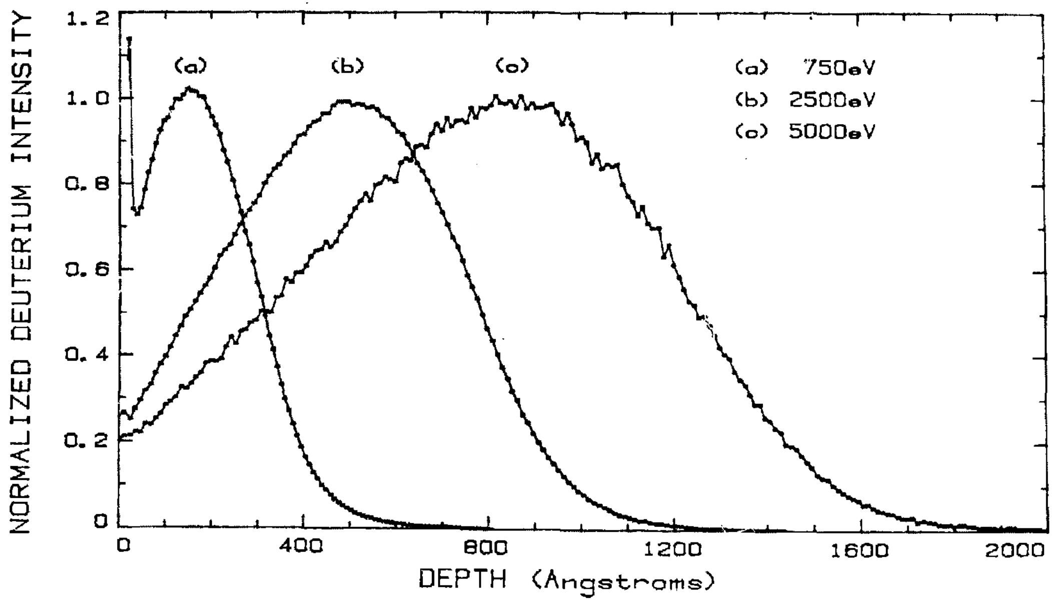

NORMALIZED DEUTERIUM INTENSITY
1.2
1.0
0.8
0.6
0.4
0.2
0
(a)
(b)
(c)
(a) 750eV
(b) 2500eV
(c) 5000eV
0
400
800
1200
1600
2000
DEPTH (Angstroms)

Figure 3

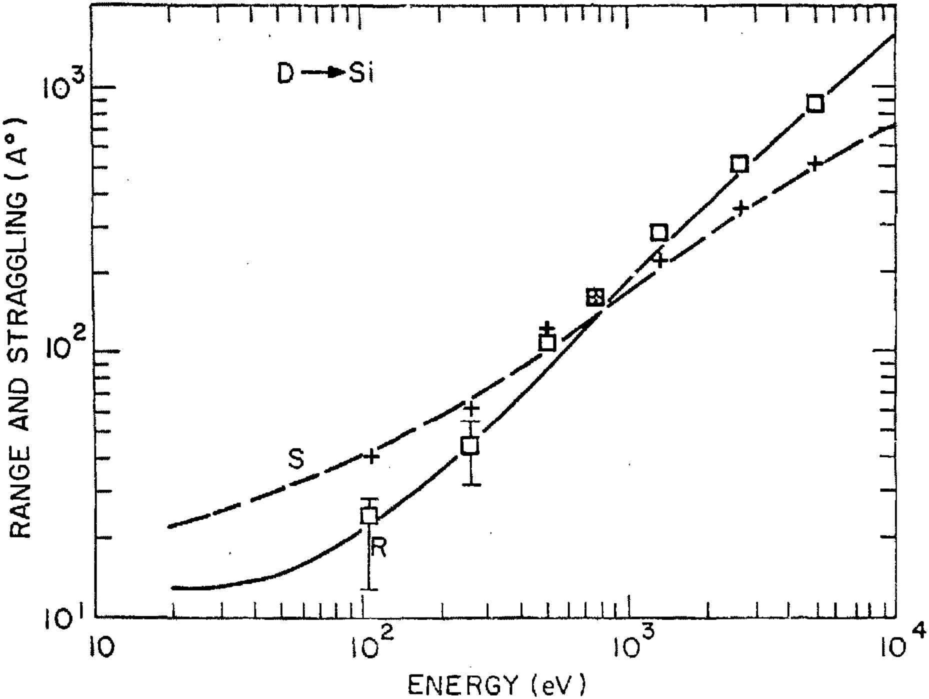

D → Si
RANGE AND STRAGGLING (A°)
ENERGY (eV)
S
R
10
10²
10³
10⁴
10¹
10²
10³

Figure 4

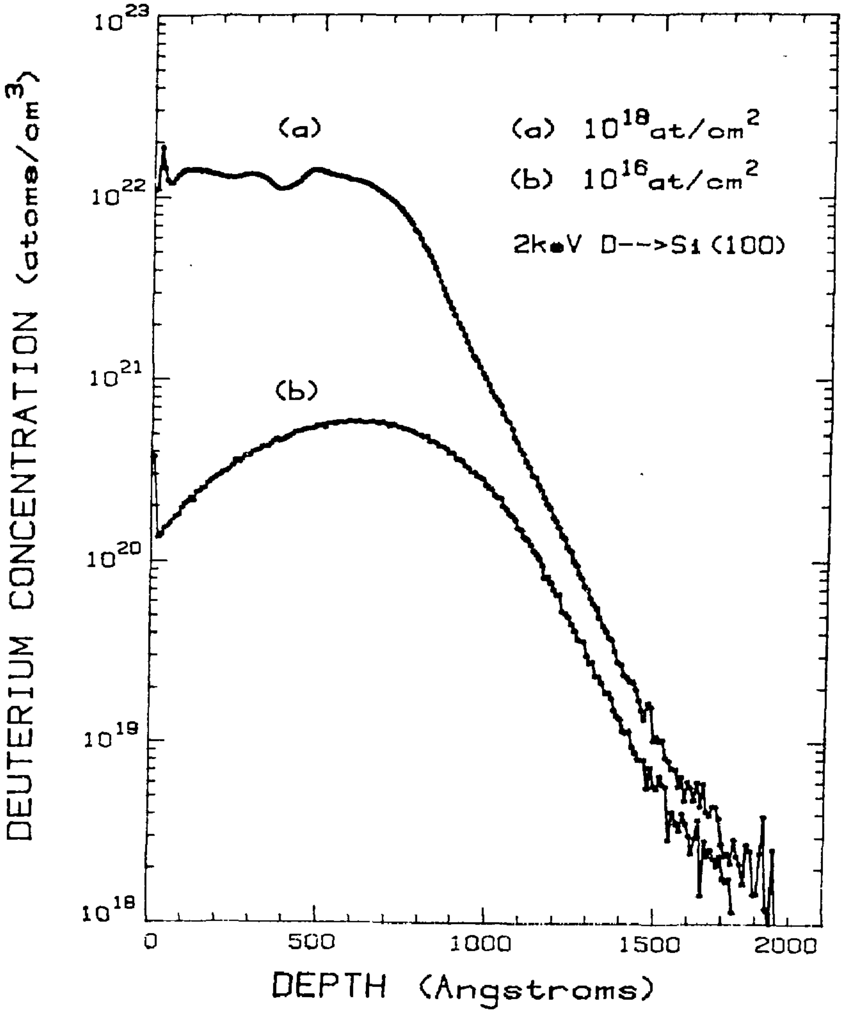

DEUTERIUM CONCENTRATION (atoms/cm³)
DEPTH (Angstroms)
(a)
(b)
(a) 10¹⁸at/cm²
(b) 10¹⁶at/cm²
2keV D-->Si(100)
10²³
10²²
10²¹
10²⁰
10¹⁹
10¹⁸
0
500
1000
1500
2000

Figure 5