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Low Temperature Plasma for the Preparation of Crater Walls for Compositional Depth Profiling of Thin Inorganic Multilayers

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Abstract

An indirect, compositional depth profiling of an inorganic multilayer system using a helium low temperature plasma (LTP) containing 0.2% (v/v) SF₆ was evaluated. A model multilayer system consisting of four 10 nm layers of silicon separated by four 50 nm layers of tungsten was plasma-etched for (10, 20, and 30) s at substrate temperatures of (50, 75, and 100) °C to obtain crater walls with exposed silicon layers that were then visualized using time-of-flight secondary ion mass spectrometry (ToF-SIMS) to determine plasma-etching conditions that produced optimum depth resolutions. At a substrate temperature of 100 °C and an etch time of 10 s, the FWHM of the 2nd, 3rd, and 4th Si layers were (6.4, 10.9, and 12.5) nm, respectively, while the 1/e decay lengths were (2.5, 3.7, and 3.9) nm, matching those obtained from a SIMS depth profile. Though artifacts remain that contribute to degraded depth resolutions, a few experimental parameters have been identified that could be used to reduce their contributions. Further studies are needed, but as long as the artifacts can be controlled, plasma etching was found to be an effective method for preparing samples for compositional depth profiling of both organic and inorganic films, which could pave the way for an indirect depth profile analysis of inorganic-organic hybrid structures that have recently evolved into innovative next-generation materials.

Keywords

dynamic sims; ToF-SIMS; bevel crater; depth profiling; low temperature plasma

Introduction

Low temperature plasma (LTP) is a dielectric barrier discharge (DBD) technique that generates a non-thermal plasma in atmosphere,¹ and has been used for ozone generation,^{2–3} polymer surface treatments,^{4–7} biological decontamination,^{8–9} and recently as a way to prepare crater walls for the indirect depth profiling of organic multilayers using time-of-flight secondary ion mass spectrometry (ToF-SIMS).¹⁰ The use of LTP for indirect depth profiling showed many advantages over a traditional ToF-SIMS molecular depth profile, where buried structures were easily resolved with high lateral magnification without the formation of ion bombardment artifacts such as chemical damage accumulation,^{11–13} polymer crosslinking,¹⁴ and topography generation^{15–16} that often results in degraded depth

resolutions and broadening of interface widths.^{11–12} This ambient etching method generates a large crater size which allows access to numerous imaging techniques for depth composition analysis of multilayer systems, such as for the forensic investigation of paint layers using Raman imaging or kelvin probe force microscopy of organic solar cell heterojunctions,¹⁷ making it a readily available sample preparation tool that offers an extra dimension of data at a significantly reduced cost and time.

The properties of the plasma can be altered relatively easily by changing the composition of the carrier gas; while pure argon or helium gas plasmas lead primarily to etching of organic molecules through chain-scission and formation of volatile molecules,^{7, 18} the addition of highly fluorinated molecules such as CF₄ or SF₆ into the carrier gas can lead to etching of metals through the formation of volatile fluorides.^{19–20} The ability to etch metals is particularly intriguing as it provides a way to prepare crater walls for the subsequent analysis with an imaging tool for compositional depth profiling of inorganic multilayer systems. The successful application of this technique has the potential to pave the way for indirect depth profile analysis of inorganic-organic hybrid structures that have recently evolved into innovative next-generation materials.^{21–22} As the organic portion of these materials can be sensitive to ion bombardment artifacts mentioned above, and as the inorganic portion can be altered due to topography generation and collisional mixing,^{23–25} the use of a more traditional method of inorganic depth profiling using energetic ions such as SIMS, focused ion beam (FIB) cross-section imaging, FIB-SIMS, or even x-ray photoelectron spectroscopy (XPS) depth profiling may not be suitable for their analysis.

As a first approach, LTP is used in a metal multilayer system to demonstrate its potential for preparing crater wall surfaces for compositional inorganic depth profiling. A He LTP mixed with 0.2% (v/v) SF₆ gas was used to prepare craters in a model inorganic multilayer system consisting of four 10 nm layers of silicon separated by four 50 nm layers of tungsten metal, at substrate temperatures of (50, 75, and 100) °C. The resulting crater walls were visualized using ToF-SIMS for compositional depth profiling, and the 1/e decay lengths of the silicon layers were measured, which was demonstrated to be an effective parameter for evaluating the “quality” of a depth profile.¹⁰ The results were compared to those obtained using a sputter depth profile technique using a dynamic SIMS instrument with an O₂⁺ source to validate the structure of the multilayer system.

Experiment

Sample Preparation

10 cm Si(100) wafers purchased from Virginia Semiconductors* (Fredericksburg, VA) were diced into 15 mm × 15 mm square pieces using the Disco DAD341 dicing saw equipped with a 15 μm diamond impregnated metal blade (Tokyo, Japan). The individual pieces were soaked overnight in an 18 MΩ/cm deionized water obtained from a Milli-Q ultrapure water system (EMD Millipore, Billerica MA) to remove salts, and sonicated sequentially for 10

*Certain commercial equipment, instruments, or materials are identified in this paper to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

min each in methylene chloride, acetone, and methanol (Sigma-Aldrich, Co., St. Louis, MO) to remove organic contaminants. Roughly 20 nm of Au was deposited onto the substrate with a 10 nm Cr adhesion layer using a sputter deposition system (Denton Discovery 550, Moorestown, NJ) with deposition rates of approximately 0.08 nm/s and 0.17 nm/s, respectively. On top of the Au surface, alternating layers of 50 ± 1 nm W and 10 ± 1 nm Si films (four layers each) were deposited at rates of 0.12 nm/s and 0.15 nm/s, respectively, with the total thickness of approximately 246 ± 4 nm (Figure 1a). The deposition rate of the layers was verified using a Dektak XT stylus profilometer (Bruker Corporation, Tucson, AZ), with a $0.7 \mu\text{m}$ radius stylus tip with an applied stylus weight of 0.9 mg, measured for each metal at three different deposition times outside of the sputter deposition chamber.

For preparation of plasma-etched craters, an LTP probe that was constructed in-house from a glass capillary tube (2 mm ID) was used. A copper electrode made of thin copper foil was wrapped around the tube, and the ground electrode was connected to the sample to deliver an alternating high voltage of 2.0 kV (rms) at a frequency of approximately 20 kHz.¹⁸ Ultrapure helium gas (500 mL/min) containing a small admixture of SF₆ (1 mL/min) was supplied to sustain the discharge. The tip of the glass capillary was positioned 4 mm above and directed normal to the sample surface. The samples were separately exposed to the plasma discharge for (10, 20, and 30) s while they were maintained at temperatures of (50, 75, and 100) °C using the DataPlate digital hot plate (Cole-Palmer Instrument Company, Vernon Hills, IL) with programmable temperatures. Verification of plate temperature with the Fluke 52 II Digital Thermometer (Everett, WA) yielded an error of up to ± 6 °C at the higher temperatures. All plasma etching experiments were performed inside a fume hood for safety.

Sample Analysis

Imaging of the craters was performed using a ToF-SIMS IV instrument (IONTOF, Münster, Germany) immediately after the craters were formed. The instrument was equipped with a 15 keV Bi₃⁺ liquid metal ion source at an incidence angle of 45°, operated in high mass resolution mode over an area of $500 \mu\text{m} \times 500 \mu\text{m}$ with an imaging pixel density of 256 pixels $\times$ 256 pixels. The ion dose density was 2.0×10^{10} ions/cm². Dynamic SIMS depth profiles were obtained using a Cameca 6F instrument (Gennevilliers, France) using an oxygen (O₂⁺) primary ion source, with an incident energy of 3.0 keV or 5.5 keV (7.5 kV or 10 kV accelerating voltage and a sample voltage of 4.5 kV, with incidence angles of 50° and 42°, respectively). Depth profiling was performed within a $150 \mu\text{m} \times 150 \mu\text{m}$ sputter area, while monitoring the ²⁹Si and ¹⁸⁴W positive secondary ions with a dwell time of 1 s each.

Profilometer traces of the craters were obtained using the above-mentioned profilometer. A 2 mm line scan was used to ensure that a large enough region outside the crater was measured to provide sufficient area for leveling. Roughness of the crater surface was measured from $15 \mu\text{m} \times 15 \mu\text{m}$ scans using an Asylum Cypher S Atomic Force Microscope (Santa Barbara, CA) operated in intermittent contact mode in air, equipped with the Bruker OTESPA silicon cantilever (Camarillo, CA) with a 9 nm tip radius, 315 kHz resonant frequency, and a 42 N/m force constant. For all analyses, values are averages of at least four measurements and the error represents the standard deviation.

Results and Discussion

With the angle of the plasma relative to the surface held at 90°, the isotropic etching of the plasma results in the formation of a cone-like crater with a roughly linearly-sloped crater wall that can then be imaged to resolve the buried layers in two dimensional space, similar to bevel crater depth profiling.^{26–31} Rather than using an incidence angle of 40° from the surface for optimum resolution,¹⁰ the use of a 90° angle for plasma etching resulted in a more consistently linear crater wall. As an example, Figure 1 shows the ToF-SIMS secondary ion image of the crater wall resulting from a 10 s plasma etch of the sample that was maintained at a temperature of 75 °C. The Si⁺ secondary ion image shows rings indicative of the thin Si layers buried in the matrix. The profilometer trace of the crater showed a roughly 250 µm diameter crater bottom where the substrate was exposed, with a roughly 450 nm tall crater wall that spanned a horizontal distance of about 195 µm, making an angle of 0.0023 rad with respect to the substrate. This equated to roughly a 430 magnification of the buried layers, which in theory meant that the 10 nm Si layers would appear to be approximately 4 µm wide lines, well within the lateral resolution of the instrument. From the Si⁺ ion image, the approximate width of the Si⁺ rings determined from the line scans varied from 9.8 µm to 11.7 µm (FWHM), which were within the anticipated values. The widening of the widths is thought to be due to the deposition of the less volatile plasma condensate around the Si bands (most likely some complex of Si and F), as indicated by the presence of the SiF⁺ ion co-localized with the Si⁺ ion (Figure 2). The widths of the SiF⁺ ion varied from 9.7 µm to 13.7 µm (Table 1), perfectly overlapping those of the Si⁺ ion.

Higher substrate temperatures were associated with a reduced re-deposition of the etched silicon fluoride molecules around Si, leading to enhanced depth resolutions. A similar phenomenon was observed in a different multilayer system (Mo/W) where fluoride complexes of Mo were found everywhere along the crater wall (Figure S-1, Supporting Information). Interestingly, their concentration on the surface could be reduced by heating the substrate during plasma etching, which seemed to suggest that these fluoride complexes were condensing onto the surface.³²

Similar to laser ablation, re-deposition of the plasma-etched material^{33–34} was also seen to a significant extent around the outside perimeter of the craters. As can be seen in the profilometer trace, there is a significant buildup of material that extends beyond the surface, forming a crater rim. This resulted in the 1st Si layer (topmost layer) being covered by the re-deposited material and the creation of an apparent buried Si layer; hence the reason for the observation of four to five Si rings in the ion images (only three are buried in the matrix). Due to this deposition around the rim, depth resolutions of the 1st Si layer were not reported. Also for SIMS, the 1st Si layer occurred at a transient region of the depth profile where the ionization yields have not yet stabilized, therefore it was deemed that the accurate comparison of depth resolutions for this layer could not be performed.

Figure 2 shows the ToF-SIMS ion images of the craters in the Si/W multilayers held at temperatures of 50 °C, 75 °C, and 100 °C. Higher temperatures were associated with a linear increase in plasma etching rate (Figure S-2, Supporting Information), and because the Au substrate does not etch, the crater was seen to grow wider and steeper, leading to an apparent

reduction in the width of the Si⁺ rings as a function of temperature. To obtain the depth resolution of these layers, the Si⁺ intensity vs position plots obtained from their lines cans were converted into a depth profile using a nonlinear transformation method described in a previous study.¹⁰ Briefly, one obtains a power function of the form described in Equation 1 by plotting the position vs depth, using knowledge of the actual depths of the four layers within the matrix;

$$y=ax^b \quad (1)$$

where y is the Si layer line scan position in μm , x is its actual depth in nm, and a and b are constant real numbers determined using a mathematical fit, respectively. Here, it is assumed that the interfaces occurred at 50% maximum intensity of the peaks, on the leading wall side. A nonlinear transformation was then used to convert the position into depth, where taking the logarithm of Equation 1 resulted in the regression equation shown in Equation 2. This was then used to re-construct the depth profiles shown in Figure 3. On the re-constructed plot, the onset of the Si interfaces corresponded linearly with the actual interface positions (Figure 3a).

$$x'=10^{\log(x)}=10^{\frac{1}{b}\log(y)-\frac{1}{b}\log(a)} \quad (2)$$

if the depths of the interfaces are not known, a profilometer trace of the crater wall can be used, whose slope also shows the same power function described in Equation 1. The curve is calibrated by using two known depths; one at the substrate interface, and the second at the top of the multilayer.

Using the constructed depth profiles, the depth resolutions of the buried Si layers were determined by calculating the depth over which the signal of the delta layer fell to 1/e of the maximum value, referred to as the decay length and calculated using the following equation,³⁵

$$\lambda=\frac{(x_1-x_2)}{\ln(I_{x_2}/I_{x_1})} \quad (3)$$

where x_1 and x_2 are depths in nm, and I_{x_1} and I_{x_2} are the secondary ion intensities at those depths. In addition, the FWHM of the intensity peaks were determined as a way to show any peak broadening due to artifacts from plasma etching, although the use of FWHM as a measure of depth resolution is typically reserved for thin delta layers in the order of a few nanometers.³⁶

Table 2 shows the depth resolutions of the Si layers exposed to the plasma at three different temperatures. For both the decay length and the FWHM, there were two general trends regarding depth resolutions that were observed in the data: one, they were seen to improve with the use of higher substrate temperatures, where it was thought to have assisted in

reducing the deposition of fluorinated silicon molecules around the Si bands; and two, they were seen to degrade linearly with depth, suggesting a dose-dependent effect similar to a SIMS depth profile typically caused by topography generation at the crater bottom. As can be seen in Figure 3b, the roughness along the plasma-etched crater wall did increase exponentially with depth (the virgin film had a few locally rough spots, which contributed to its relatively higher average roughness). At this time, it is not clear how topography is generated by the plasma, or how it could affect the broadening of the Si bands. Although the reduction in both roughness and FWHM with temperature seem to suggest that topography is related to the re-deposition of the etched material.

At a substrate temperature of 50 °C, the FWHM values were approximately (12.9, 16.9, and 22.4) nm for the 2nd, 3rd, and 4th Si layers, respectively, much larger than the actual layer thickness of approximately 10 nm. For 75 °C, the values were slightly smaller and averaged (8.3, 11.9, and 21.5) nm, respectively, showing a puzzling jump in value between the 3rd and 4th layers. The depth profile at 100 °C were more in alignment with the actual thickness of the Si layers, averaging (6.4, 10.9, and 12.5) nm, respectively. At this temperature, the 1/e decay lengths were also the smallest in the group, at (2.5, 3.7, and 3.9) nm for the 2nd, 3rd, and 4th Si layers, respectively. For lower temperatures of 50 °C and 75 °C, the average values were higher, and there was a similar jump in value between the 3rd and 4th layers. For the conditions studied, heating the substrate to 100 °C seemed to produce depth profiles that were much better behaved, showing much higher depth resolutions with minimal plasma-induced artifacts.

To verify the structure of the manufactured multilayer film, the samples were sputter depth profiled using O₂⁺ primary ion bombardment at incident energies of 3.0 keV and 5.5 keV. The depth profiles showed anticipated trends as a result of influences from ion bombardment artifacts; both the decay length and FWHM degraded with depth as well as with the use of higher primary ion energy, which were most likely caused by the increase in topography (Figure 4b). As can be seen in Table 3, the decay lengths obtained using the 5.5 keV beam increased from (3.3, 4.1, to 4.7) nm for the 2nd, 3rd, and 4th Si layers, respectively, and those obtained using the 3.0 keV beam increased from (2.1, 2.7, to 3.7) nm, respectively. The values obtained using the lower energy beam were very similar to those obtained using the LTP at a substrate temperature of 100 °C, showing that the metal multilayer structure revealed by the two techniques was indeed similar. Although SIMS depth profiling using lower incidence energies would have achieved better depth resolutions, this was outside the scope of the experiment as the objective was not to find the best technique for depth profiling, but to find qualitative differences (*i.e.*, artifacts) between the two techniques at experimental conditions that produced similar depth resolutions. Ideally, this would allow further development of the LTP as an indirect depth profiling technique.

One striking difference produced by SIMS depth profiling was the asymmetrical widening of the peaks that resulted from the use of a higher energy beam. This asymmetry was illustrated in part by comparing the integrated counts for the peaks in Figures 4c and 4d: for the depth profile generated using an incidence energy of 3.0 keV, the areas under the curve roughly remained constant to within 10% variation around (4.3, 6.7, 6.5, and 6.2) × 10⁶ counts for the 1st, 2nd, 3rd, and 4th Si layers, respectively, while for an incidence energy of

5.5 keV, the integrated counts increased from (4.3, 6.3, 7.3, to 7.6) $\times 10^6$ counts, respectively. Although the significantly lower values for the 1st Si layer are most likely a byproduct of being in the transient region of the depth profile, the broadening of the subsequent layers is thought to be due to a variation in ion yield brought about by changes in the sputter rate from a combination of collisional mixing and roughening. The exact mechanism of ion yield variation is not clearly understood at this time, but illustrates an artifact that was not observed in the crater walls etched by LTP. Overall, the use of lower incident energies seemed to reduce ion bombardment artifacts, and produced depth profiles with much better depth resolutions. Using the 3.0 keV source, the FWHM values were (8.5, 9.9, and 10.9) nm for the 2nd, 3rd, and 4th Si layers, respectively, very close to the actual Si layer thickness and slightly better than what was observed for the plasma-etched crater wall at 100 °C.

For the plasma-etched crater, heating the substrate to 100 °C seemed to produce FWHM and 1/e decay length values that were as good as those obtained using SIMS depth profiling, at least for the Si/W multilayer system. In addition, the symmetrical Si^+ peaks in the lines cans showed that the technique did not experience a mixing artifact that was observed in SIMS. However, the technique did have artifacts of its own, with the most obvious being the re-deposition of the etched fluoride complexes that led to the broadening of the peaks, negatively affecting the depth resolution. Increasing the substrate temperature was seen to limit this phenomenon, but also led to the formation of steeper crater walls that resulted in an apparent enhancement in depth resolution, caused by the enhanced contrast from the reduced number of data points contained within the thinning Si bands. The second observed artifact was the loss of signal with increased plasma exposure, observed toward the center of the crater, which precludes the technique for quantitative analysis. This suppression effect was seen for all ions, some of which are shown in Figure 2 where intensities of the Si^+ , SiF^+ , and WO^+ ions were generally seen to decrease toward the crater bottom. One way to minimize this artifact was to use cluster sputter cleaning using an argon gas cluster source (Figure S-3, Supporting Information), suggesting a presence of a contaminant overlayer that was suppressing the signal of the metals. These contaminants are most likely nitrogen-containing organic molecules, observed as NH_4^+ , CH_4N^+ , NO_3^- , and $\text{C}_2\text{H}_8\text{N}^+$ ions using ToF-SIMS, which were shown to deposit on the surface as a byproduct of plasma interaction with air.^{10, 18}

Another potential plasma-induced artifact worth noting is the formation of non-linear crater walls, such as waves and ripples, that cannot be mathematically modeled. Since the conversion of a ToF-SIMS intensity line scan into a depth profile requires an accurate crater profile measurement and a best-fit mathematical line, not being able to fit a model will adversely affect the quality of the depth resolution achievable. The assumption of a linear crater wall in place of a curved wall, for example, can greatly distort peak widths, which can lead to incorrect calculation of depth resolutions. This artifact is important to note since various factors can affect the linearity of the crater wall, such as non-uniform etching behavior caused by variations in plasma density of the filamentary jet during etching, or materials with different etching rates. Although no observable differences in etching rates were seen for the Si and W system studied, combinations of other metals may be problematic. As for inorganic-organic hybrid systems, a large difference in etching rates do

exist,^{10, 18} however its effect on the linearity of the crater wall, and consequently the depth resolution, is unknown given the miniscule size scale of the film (tens of nanometers) relative to the plasma etching rate (hundreds of nanometers per second). A better understanding of these effects is clearly needed, and will be the subject of future investigations.

Conclusions

An inorganic thin film multilayer system containing layers of Si and W were used to evaluate the effectiveness of using a low temperature plasma for crater wall depth profiling, using FWHM and 1/e decay lengths as a parameter to evaluate their effectiveness. Crater walls prepared using LTP suffered from artifacts such as the deposition of fluoride complexes and the generation of crater wall topography, but could be controlled by heating the substrate which consequently improved the depth resolution. For the conditions studied, a substrate temperature of 100 °C was able to produce depth profiles with FWHM and decay lengths that were in close agreement to those obtained using SIMS with an O₂⁺ source at 3.0 keV. Future work will investigate the applicability of the plasma for etching craters in the inorganic-organic hybrid structures as well as to obtain a clear understanding of the effect of differential etching rates on depth resolution.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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Figure 1.

(a) Schematic drawing of the multilayer system along with their measured thicknesses, and (b) secondary ion image of the plasma-etched crater wall showing the lateral distribution of the Si⁺ ion. The numbering of the individual Si layers is also shown; the 1st layer of Si is located at the top of the multilayer system, but appears as a ring due to the re-deposition of the etched material along the crater rim. (c) Profilometer trace showing the crater cross-section and the rim formed by the re-deposition of the etched material. Plasma was ignited with a discharge voltage of 2.0 kV with an exposure time of 10 s, with the sample maintained at 75 °C.

Figure 2.

ToF-SIMS secondary ion images of the crater wall showing the distributions of Au^+ , Si^+ , SiF^+ , and WO^+ species. The craters were formed using a 10 s plasma etch, with the samples maintained at temperatures of 50 °C, 75 °C, and 100 °C. The red box seen in the Si^+ ion image (75 °C) is the area used for line scans to extract the intensity vs position data. The box is approximately 65 μm wide (33 pixels). Images are 500 $\mu\text{m} \times 500 \mu\text{m}$.

Figure 3.

Measurements obtained from plasma-etched craters: (a) a plot comparing the empirically-determined actual positions of the Si layers with those converted from line scan positions, showing the effectiveness of using nonlinear transformation as a way to recalibrate the intensity vs position plot into depth profiles. (b) RMS roughness measured at the Si layers along the crater wall. Values are averages of at least three measurements; error bars are smaller than the data point markers. (c) and (d) Crater wall depth profiles obtained at substrate temperatures of 50 °C and 100 °C, respectively, showing the evolution of the Si⁺ peaks. Dots indicate experimental values, and the solid line shows the Gaussian fit. The depth profile for 75 °C was very similar to (c) and was not included here to save space.

Figure 4.

Measurements obtained from SIMS depth profiles: (a) a plot showing the sputter rates of the O_2^+ primary ion source at incident energies of 3.0 keV and 5.5 keV, and (b) RMS roughness of the crater bottom measured at each of the Si layers. (c) and (d) show the depth profiles monitoring the $^{29}Si^+$ ion, obtained using the primary ion at incident energies of 3.0 keV and 5.0 keV, respectively. Dots indicate experimental values, and the solid line shows the Gaussian fit.

Table 1

Lateral width of the Si^+ and SiF^+ rings (FWHM) as determined from a line scan of the ion images shown in Figure 1. The 1st Si layer refers to the topmost Si layer in the multilayer structure, and the 4th Si layer refers to the last layer at the bottom of the structure.

	Si^+ (μm)	SiF^+ (μm)
1st Si layer	15.7 ± 1.1	15.6 ± 1.2
2nd Si layer	11.6 ± 0.9	11.7 ± 1.0
3rd Si layer	9.8 ± 1.0	13.7 ± 2.1
4th Si layer	11.7 ± 1.1	9.7 ± 3.0

1/e trailing edge decay lengths and FWHM depth resolution measurements determined from the compositional depth profiles of the plasma-etched crater wall. Etch time was 10 s.

Table 2

	1/e decay length (nm)			FWHM (nm)		
	50 °C	75 °C	100 °C	50 °C	75 °C	100 °C
1st Si layer	1.2 ± 0.3	1.0 ± 0.2	0.8 ± 0.1	4.9 ± 0.1	2.8 ± 0.1	2.7 ± 0.1
2nd Si layer	4.2 ± 0.3	3.1 ± 0.3	2.5 ± 0.8	12.9 ± 0.7	8.3 ± 0.3	6.4 ± 0.2
3rd Si layer	5.3 ± 0.3	5.5 ± 0.4	3.7 ± 0.9	16.9 ± 2.1	11.9 ± 1.1	10.9 ± 0.7
4th Si layer	12.7 ± 1.8	10.7 ± 1.3	3.9 ± 0.1	22.4 ± 3.7	21.5 ± 3.2	12.5 ± 1.7

Table 3

1/e trailing edge decay lengths and FWHM depth resolution measurements, determined sputter depth profiles using the 6f SIMS instrument, using an O_2^+ primary ion source at incidence energies of 3.0 keV and 5.5 keV.

	1/e decay length (nm)		FWHM (nm)	
	3.0 keV	5.5 keV	3.0 keV	5.5 keV
1st Si layer	0.6 ± 0.1	0.8 ± 0.1	2.9 ± 0.7	4.3 ± 0.3
2nd Si layer	2.1 ± 0.1	3.3 ± 0.4	8.5 ± 0.6	9.6 ± 1.9
3rd Si layer	2.7 ± 0.1	4.1 ± 0.3	9.9 ± 1.1	16.1 ± 2.1
4th Si layer	3.7 ± 0.2	4.7 ± 0.2	10.9 ± 1.1	21.2 ± 4.8