

Ultrathin SiO₂ on Si. VII. Angular accuracy in XPS and an accurate attenuation length

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Following an earlier study of the uncertainties for defining thicknesses by angle-resolved XPS, one of the major instrumental uncertainties has been evaluated that limits both precision and accuracy. For analysis of the thicknesses of SiO₂ on Si, certain angles of emission have been recommended, but an error of 1° in these angles leads directly to an error in the thickness of ~1% from this contribution alone. This is significant since the total uncertainty required in the (International technology roadmap for semiconductors) ITRS is only 1%. In many instruments, to reduce sample-stage vibration and for other reasons, the angle and other setting adjustments are engineered with backlash. This, combined with the manufacturer's tolerances, can lead to angular errors above 1°. We report here a device using a laser and reflectors, fixed to the sample mount, that allow the angle of emission to be set to a precision better than 0.1° and, furthermore, a method to set the zero angle of emission to 0.1°. Using this geometrical device as well as by measurements of intensities in XPS, it is deduced that the data in our earlier report for the CCQM (Consultative Committee for Amount of Substance) intercomparison were for angles $1.89^\circ \pm 0.15^\circ$ too high. Consequently, by a re-analysis of all of that data, we find that our recommended attenuation length data for the Si 2p photoelectrons in thermal SiO₂ using Mg or Al K α X-rays should be increased to 2.996 nm and 3.485 nm, respectively, an increase of 1.2% on the originally calculated values. These values now have standard uncertainties of 0.54% instead of the 20% of the TPP-2M calculations. This leads to an improved accuracy in the measurement of ultrathin thermal oxides on silicon by XPS, of better than 1% for thicknesses greater than 1.5 nm but less than 8 nm. © Crown copyright 2005. Reproduced with the permission of Her Majesty's Stationery Office. Published by John Wiley & Sons, Ltd.

KEYWORDS: angle of emission; calibration; silicon dioxide; thickness measurements; traceability; XPS

INTRODUCTION

For the measurement of the thickness, d , of an ultrathin oxide on a substrate of the unoxidised element, it is difficult to find a better technique than x-ray photoelectron spectroscopy (XPS). For these films, especially on low atomic number substrates, the simple equation:^{1,2}

$$d = L \cos\theta \ln(1 + R/R_o) \quad (1)$$

can be very accurate,² where L is the attenuation length (AL) of the electrons in the oxide overlayer, R is the ratio of the oxide- and substrate-measured intensities, R_o is the similar ratio for the bulk oxide and bulk substrate materials and θ is the angle of emission of the electrons from the surface normal. In this formulation, the oxide and substrate intensities are measured using the substrate element peak in both the oxide and elemental states by peak synthesis. By this approach, we only obtain the intensity from oxygen related

to the oxide of the substrate element³ and we also avoid the complexities and uncertainties associated with measuring intensities at significantly different energies.⁴ Note that, in practice, the peak synthesis also gives three further peaks for the intermediate oxides, and so the calculations are more complex^{1–3} involving four equations like Eqn. (1) but, for the present discussion, Eqn. (1) is adequate.

Equation (1) is derived for amorphous materials and can only be applied to crystalline overlayers or substrates, with care.¹ By a suitable choice of azimuth and angles of emission together with the angle-averaging of a spectrometer with an input lens semi-angle in the range 3 to 10°, the effects of forward focusing associated with a crystalline lattice may be significantly reduced.¹

The attenuation length, L , may be obtained from the inelastic mean free path^{5–7} together with some reduction associated with the elastic scattering.^{8,9} The reduction factor may be calculated accurately, but the inelastic mean free paths have not been measured accurately and current calculations are only estimated to be accurate to a standard uncertainty of the order of 20%. This uncertainty dominates the uncertainty in d in Eqn. (1) and leads to XPS being considered as a good method only for relative measurements.

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However, recent work³ has shown that, for a given system, L can be calibrated, in principle with accuracies better than 1%. In this case, Eqn. (1) could be valid with accuracies better than 1% and XPS then becomes a simple, traceable, accurate measurement method for, in this case, thermal SiO₂ on Si for thicknesses less than 8 nm. In this thickness range, the uncertainty in R , for reasonable signal levels,² is not a dominant factor in the uncertainty in d . For $d > 8$ nm, the Si intensity rapidly weakens and the measurement of this signal dominates the uncertainty contributions.² We therefore limit the method to $d \leq 8$ nm. It is expected that the lower limit is zero thickness but the accuracy has only been tested down to 1.4 nm although we have found that material of 0.7 nm thickness is stable for a period in excess of over 3 years. Of course, as d reduces below 1.5 nm, an absolute level of uncertainty exists from the statistical contribution of the background.² This leads to the absolute uncertainty levelling out below 1 nm and the relative uncertainty then rising above 1%. In the calibration of L , many analytical techniques were used.³ There is always some anticorrelation between the value of L obtained and the value of R_0 assumed. This anticorrelation is built into the calibration for L such that, for the small possible range in R_0 , L is established to better than 1% for a chosen fixed value of R_0 .

Thus, in the end, we may use XPS to measure d to within 1% for the thickness range 1.5 to 8 nm, if we can set θ accurately. This 1% is an important goal required in the ITRS (International technology roadmap for semiconductors).¹⁰ In our earlier study,² we reported that θ could be set to a standard uncertainty of 0.56°. This uncertainty is for repeatedly setting one sample to the same angle of emission using the sample-stage tilt mechanism rotary drive markings. Over a longer term, 0.75° was thought more reasonable. This led to a contribution to the uncertainty in d of 0.9% for (100) surfaces, reducing to 0.6% for (111) surfaces with their lower angle of emission. However, it was clear from the earlier studies^{2,3} that a method was needed to confirm the accuracy of the angle scale in addition to improving the above precision for setting that value.

Our instrument is based on a VG Scientific ESCALAB Mk II and was not designed for setting accurate angles of emission; the system angles being only intended to be accurate to 1°. Combining the positioning of the vacuum port for the lens to the spectrometer, of that lens and of the rotary drive for setting the sample holder for the angle of emission, there are at least three occasions for errors to accumulate. The design of the sample stage, furthermore, decouples the x and y movement drives during analyses in order to reduce stage vibration and drift. Thus, settings are made against a designed backlash. This could add further errors. Finally, even if one knows the geometry of the input lens accurately, there is the possibility of stray magnetic fields causing a further shift to the average angle of emission analysed.

In this work, therefore, we describe a method for setting the angle of emission, θ , accurately. As required from our earlier study, 34° will be used for (100) surfaces and 25.5° for (111) surfaces. These angles are measured from the surface normal. The new method, described here, shows

that the angles of emission in our earlier study,³ in which the attenuation length for thermal SiO₂ was evaluated, are between 1° and 2° too high. We, therefore, recalculate all of the previous correlations of data in the interlaboratory study³ and re-present the essential table from that study corrected for this small offset. The new value of the attenuation length for thermal SiO₂ is then calculated.

METHOD FOR SETTING THE ANGLES

We base our method on a laser system with mirrors fixed to the sample mount. A very inexpensive diode laser with an attached lens provides a beam of light that is collimated with a diameter of less than 1 mm. Mounted outside the main viewport, this illuminates a spot on the sample and, for a Si wafer appropriately tilted, will produce a reflected spot of light onto a graticule located on the outer surface of the main viewport window. For a rotation of δ , in the sample stage tilt angle, relative to the sample orientation for the laser beam to be reflected along the direction of the viewport axis, the shift of the spot on the graticule, x , is given by

$$x = P \tan(2\delta) \quad (2)$$

Here P , the distance of the graticule from the sample rotation axis, is 237.2 mm. Thus, x is approximately 8.28 mm per degree change in the angle of emission. To improve the precision in d , we use an optical microscope with a digital image recording system to observe the sample stage tilt scale setting. This clearer recording of the drive marking, on its own, led to significant improvements. The difference between $\tan(2\delta)$ and 2δ leads to an error of less than 0.02° in this range and can be ignored. The Si wafers are excellent surfaces to provide the reflection since they may be cleaved into suitable sizes, are not insulating and reflect from just the upper surface.

With the above, we may mount a small block on the sample holder, as shown in Fig. 1, on which there are three small wafer reflectors, A, B and C. The first, A, is positioned to reflect the beam approximately to the graticule zero when the sample is set normal to the input lens for our spectrometer ($\theta = 0^\circ$), the second, B, is nominally at 25.5° to this ($\theta = 25.5^\circ$) and the third, C, nominally at 34° to A ($\theta = 34^\circ$). Note that the reflectors are all on the same radius, 5.8 mm, from the rotation axis of the sample holder. This value needs to be subtracted from P for measurements on these reflectors so that the shift of the laser spot reduces to 8.08 mm per degree change in the angle of emission. Of course, when machining the small block, coupled with the need to adhere the small wafer reflectors, the final angles are not exactly as above. Measurements of the angles were easily made on a workbench, tracing the lines of the laser light against a draftsman's protractor. This gave the required angles as 23.17° and 34.18° with an estimated uncertainty from the tracing as 0.1°. This effectively uses the device as it is used on the sample holder. Using a traceable co-ordinate measuring instrument, certified values of $23.07 \pm 0.07^\circ$ and $34.21 \pm 0.07^\circ$ were obtained (Cross N, NPL, private communication 2004.) for a 95% confidence level, showing how easy it is to

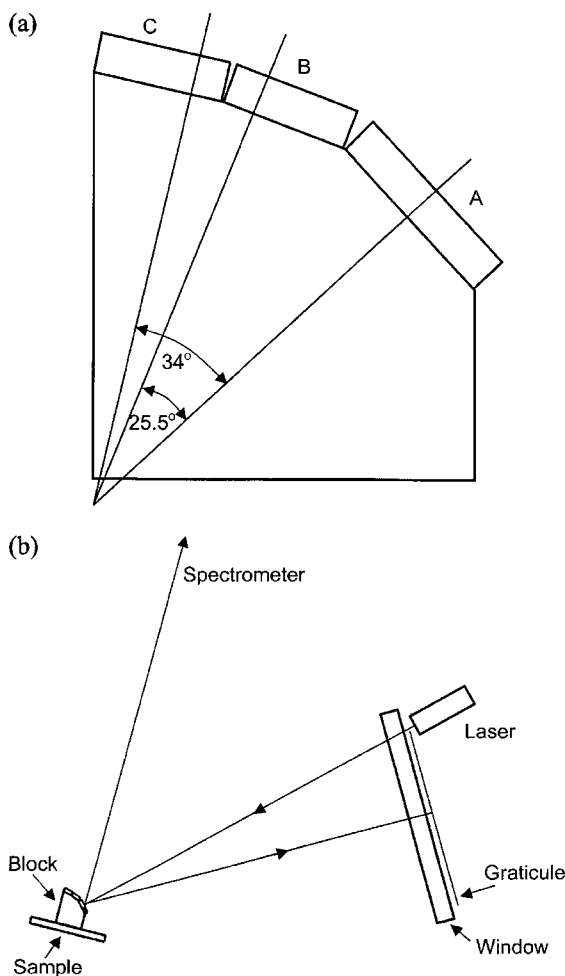


Figure 1. The small angle block: (a) details of the block geometry, face A is used for alignment of the sample normal, face B for the (111) orientation and face C for the (100) orientation, and (b) the laser alignment system with the block, window and graticule.

define the angles accurately. This second method measured the orientations of the surfaces themselves rather than the orientations of the bisectors of the specular beam angles.

Using the above data, if the beam position for the laser reflected from face A is at x_0 mm on the graticule for normal emission of the electrons, then for (100) surfaces the light should be reflected from face C and be at $0.21 \pm 0.07^\circ$ above x_0 or at $x_0 + (1.7 \pm 0.6)$ mm. On the other hand, for (111) surfaces, the light should be reflected from face B and be set at 2.43° below x_0 or at $x_0 - (19.7 \pm 0.6)$ mm. This is easy to set to 0.5 mm or to 0.06° ; a level far more precise than we really need.

Before we can set the above angles, we need to check the correct orientation for normal emission. To do this, we take a piece of (100) wafer with a very thin oxide so that the forward focusing¹ is maximised. We then check that the wafer is cut accurately to the (100) plane and measure the thickness by Eqn. (1) as a function of the orientation for angles about the normal emission. The emission angle corresponding to the minimum calculated thickness is the angle for $\theta = 0^\circ$ and the position of the laser beam on the graticule allows this to be set accurately and repeatedly thereafter.

RESULTS

First, we check that Eqn. (2) is valid and the extent to which it may be applied. An average of some four sets of data for a range of 4° either side of $\delta = 0^\circ$ gave a value of x/δ for Eqn. (2) of 8.23 mm per nominal degree change in angular setting from a sample surface and 8.08 mm per degree from the angle block faces A, B and C, confirming Eqn. (2) within experimental error. The deviation of the individual points about this function in each data set showed a standard deviation in x of 0.4 mm equivalent to 0.05° . Next, we replace the sample, which is fixed to the sample holder, many times in the sample stage and set the sample-stage tilt at defined settings. This shows a standard deviation of 2.14 mm on the graticule equivalent to 0.25° of sample stage tilt. This may arise from a non-kinematic seating for the sample holder on the sample stage and indicates a contribution to the scatter in our historical data that was set by the sample stage tilt angle. For the present work, the settings all use the graticule values and this avoids any failure of the sample holder to bed properly onto the sample stage.

Next, we evaluate a reference sample of (100) Si with 0.7 nm of oxide for the closeness of the [100] direction to the surface normal. Analyses using a Siemens Diffraktometer Kristolloflex D500 showed that the angle of the [100] direction was off normal by 0.053° in one azimuth and 0.04° in the orthogonal azimuth. For the purposes of the present work, this surface is thus a true (100) surface. This sample was then used for the next part where we analyse the samples by XPS using Mg K α X-rays as previously.¹ As before¹ we remove the x-ray satellites as well as the spin orbit $2p_{1/2}$ splitting to leave spectra with one peak for each chemical state. We set the zero of emission by analysing the apparent thickness, d , from Eqn. (1) with $L = 2.964$ nm and $R_0 = 0.9329$ for many angles around the surface normal, as shown in Fig. 2. We measure these angles using the laser spot on the graticule and also by the sample stage tilt scale setting. As we saw before,¹ we observe a symmetrical minimum about the [100] direction with a full width at half maximum of 9° . By fitting data at 1° intervals for two separate days of measurements, the minimum is found to be at a sample stage tilt scale setting of 12.36° and, in a repeat, at 12.45° . After rotating the sample by 180° azimuthally, this changed to 12.31° . These are 1.89 , 1.80 and 1.94° less than the nominal 14.25° setting used in our earlier work.³ The scatter is reduced from the $\pm 0.75^\circ$ when using the sample stage tilt scale setting by eye to the lower value of $\pm 0.1^\circ$ when using the graticule. The graticule values of the minima were $x = 0.94$ and 0.29 mm for one azimuth and 1.27 mm for the other azimuth with an average taken as $0.9 \text{ mm} \pm 0.3 \text{ mm}$ equivalent to $12.36 \pm 0.04^\circ$. The x values on the graticule are converted to the above angles using the observed correlation between x and the angle setting using the microscope. This indicates an offset of 1.89° . If there is a 0.1° uncertainty on each graticule reading and if the defined angles are accurate to 0.035° , the total standard uncertainty for an emission angle setting is 0.15° . The results shown in Fig. 2 show no significant change in thickness over a period of nearly 4 years.

We may deduce the angular offset also by using the geometry of the system as follows. With the sample set at

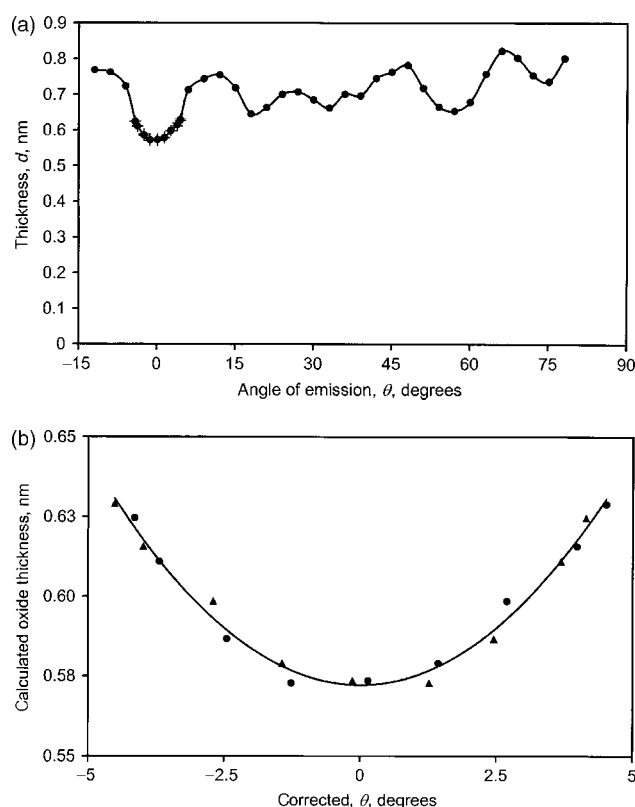


Figure 2. Values of d from Eqn. (1) around the [100] pole on a (100) surface to set the zero of emission angle, (a) (●) data from the earlier study¹ with the new data (+) superimposed at the [100] pole and (b) detail of the new data around the pole (●) with, superimposed (▲), the new data inverted on the abscissa for symmetry. The fitted curve is a function with terms of up to order two. The data have been shifted, as described, to correct the minimum to the zero emission angle.

the (111) orientation, the laser reflects off the sample surface and up the spectrometer lens axis at the same time as the observer, looking through the window can see, using the sample as a mirror, an image of the laser spot on the front of spectrometer lens structure where there is an iris acting as a variable aperture. This is, conveniently, at right angles to the lens axis and the line of sight. By adjusting the sample stage tilt, the laser spot can be placed at the upper and lower edges of the input lens iris as well as its centre. The average sample stage tilt scale setting for several readings of the two aperture edges were 32.74 and 39.43° giving the centre at 36.085°. Averaging this value and the measured centre value of 35.98° gives the geometrical axis at $36.03 \pm 0.06^\circ$ where the uncertainty is the precision. The surface normal is now along the bisector of the laser direction and the spectrometer axis. At a sample-stage tilt setting of 58.378°, the laser beam reflects back upon itself and so the spectrometer axis is at

$36.03 - (58.378 - 36.03) = 13.69 \pm 0.28^\circ$. This is only 0.56° less than the nominal 14.25° setting used in our earlier work.³ Of course, this value is less traceable than the 12.36° value given above since the sample-stage tilt mechanism setting markings are not traceable. The reason for conducting this analysis is to show that the geometrical and electron-optical axes may not be exactly the same. Here the difference is $1.33 \pm 0.28^\circ$. The spectrometer that we use has a 150° spherical sector analyser with angular selectors that define the input angle at a nominal $\pm 6^\circ$. When analysing the Si 2p photoelectrons of 1150 eV at a pass energy of 20 eV and with a lens magnification of three, only $\pm 2.4^\circ$ of the nominally $\pm 6^\circ$ input to the lens system is filled by detected electrons. The spherical sector entrance angle defining slits are effectively small parallel plates set tangentially to the sector radii, just inside the sectors, and these may not keep the accepted electrons truly on axis. If the electrons are 1° offset here, then the electrons emitted from the sample and detected are those 0.4° away from the geometrical axis. This effect and stray magnetic fields may account for the above 1.33° offset and illustrate the importance of the method shown in Fig. 2(a).

In summary, for this instrument, we have found the following angular offsets. Prior to the CCQM study, an offset of 0.75° was measured arising from the setting of the sample stage tilt mechanism zero. A further offset of 0.56° arises from the alignment of the spectrometer input lens and a final offset of 1.33° occurs between the electron optical and geometrical axes of the spectrometer inputs. All these offsets, unfortunately, are in the same direction! The [100] pole alignment method shows the combination of the above offsets to be 1.89° more than originally thought.³

More important than defining the angular error as above is to define the thicknesses using these correct angular settings. Table 1 shows the ratio of the new thicknesses, d , to those previously used in the CCQM pilot study,³ d^* . In the present study, four careful measurements were made for the reference set of samples and two for NPL's test set (sets 12 and 9, respectively). The standard deviation of the four repeat measurements for the 10 samples of set 12 is now characterised by $0.004d$ using the laser spot position on the graticule. Additionally, here we use a 25-point smooth to define the end points for the Shirley background removal.² For our experimental arrangement, this smooth is from 6.9 to 9.3 eV kinetic energy below the Si elemental peak and 1.2 to 3.6 eV kinetic energy above the Si peak, inclusively (a 21-point smooth from 6.9 to 8.9 eV kinetic energy below the Si elemental peak and 1.2 to 3.2 eV kinetic energy above the Si peak, inclusively, is almost equivalently as good²), but the user needs to check in all cases that the tails of the peaks are insignificant in the smoothing region. It may be that in other experimental arrangements there is a larger or smaller shift

Table 1. Change in reported thicknesses of the NPL reference samples (set 12)

Substrate orientation Nominal thickness, nm	(100)						(111)			
	2	3	4	4.5	5	8	2	4	6	7
d^*/d	1.019	1.009	1.007	1.047	1.022	1.021	0.987	1.003	1.012	1.015
Average d^*/d				1.021					1.004	

of the Si 2p peak for the oxide state in the thicker samples.¹¹ Seven repeats for three different sample thicknesses give standard deviations described by $0.003d$. This new procedure represents a factor of three improvement in both the precision and accuracy over the previous analyses.²

The average increase in thickness for the (100) samples of both sets 12 and 9 is 1.024 and for the (111) samples 1.010. We may deduce the angular offset approximately from such data as follows. The thickness, d , at an angle, δ , beyond the correct angle, θ (34° for (100) and 25.5° for (111) samples), is given by

$$d = L \cos(\theta + \delta) \ln \left(1 + \frac{R_\delta}{R_o} \right) \quad (3)$$

assuming there to be little change in the forward focussing over this small range. If we write the previous erroneous value of d as d^* , then

$$d^* = L \cos(\theta) \ln \left(1 + \frac{R_\delta}{R_o} \right) \quad (4)$$

Thus,

$$\frac{d^*}{d} = \frac{\cos(\theta)}{\cos(\theta + \delta)} \quad (5)$$

from which δ may be determined. For the six (100) samples of sets 9 and 12, reprocessed using the 25-point smooth, $\delta = 1.90^\circ \pm 0.91^\circ$ and, for the four (111) samples from each set, $\delta = 1.11^\circ \pm 1.31^\circ$ giving an average of $1.58^\circ \pm 1.02^\circ$, slightly less than, but consistent with, the value of $1.89^\circ \pm 0.04^\circ$ found for the electron-optical spectrometer axis.

We now correct all of the reference thicknesses for the NPL XPS reference data in Ref. (3) by dividing all the respective data by the values in Table 1. We then re-evaluate all of the plots of

$$d_{\text{NPL}} = m d_{\text{respondee}} + c \quad (6)$$

for each of the data sets given in Ref. (3). We would not expect much change in the offset c but would expect the m

values to increase by a factor of between 1.010, if respondees had only used (111) samples, and 1.021, if they had only used (100) samples. Since there were more (100) samples, one would expect a value near 1.021. Unfortunately, inspection of Table 1 shows that the values of d^*/d are somewhat higher for the higher thicknesses, leading to a slight further increase in m and a slight reduction in the c values. Table 2 is thus a revision of our earlier³ Table 13 allowing for these corrections.

Converting the figures provided previously,³ the unweighted averages give $m = 1.012 \pm 0.017$, whereas with weightings² $m = 1.011 \pm 0.005$. These are 1.025 times the values given earlier.³ If the weighted value of $m = 1.011 \pm 0.005$ is used, we may recalibrate NPL XPS data by increasing the thicknesses by 1.2%. This is done by scaling L_{SiO_2} by 1.011 to give 2.996 nm for Mg K α X-rays and 3.485 nm for Al K α X-rays, instead of the respective values 2.964 and 3.448 nm originally used.¹ The standard uncertainty here arises from the uncertainty in the weighted value of m of 0.51% and the uncertainty in the new data averaged over four data sets being, from the standard deviation of the mean, 0.19%. This gives a total uncertainty of 0.54% or ± 0.016 and ± 0.019 nm, respectively, for these two attenuation lengths. These, coupled in quadrature with the previous uncertainties for setting the angles and for the repeatability of the data, give a final standard uncertainty in the value of d of 0.64% when using XPS to analyse thermal oxides on single crystal silicon of (100) or (111) orientation. For other forms of oxide, more work is needed before this low uncertainty can be used. Furthermore, this value of L_{SiO_2} is only valid for the application of peak area analysis using a Shirley background¹² as described in Refs (1) to (3), with $R_o = 0.9329$ for Mg and Al X-rays. L_{SiO_2} will be very similar in other cases but the stated uncertainty may be increased. In addition to the above uncertainty is the non-linearity of XPS when using equations of the type shown in Eqn (1) without corrections for elastic scattering. This non-linearity is small for light-element solids and is smallest^{13,14} for angles of emission below 60° . This and other small non-linearity contributions are contained within ± 0.025 nm.¹⁵ Thus, in

Table 2. Summary of results using homogenised data for the average values of m and c by method in ascending order of offset value with the standard deviation of the results and, in brackets, standard deviations of the means – an update of Table 13 in Ref. (3)

Method	m	c , nm
XPS – using reference geometry and NPL L and R_o values	1.019 ± 0.029 (0.010)	-0.048 ± 0.121 (0.041)
NR ^a	1.020 ± 0.008 (0.008)	0.116 ± 0.050 (0.050)
NRA ^a	1.101 ± 0.013	0.452 ± 0.122
MEIS	0.973 ± 0.042 (0.020)	0.472 ± 0.131 (0.093)
GIXRR	0.996 ± 0.003 (0.002)	0.522 ± 0.011 (0.007)
RBS, EBS	1.039 ± 0.065 (0.027)	0.538 ± 0.267 (0.133)
TEM	0.941 ± 0.109 (0.039)	0.824 ± 0.426 (0.151)
Ellipsometry	1.010 ± 0.011 (0.004)	Pre-heat: 0.853 ± 0.087 (0.062) No pre-heat: 0.992 ± 0.174 (0.062)

^a Only one result and so the standard deviation is calculated from the fit for m and c .

NR = neutron reflectometry, NRA = neutron activation analysis, MEIS = medium energy ion scattering spectrometry, GIXRR = grazing incidence X-ray reflectometry, RBS = Rutherford backscattering spectrometry, EBS = elastic backscattering spectrometry, TEM = transmission electron microscopy.

terms of the standard uncertainty, the total uncertainty will be 0.66% at 8 nm but rising to 0.76% at 3 nm and 1.05% at 1.5 nm with these uncertainties doubled for 95% confidence.

On the other hand, at the reference geometry, XPS as a general method as conducted in many laboratories appears to have a standard uncertainty³ of around 2.8%, mainly arising from uncertainties in the emission angle, from inconsistencies in the choices of the numbers of peaks to measure, from variations in the algorithms in the software used, as well as variations in the decisions on how to use that software in the different laboratories.²

It should be made clear that the present analysis makes little significant change to the relative results of all the data supplied to us in the earlier interlaboratory study.³ Had the scalings for all the (100) and (111) samples been identical, the result would have simply scaled all the m values and left c unchanged. Since there are small differences in the d^*/d values, the c values, excluding XPS, are typically 0.026 nm lower and the m values $2.5 \pm 0.2\%$ higher. Small relative shifts also occur in the values of m , depending on the precise samples analysed. These mainly occur in the TEM data where, often, only two or three of the ten samples were used. The only significant outlier to this is the NR result where c is reduced significantly from 0.185 to 0.116 nm. Thus, the ratio of the highest (NRA) and lowest (TEM) m values in Table 2 is unchanged. Figure 3(a) shows the updated results for m and c for XPS at the reference geometry (equivalent to Fig. 19 of Ref. (3)). This is consistent with the uncertainties in m attributed to each value from the statistical uncertainty in the fit to Eqn (6) of, typically, 1.4% and a root mean square scatter in the biases of the emission angles of 2° . Figure 3(b) shows the corrected version of Fig. 20 from the previous³ study for the averages for each technique. This shows the general shift of the data points to the right by a factor of approximately 1.025.

CONCLUSIONS

A method has been described using a laser beam and reflectors to allow users to define the angle of emission of electrons for XPS with a precision significantly better than 0.1° and to define their zero in the angle of emission to 0.1° , both as standard uncertainties. Using this system, analysis of our previous method for setting the sample angle of emission using the tilt rotation shows it to be biased to a small extent. A geometrical analysis shows this to be $0.56 \pm 0.28^\circ$ high, whereas an intensity analysis gives $1.89 \pm 0.04^\circ$. Using new intensity measurements, it appears that XPS may be able to achieve an accuracy generally better than 1% in the thickness range 1.5 to 8 nm for thermal oxide on (100) or (111) Si wafers. Using the new data, the results of the previous intercomparison³ are re-evaluated. This shows that previous m values should all be scaled up by factors around 1.025 and that the c values typically reduce by 0.026 nm, except for NR where the new c value is reduced to 0.116 nm. New values for the attenuation length of Si 2p electrons, in thermal SiO₂ on (100) or (111) Si wafers excited by Mg and Al X-rays, are given as 2.996 ± 0.016 and 3.485 ± 0.019 nm, where the uncertainties are the standard uncertainties, respectively.

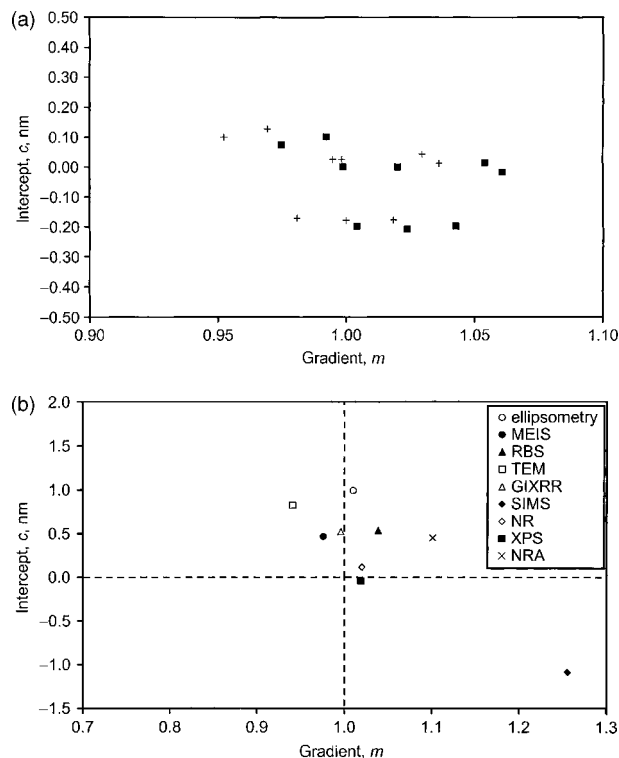


Figure 3. Plot of m and c for the averaged homogenised data (a) for XPS at the reference geometry showing how the data have shifted to the new values (■) from the values used in the earlier study³ (+) (note that the scales are much expanded from previous plots) and (b) the average for each technique updated from Fig. 20 of Ref. (3).

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