REPORT

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Results from a Comparison of Optical Thin Film Thickness Measurement

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Results from a comparison of optical thin film thickness measurements

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ABSTRACT

In this report we present the results of a comparison of non-contact methods for measuring the thickness of thin film artefacts. Only well-controlled calibration artefacts have been measured to assess the base-line measurement capability of instruments used in industry. With the exception of those from one instrument, the results show agreement between the methods, and with the calibrated values, to within 2 % of the nominal height.

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1 Introduction

Thin films are used in a wide range of industries and applications. Examples include ophthalmics, displays, paints, protective layers, anti-reflection coatings, detectors, solar cells, mobile telephone filters, engineering coatings in the automobile and aerospace sectors and many more. The economic benefits of improved surface coatings will be highly significant. To take one example, a recent survey undertaken by the UK government predicted that the value of products in the UK affected to some degree by surface engineering will be over €200 billion *per annum* by the year 2005, conservatively implying a sum of €2000 billion *per annum* on a European scale (Mathews *et al.* 2005). A significant proportion of these products will require accurate or traceable measurement of film thickness.

Traceability of thin film thickness can be achieved using transfer artefacts – essentially samples with a film thickness that has been measured on a traceable instrument (Yacoot, Leach 2007, Belzer *et al.* 1998). The route to traceability is usually via a surface texture measuring instrument. Ellipsometers and other, tomographic instruments are then used as secondary calibration instruments. In practice, it is rare to see the result of a thin film measurement presented with an associated measurement uncertainty; so it is difficult to compare measurement techniques. To address this issue a comparison has been performed in which we have circulated three silicon dioxide on silicon calibration artefacts and the participants have used a number of thin film measurement techniques to measure their optical thickness. The samples were originally supplied with a calibration certificate, traceable to the National Institute of Standards and Technology (NIST) in the USA.

The participants in the comparison were NPL, Intense Ltd., Applied Multilayers Ltd., PANalytical, Ametek Taylor Hobson and the University of Southampton. NPL measured the artefacts before and after the comparison using an ellipsometer. The results have not been identified with the associated participant for reasons of confidentiality.

2 The calibration artefacts

The nominal thicknesses of the samples were 40 nm, 200 nm and 1000 nm. Each sample consisted of thermally grown silicon dioxide (refractive index $n = 1.46$ for a wavelength of 632.8 nm) on a silicon wafer ($n = 3.875$, extinction coefficient, $k = 0.016$ for a wavelength of 632.8 nm). The working area of each sample was a circular region 10 mm in diameter situated in the middle of the wafer. Four arrows are printed on the silicon wafer as a guideline to the position of the silicon dioxide film.

3 The measurement procedure

Four of the participants (Lab 1, Lab 2, Lab 3 and Lab 6) used spectroscopic ellipsometers, Lab 4 used an x-ray reflectometry technique and Lab 5 used a vertical scanning white light interferometer.

Three sets of measurements were made on each silicon wafer. The first set were made on an ellipse, as near as possible to the centre of the disc, when judged by eye. The other two sets were made at two positions north and south, 2 mm away from the first set of measurements, as shown in Figure 1.

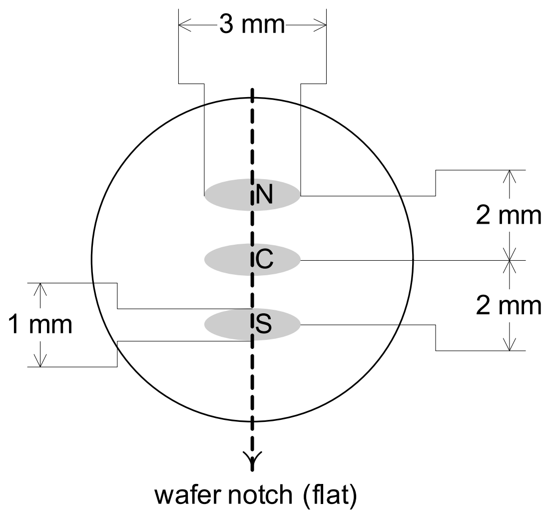

Figure 1 Diagram of measurement positions on silicon wafer

4 Results

Table 1 presents the results of the comparison and Figure 2 to Figure 4 present the results graphically. “NIST” refers to the values on the calibration certificate. The results from Lab 3 have not been included in the graphs as they are very different to the results from the other participants. The participants have provided standards deviations but the number of degrees of freedom was not generally available.

Table 1 Results of comparison, Std Dev is the standards deviation of the results

Laboratory	**50 nm sample**		**200 nm sample**		**1000 nm sample**	
	Value /nm	**Std Dev /nm**	**Value /nm**	**Std Dev /nm**	**Value /nm**	**Std Dev /nm**
NIST	47.00	0.40	191.20	0.40	1055.70	1.10
Lab 1	47.28	0.57	193.57	0.51	1062.79	0.82
Lab 2	46.74	0.44	192.06	0.049	1056.85	0.17
Lab 3	-	-	238.00	12.00	1669.00	83.50
Lab 4	46.50	0.50	189.00	0.50	-	-
Lab 5	47.47	1.10	200.04	0.014	1075.83	0.82
Lab 6	47.049	0.075	192.87	0.15	1060.93	0.20
Lab 1	47.13	0.24	192.81	0.19	1060.24	0.42

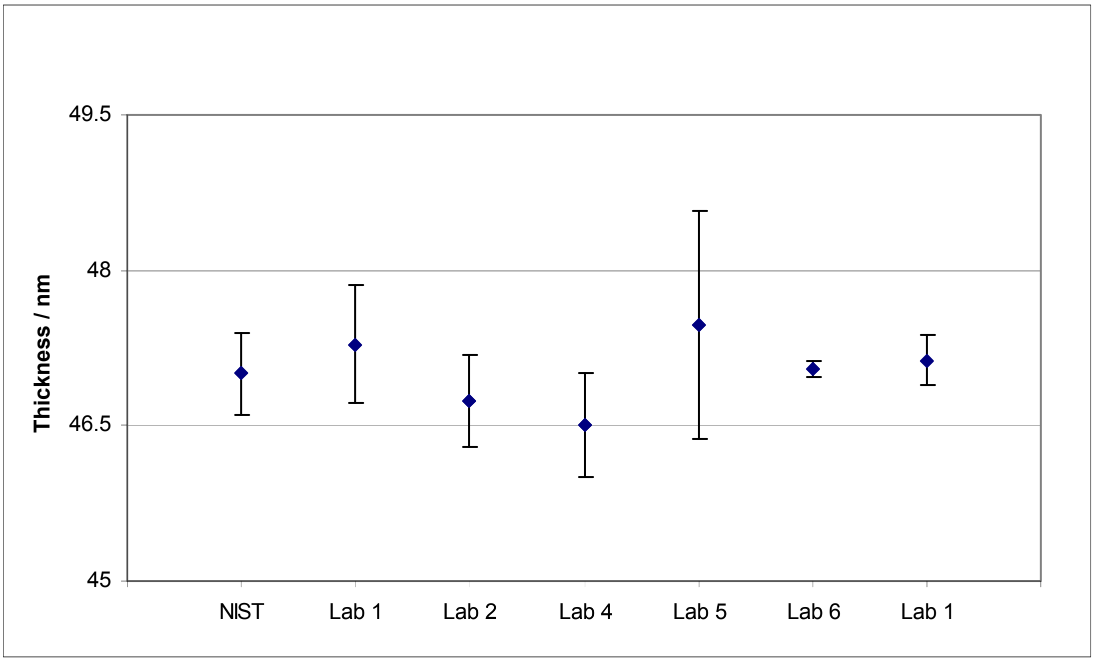

Figure 2 Comparison results on 50 nm film thickness sample

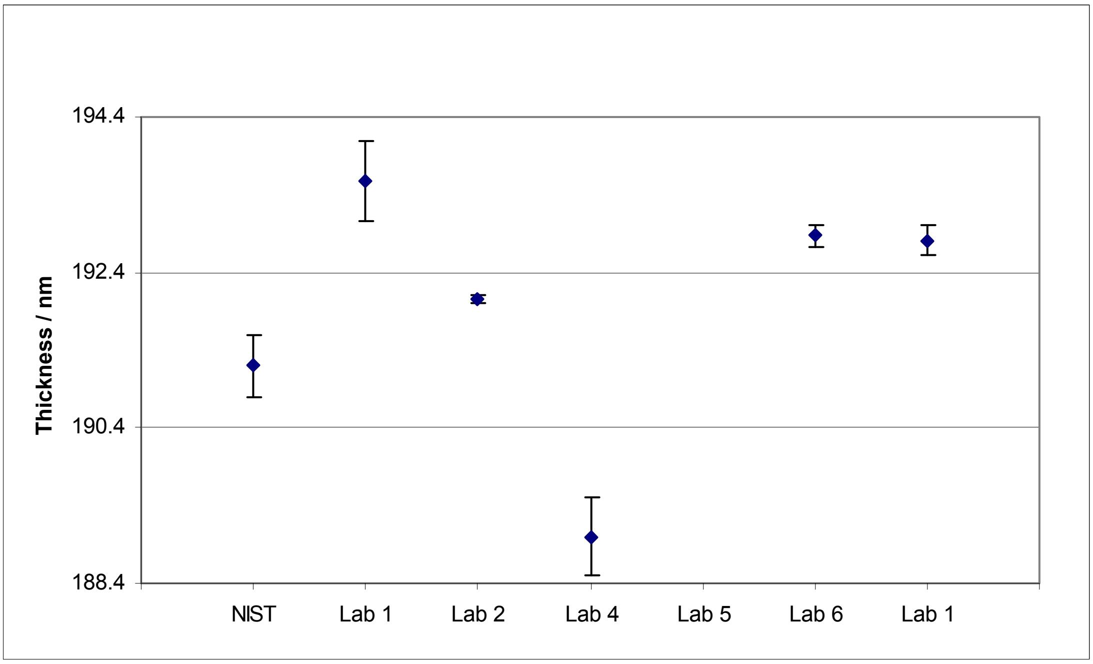

Figure 3 Comparison results on 200 nm film thickness sample. Note that Lab 5 result does not appear on this chart because of the contracted thickness scale

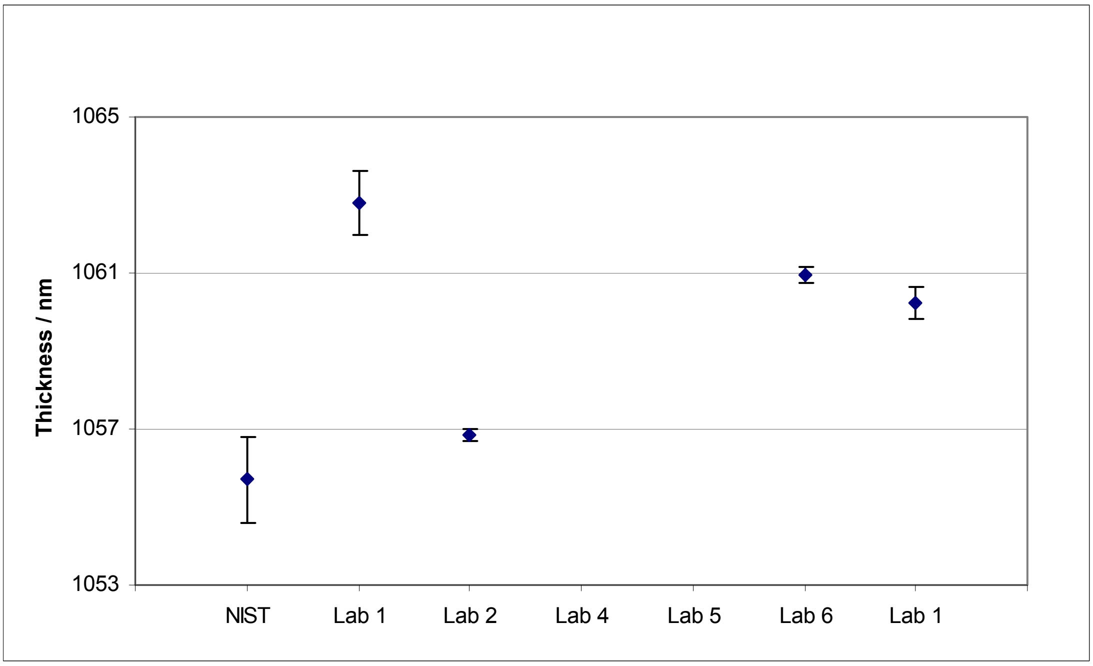

Figure 4 Comparison results on 1000 nm film thickness sample. Note that Lab 5 result does not appear on this chart because of the contracted thickness scale. Note also, there is no result for Lab 4 for this artefact

The NPL (Lab 1) results at the start and finish of the comparison are within their combined standard deviation. The results for Lab 3 are clearly in gross disagreement with those from the other laboratories and are most probably caused by error in the set up of the instrument (the relevant participants could not find the source of the error). Lab 4 could not measure the 1000 nm sample as it was out of the range of the x-ray instrument. With the exception of the 1000 nm sample, the results from Lab 1, Lab 2 and Lab 6 are all in very good agreement (they all used ellipsometers). The results from Lab 5, taken using vertical scanning white light interferometry, show a small systematic deviation from the results from the ellipsometers. With the 1000 nm sample, there is noticeable spread in the results that cannot easily be put down to instrument noise. This variation is unlikely to be due to a change in dimension of the sample, but is more likely due to systematic length-dependent uncertainties, for example the uncertainty in the value for the optical constants.

5 Conclusion

The results from this comparison show that ellipsometers and the white light interferometer are in good agreement. Where the x-ray method could be compared the results were also good.

The spread in the data could be attributed to general instrument noise, a difference in spot sizes and the condition of the samples (cleanliness, laboratory humidity, *etc.*). However, the results from one participant (Lab 5) showed an alarming difference that can be attributed to the way the samples were set up. We can conclude that for the majority of the instruments thin film measurements on these simple architecture samples give results that are within 2 % of the calibrated value.

6 Acknowledgements

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7 References

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