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Review Article

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Studies of XPS Technique and Application in Thickness Measurement of Ultra-Thin Film

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ABSTRACT

X-Ray Photoelectron Spectroscopy (XPS) analysis technique has been widely applied in semiconductor manufacturing and failure analysis. We used it for defects analysis and thin film characterization in wafer fabrication and for XPS valence state analysis of copper materials. XPS technique is also applied jointly with TOF-SIMS technique. In wafer fab, semiconductor & LED manufacturing, it is very challenging to measure the thickness of an ultra-thin film at nano meter range. In general, TEM is widely used for ultra-thin film physical measurement but usually its lateral dimension is limited. In this paper we will study X-Ray Photoelectron Spectroscopy analysis technique which employ a new analysis method by using angle resolved analysis technique. Furthermore, we have applied the new method in analysis of SiON film. It is achieved to measure an ultra-thin film at about 1.4nm. This method can be used for SiO_2 thickness measurement, self-assembled thiol monolayer on Au and thickness of HfO_2 on silicon substrate.

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Introduction

X-Ray Photoelectron Spectroscopy (XPS) technique has been widely applied in semiconductor manufacturing for material analysis and failure analysis. Especially XPS analysis technique can provide information of the valence state of the materials. In our previous studies, we used it for defects analysis and characterization in wafer fabrication and for studies on XPS valence state analysis of copper materials [1-3]. XPS technique is also applied jointly with TOF-SIMS technique so as to obtain more accurate analysis results [4].

It is well-known that in wafer fab, semiconductor & LED manufacturing, it is very challenging to measure the thickness of an ultra-thin film at nano meter (nm) range. In general, TEM is widely used for ultra-thin film physical measurement as its resolution could be achieved to 0.12 nm. We will study X-Ray Photoelectron Spectroscopy (XPS) analysis technique which comes up with a new analysis method by using angle resolved analysis technique. We have applied it in analysis of $S_i O_N$ film. It has achieved to measure an ultra-thin film at about 1.4nm. This method can be used for SiO2 thickness measurement, self-assembled thiol monolayer on Au and thickness of HfO_2 on silicon substrate.

Experimental

In this study, the experiments and measurements were carried out by using Scanning XPS Microscope PHI Quantera II (Figure 1).

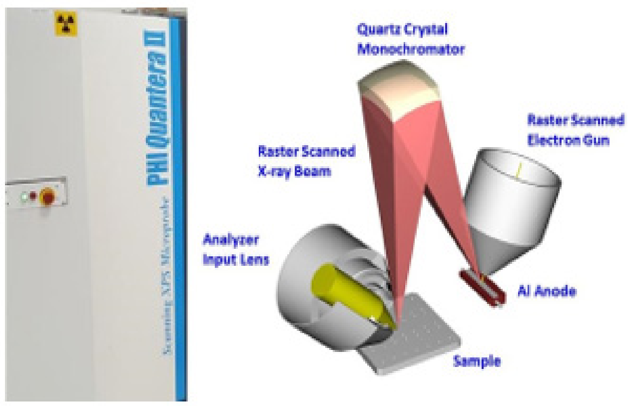


Figure 1: The Advanced XPS Machine was Used for Experiments in this Study

In the experiments, Al source (at 1486.6 eV) was applied and minimum X-ray beam size could be at 7.5 µm.

This advanced XPS machine has the function of the angle resolved analysis, which is shown in Figure 2. During operation, the analysis penetration depth is varied by changing the sample tilt angle at 15^0, 25^0, 35^0 45^0, 60^0, 75^0, 90^0.

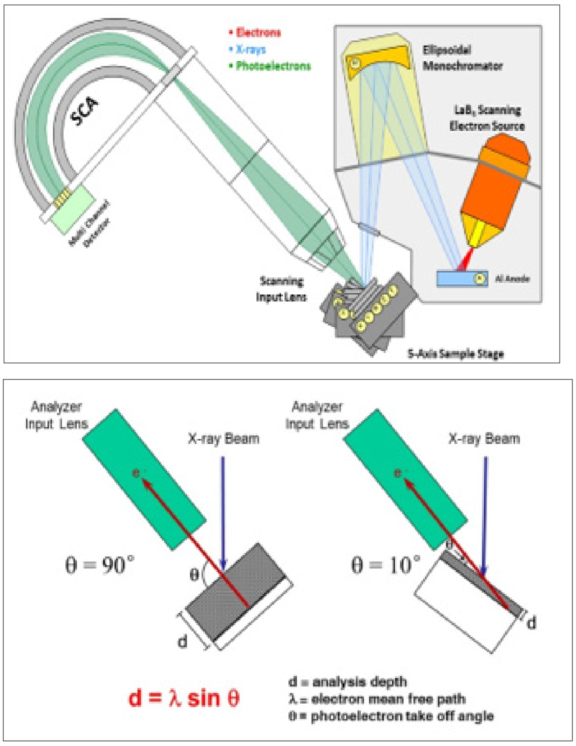


Figure 2: This Advanced XPS Machine has the Function of XPS Angle Resolved Analysis

Theretical Study

In XPS analysis, the signal depth (d) is considered as a function of 3λ (the attenuation length for a photoelectron emitted within a uniform material). By tilting the XPS sample, the take-off angle (TOA, θ) of photon electron can be adjusted. After stage tilting, the signal depth(d) becomes:

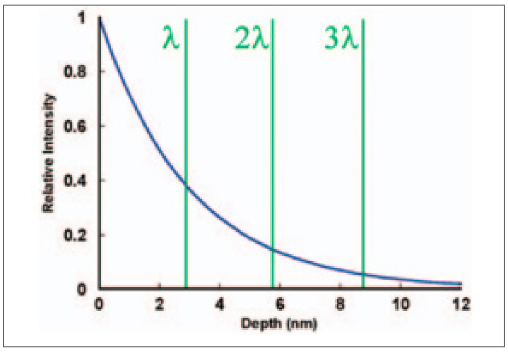
$$d=3\lambda \sin\theta \qquad (1)$$

In this case, the take-off angle (TOA, θ) vs sinθ values are shown in table 1. Thus Angle-Resolved XPS can be applied if very top layer of sample which is thin as 5nm is requested.

Major Applications of XPS

The major applications of XPS are:

- Surface analysis of inorganic and organic materials, contamination, stains or residues;
- Quantification of surface elemental composition;
- Determination of chemical state/binding information;
- Depth profiling.
- Film/material and oxide thickness measurement;
- Line scans & area mapping;
- Angle resolved analysis.



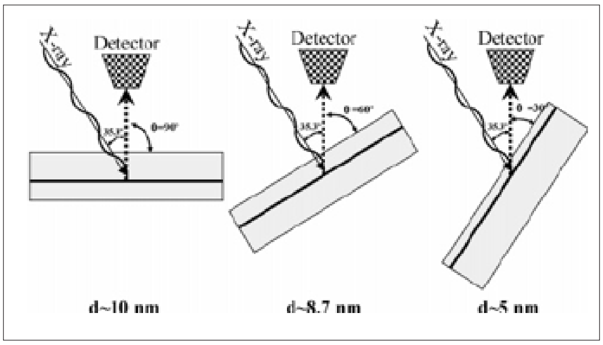


Figure 3: In XPS Analysis, the Signal Depth (d) is Considered as: 3λ.

Table 1: The Take-off Angle (toa, θ) vs sinθ Values

TOA(θ)	sinθ
15	0.26
30	0.5
45	0.71
60	0.87
75	0.97
90	1

Results and Discussions

In this study, an ultra-thin film of S_iO_N was measured by using the method developed. The scan results of Si and Si-O from the take-off angle (TOA, θ) of 15^0- 90^0 are shown in Figure 4 and those of N1s and O1s are shown in Figure 5-6.

The known thickness from the customer is about 1.40nm. We measured the thickness at the 6 locations and the results have been summarized in Table 2.

Based on the XPS analysis results, one can see that the average result of the 6 measurements is 1.46nm, which is well agreed with the known thickness (1.40nm) of S_iO_N film.

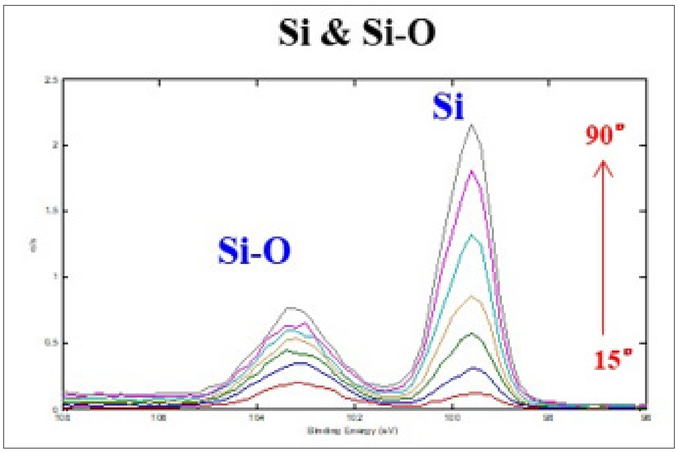


Figure 4: The Scan Results of Si and Si-O from the Take-Off Angle (TOA, θ) of 15^0- 90^0

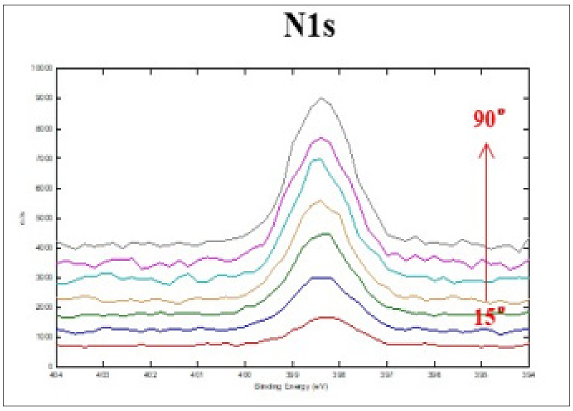


Figure 5: The Scan Results of N1s from the Take-Off Angle (TOA, θ) of 15^0- 90^0

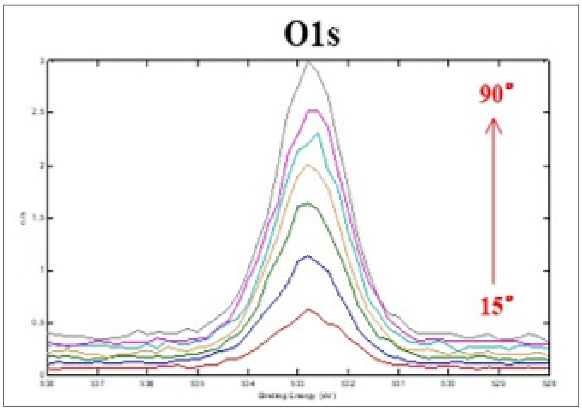


Figure 6: The Scan Results of O1s from the Take-Off Angle (TOA, θ) of 15^0- 90^0

Table 2: XPS Analysis Results of Thin S_iO_N layer at 6 Measurement Points (Known thickness of S_iO_N ~1.4nm)

Location	Thickness Measurement (nm)
Bottom	1.42
Bottom left	1.51
Right	1.43
Top Right	1.43
Top	1.46
Top Left	1.50
Average	1.46

Notice: XPS analysis results show that the average result is well agreed with known thickness of SiO_N ~1.40nm

Application

In this Angle resolved XPS application, a test sample of ultra-thin W-O-N layer(<5nm) on top of SiO_2 was applied. The purpose is to measure the atomic percentage of each element at W-O-N layer without interruption from SiO_2. Normal Angle XPS analysis method will not be applicable due to the signal depth is larger than W-O-N layer thickness. A depth profile is used to roughly study the W-O-N thickness (Figure 7). The scan results of S_i2_p, O1s, N1s and W4f are shown in Figure 8.

Based on XPS results, the $Si2_p$ peak was not showing up at 15-degree TOA angle resolved analysis, which indicatethat Si_2p photoelectron is not collected by the detector at this take off angle.

The signal depth is within the top W-O-N layer after tilting at 15-degree. The result of W-O-N ratio is shown in Table 3 and it matches with the simulation model during synthesis.

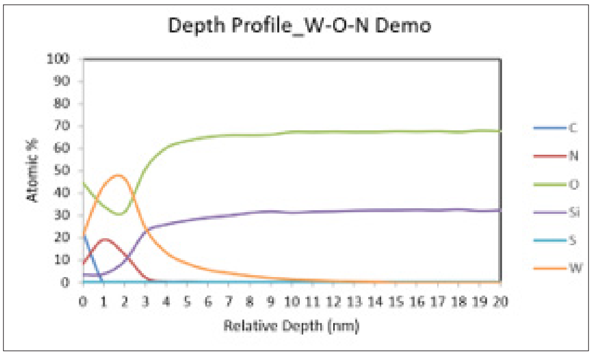


Figure 7: The Depth Profile of W-O-N layer on top of SiO_2 substrate. The thickness of W-O-N is detected at around 3nm at sputter rate of SiO_2.

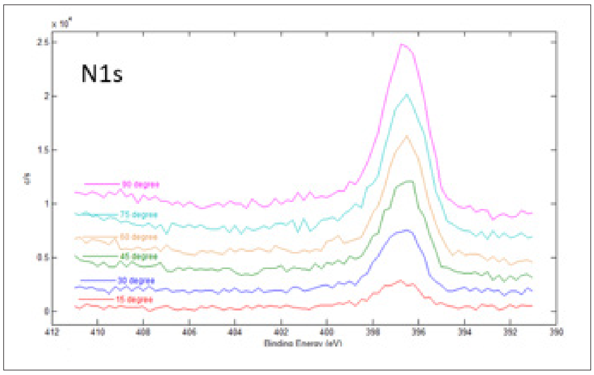


Figure 8: The Scan Results of N1s from the Take-Off Angle (TOA, θ) of 15^0- 90^0

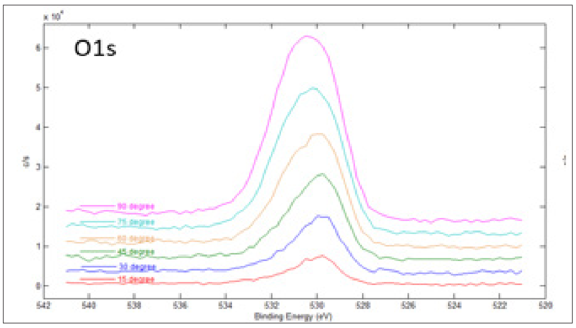


Figure 9: The Scan Results of O1s from the Take-Off Angle (TOA, θ) of 15^0- 90^0

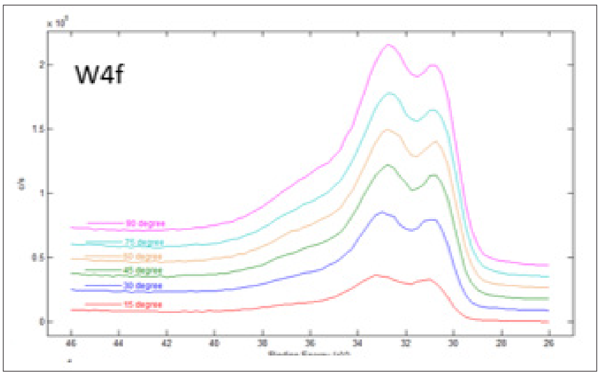


Figure 10: The Scan Results of W4f from the Take-Off Angle (TOA, θ) of 15^0- 90^0

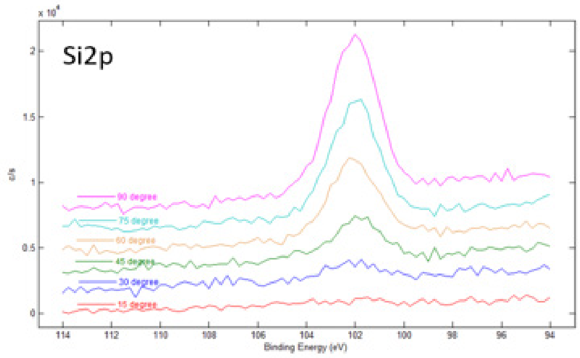


Figure 11: The Angle Resolved Scan data of W-O-N layer on top of SiO_2 substrate. Si 2p signal was not presented at 15-degree take-off angle (TOA).

Table 3: XPS Elemental Composition at Different Take-off Angle (TOA)

TOA(o)	N	O	Si	W
15	15.0	33.4	0.0	51.6
30	15.8	31.4	4.8	48.0
45	16.1	32.6	7.1	44.2
60	14.9	35.4	10.1	39.6
75	14.1	38.2	11.2	36.5
90	12.7	39.7	12.3	35.3

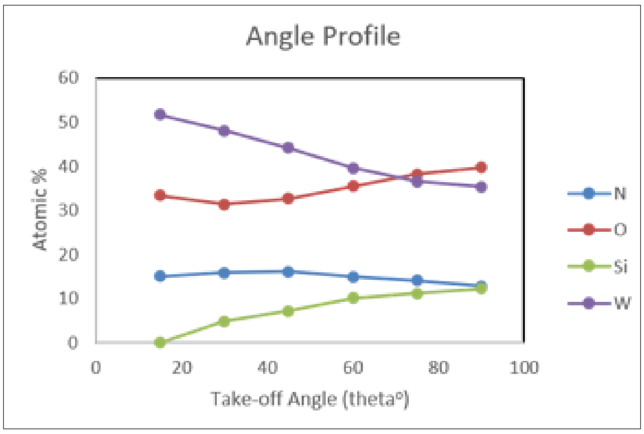


Figure 12: The Angle Resolved Profile of W-O-N layer on top of SiO_2 substrate

Conclusion

An XPS angle resolved analysis method is studied and discussed by using Scanning XPS Microscope PHI Quantera II. It has been successfully applied for thickness measurement for the ultra-thin film S_iO_N at nm range. XPS analysis results show that the average thickness (1.46nm) is well agreed with known thickness of S_iO_N (1.40nm). Anther application is also reported as using Angle Resolved XPS to achieve ultra-thin film chemical composition analysis with a decreased signal depth with the decrease of the Take-Off Angle (TOA). Moreover, this method can be used for SiO_2 thickness measurement; self-assembled thiol monolayer on Au and thickness of HfO_2 on silicon substrate.

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