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A Multi-Technique Approach for a Complete Thin Film Characterisation

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Here we provide an overview of a multi-technique investigation of layered thin film and ultra-thin film coatings. Surface sensitive analytical techniques are chosen for their different sampling depths and include a combination of conventional Al Kα and higher photon energy Ag Lα excited XPS, angle resolved X-ray photoelectron spectroscopy (ARXPS) with maximum entropy method (MEM) reconstruction of concentration depth profiles and argon cluster depth profiling.

Introduction

The application of thin film technology is of commercial importance across a range of industries and is commonly used to influence both the physical and chemical properties of bulk materials. Ranging in thickness from tens of Angstroms to microns, they are applied across a broad range of disciplines including the semiconductor, biomaterial and energy harvesting industries. Here, we provide a multi-technique investigation of a bi-layer thin film used as a model system for gate oxide structures. The combination of techniques allows a complete picture of the chemistry of these materials to be built with the understanding of how subtle differences in chemistry and stoichiometry can influence the properties of a substrate to enhance its application specificity. Figure 1 shows in a schematic form the relative sampling depths of the different analytical techniques used in this study.

Results and Discussion

Here we outline the multi-technique work flow and relate the results from the different analytical techniques to the surface characterisation of the thin film sample under investigation.

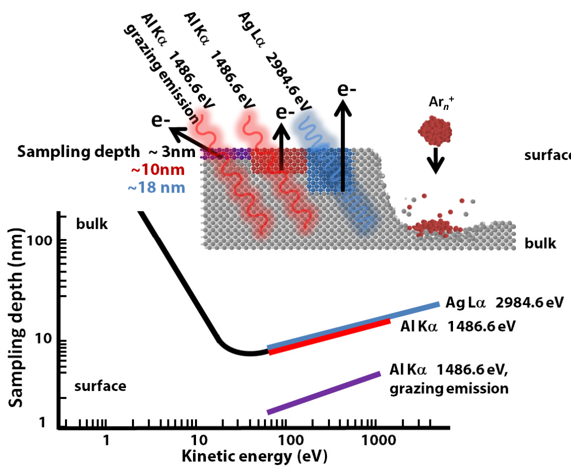

Fig.1 Illustrative schematic of experimental analytica.

The model sample used is a 2 nm HfO_2 film on 1 nm SiO_2 on a silicon substrate. Initial analysis is performed using monochromated Al Kα (1486.6 eV) X-rays to excite the photoemission spectra. This conventional XPS has an information depth of *ca.* 10 nm which can be decreased to 1 – 3 nm by changing the take-off angle of the photoelectrons in an angle resolved XPS experiment (ARXPS). The ARXPS data is used to calculate a reconstructed concentration depth profile using maximum entropy method (MEM) [1]. The result is shown in Figure 2. Reassuringly, the reconstructed depth profile shows that the HfO_2 film has a thickness of 3 nm, SiO_2 is 1.5 nm which is in broad agreement with the expected sample layer thicknesses. It is further noted that there is a 1 nm thick adventitious carbon layer at the outermost surface of the sample.

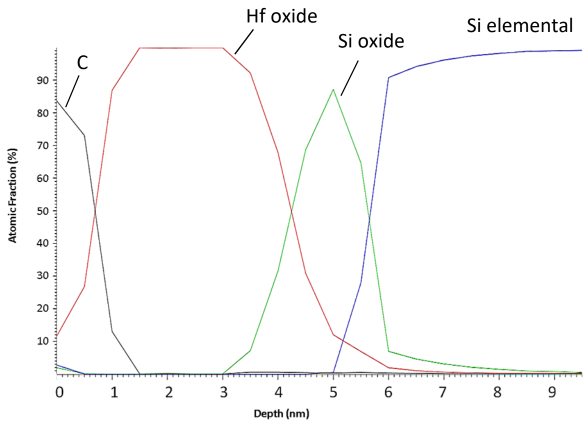

Fig. 2 Concentration depth profile from ARXPS data reconstructed using MEM.

In contrast to increased surface sensitivity, high energy excitation sources can be used to increase the analysis depth of a material to >15 nm. For the $HfO_2/SiO_2/Si$ sample, the use of a monochromated Ag Lα (2984.2 eV) X-rays will probe deeper into the bulk structure [2]. The use of higher energy X-ray sources also allows the excitation of higher binding energy core-levels not normally accessible with a conventional Al Kα excitation source. This concurrently affords higher kinetic energy electrons to be emitted and therefore offers greater sampling depths.

In Figure 3a) and b) we show the Al and Ag excited Si 2p regions respectively. It can be seen empirically that the Al Kα excited spectrum has a greater contribution of the SiO_2 components than the Ag Lα excited spectrum which is probing more of the elemental silicon substrate. This is supported by calculating the ratio of elemental $Si:SiO_2$ which is 10.1 for Ag Lα excitation and 4.5 for Al Kα excitation.

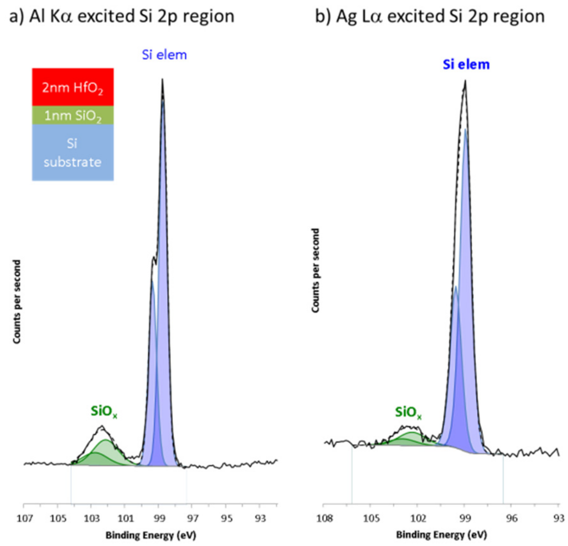

Fig. 3a) Al Kα excited spectrum and **3b)** Ag Lα excited spectrum of the $HfO_2/SiO_2/Si$ sample.

Conclusion

Here it has been shown that a model thin film bilayer sample can be characterised by conventional Al Kα excited XPS. The use of ARXPS to provide data for MEM calculated concentration depth profile allows the layer thicknesses and order to be determined. Ag Lα excited XPS extends the sampling depth and corroborates the information provided by the convention excitation source. In light of this, it is demonstrated that with a multi-technique approach to sample characterisation, it is possible to build a more detailed description of the structure of these thin film materials and their use in relevant applications.

References

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