Application Note: 31049

Parallel Angle-resolved XPS Analysis of a Self Assembled Monolayer with the Theta Probe

Key Words

- Film Thickness Measurement
- Non-Destructive Depth Profiling
- Surface Analysis

Self-assembled monolayers (SAMs) of alkanethiols on gold form organic layers whose chemical properties are determined by the functional groups on the thiol molecule.

Potential applications include sensor technology, transducers, protective layers and lubricants. They also have a role as model systems for biological interfaces. See reference 1 for a review of the formation and structure of SAMs.

These materials form ultra-thin, uniform layers. The layer thickness of these films is a good match to the sampling depth of XPS and angle resolved XPS (ARXPS). Thermo Scientific Theta Probe is therefore well suited to the analysis of these materials.

Specimen

A gold surface was prepared by evaporating a film of the metal onto a silicon wafer. The SAM was prepared by depositing dodecanethiol ($C_{12}H_{25}SH$) on the gold surface from solution. The structure is illustrated in Figure 1.

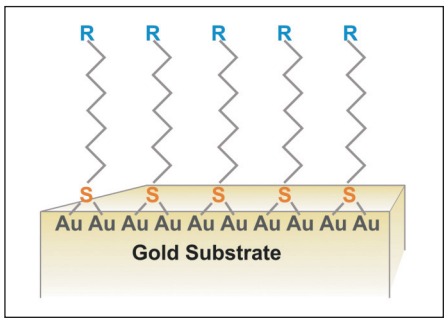

Figure 1: Self assembled monolayer on a gold substrate, (in this case R = C_5H_9)

Analysis

This material was analyzed in a Theta Probe instrument using Parallel Angle-Resolved XPS. All of the data were collected using a 400 µm spot of Al Kα X-rays from a microfocusing monochromator.

XPS spectra were collected from the C 1s, S 2p and Au 4f regions in 16 angular channels, each 3.75° wide.

The ordering of the elements in the layers was confirmed using the relative depth plot, a feature of the *Avantage* data system. This is shown in Figure 2.

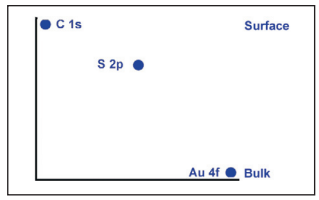

Figure 2: Relative depth plot for the SAM on gold.

The thickness of the layer was calculated using the multi-layer thickness calculator, also part of the *Avantage* data system.

The thickness of the SAM layer was found to be 1.6 nm. The chain length for the dodecanethiol molecule is about 1.8 nm. If the measured layer thickness is correct, this implies that the molecules are tilting by about 27° from the surface normal. This value is close to that reported in the literature. It should be noted that the C 1s signal might include some carbonaceous contaminant, if present in the layer.

A non-destructive depth profile, Figure 3, was constructed from the ARXPS data using a technique involving Maximum Entropy methods (this method is described in application note 31014).

The profile shows good depth resolution and accurately reproduces the thickness of the SAM layer.

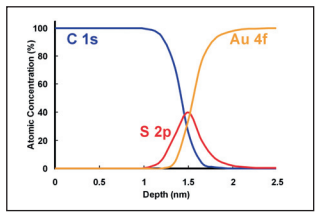

Figure 3: Non-destructive depth profile of the self-assembled monolayer.

Conclusions

ARXPS is a powerful tool for the characterization of SAMs. Layer thickness and molecular tilt angles can be calculated. The results are close to the values published in the literature.

The depth profile, constructed from the ARXPS data, was found to accurately reflect the structure of the specimen.

References

1. A. Ulman, Chem. Rev. 96 (1996) 1553.

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