PHYSICAL ELECTRONICS
A Division of ULVAC-PHI

imec

# Use of Lab-based HAXPES to Eliminate the Effect of Ion Beam Damage at Interfaces in Depth Profiles

## Overview

The PHI *Quantes* is the only automated, high-throughput lab-based Hard X-ray Photoelectron Spectroscopy (HAXPES) instrument on the market. It is a unique scanning X-ray photoelectron microprobe that combines a high energy (hard) monochromatic X-ray source (Chromium Kα, *hv* = 5414.7 eV) with a conventional monochromatic soft X-ray source (Aluminum Kα, *hv* = 1486.6 eV). The analytical information depth using the Cr source is about three times deeper than with the Al source. Having a larger sampling depth opens opportunities for probing thicker film structures and buried interfaces, as well as minimizing the effects of surface contamination and ion-induced chemical damage during depth profiling.

Using the Cr X-ray source, quantitative depth profile interface analysis of multilayer films, with accurate chemical state information, can be reliably obtained from ion-beam sensitive materials. The deeper sampling depth of the Cr source allows one to probe beyond the depth of possible damage induced by ion sputtering.

## Sample Measurements

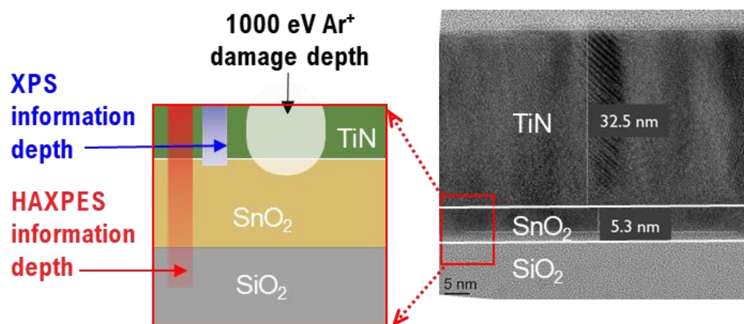


*Figure 1. A TiN/$SnO_2$/$SiO_2$/Si sample used for sputter depth profiling. The structure illustrated (left) is at the point during the depth profile when 2-3 nm of TiN remains. At this point, the damage caused by monoatomic $Ar^+$ is on the same scale as the XPS sampling depth, while the Cr X-rays probe a deeper layer beneath the damage. A TEM image (right) indicating the individual layer thicknesses is also shown.*

## Results

For a direct comparison of the decreased sensitivity to ion beam damage using a Cr source versus an Al source, depth profile spectra were obtained on a stack of 30 nm TiN/ ~5 nm $SnO_2$ films on a $SiO_2$/Si substrate (see Fig. 1) using both X-ray sources. Figure 2 shows the Sn atomic concentration as a function of sputter time calculated from both depth profiles. Sn is first detected in the Al-based data at approximately 28 minutes but, due to an increase in sampling depth, Sn is detected in the Cr-based profile as early as 16 minutes.

Figure 3 shows three high-resolution spectra of the Sn $3d_{5/2}$ transition using both X-ray sources: a) the first spectrum with high enough intensity and signal to noise to recognize the spectral shape, b) the sputter cycle immediately following that spectrum and c) the spectrum at the profile where maximum Sn concentration is detected. Figure 2 identifies the sputter times of the spectra selected for comparison.

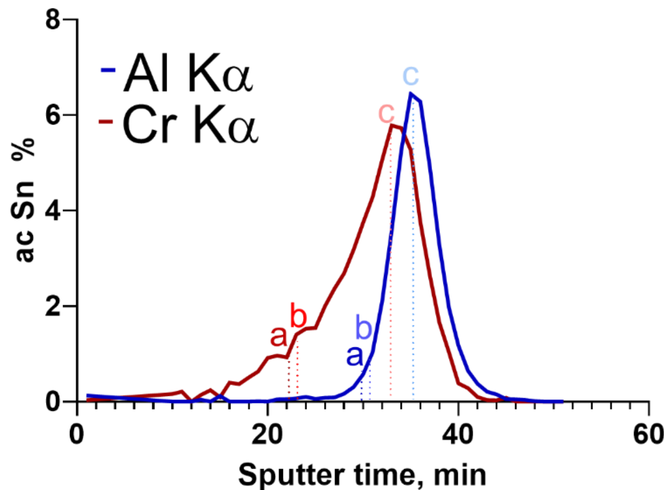


*Figure 2. Atomic concentration of Sn as a function of sputter time calculated from data obtained using Al Kα and Cr Kα sources. Sn is detected much earlier in the Cr profile than in the Al profile due to a ~3x deeper sampling depth of the Cr source. The spectra shown in Figure 3 are at 30, 31, and 37 minutes for the Al profile and at 21, 22, and 33 minutes for the Cr profile.*

In the Al Kα data, there is a peak at ~ 486.8 eV due to the Sn (IV) chemical state and a smaller peak at ~485 eV, suggesting ion-induced reduction to the Sn (0) state. The extent of Sn reduction increases during the depth profile. The sampling depth of Sn 3d photoelectrons using the Al X-ray source is ~5 nm. The damage observed in the very first Sn 3d spectrum indicates that the extent of ion beam damage is on the same scale of depth even though a low argon ion energy (1 keV) and a high data point density were used in the profile.

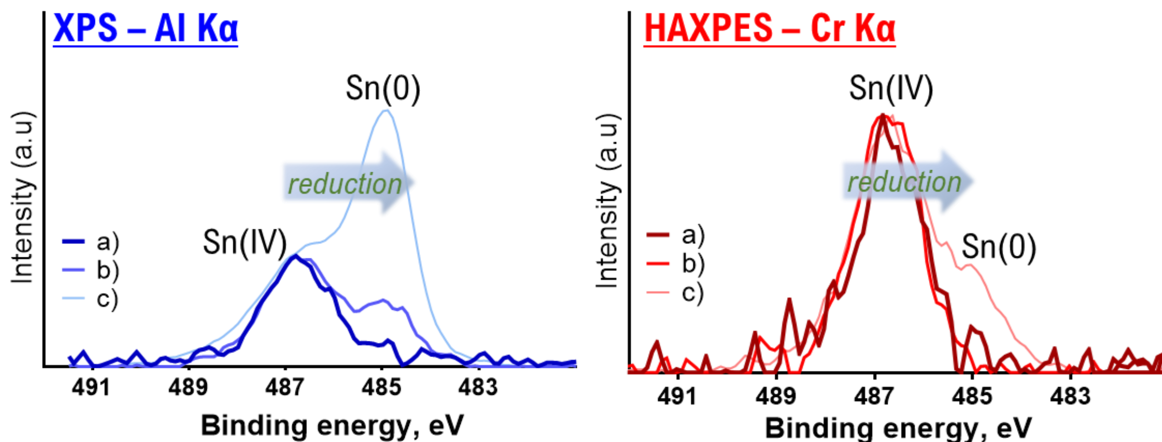


*Figure 3. High resolution $Sn3d_{5/2}$ spectra acquired at specific sputter times labeled in Figure 2 using Al Kα and Cr Kα sources. Spectra were normalized to the peak at 486.8 eV for Sn (IV) state*

With analysis performed using the Cr X-ray source, ion sputtering can be done for much longer times, after first detecting Sn, without observing ion-beam induced damage. The degree of reduction observed in HAXPES data at the maximum concentration of Sn is much smaller than in XPS data obtained using the Al X-ray source.

Importantly, the chemical composition at the interface of interest is not affected by ion beam damage when the higher energy X-ray source is used. A combination of both soft and hard X-ray sources allows estimating the depth and extent of damage caused by the ion beam.

**Conclusions**

Quantitative depth profiling of buried interfaces with accurate chemical information can be obtained using a hard X-ray source even on ion-beam sensitive materials, thus opening new capabilities for multilayer film analysis using laboratory XPS instruments.