

Lab course: X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES)

Do not carry out any action with the NanoSAM without confirmation by the supervisor! Vacuum valves are operated by the supervisor only!

1 NanoSAM setup

Our NanoSAM (SAM = scanning Auger electron microscope) consists of a scanning electron microscope with hemispherical analyzer (Figure 1). It can be used to investigate the chemical surface composition with a lateral resolution down to 10 nm via Auger electron spectroscopy. An integrated X-ray source is used for X-ray photoelectron spectroscopy.

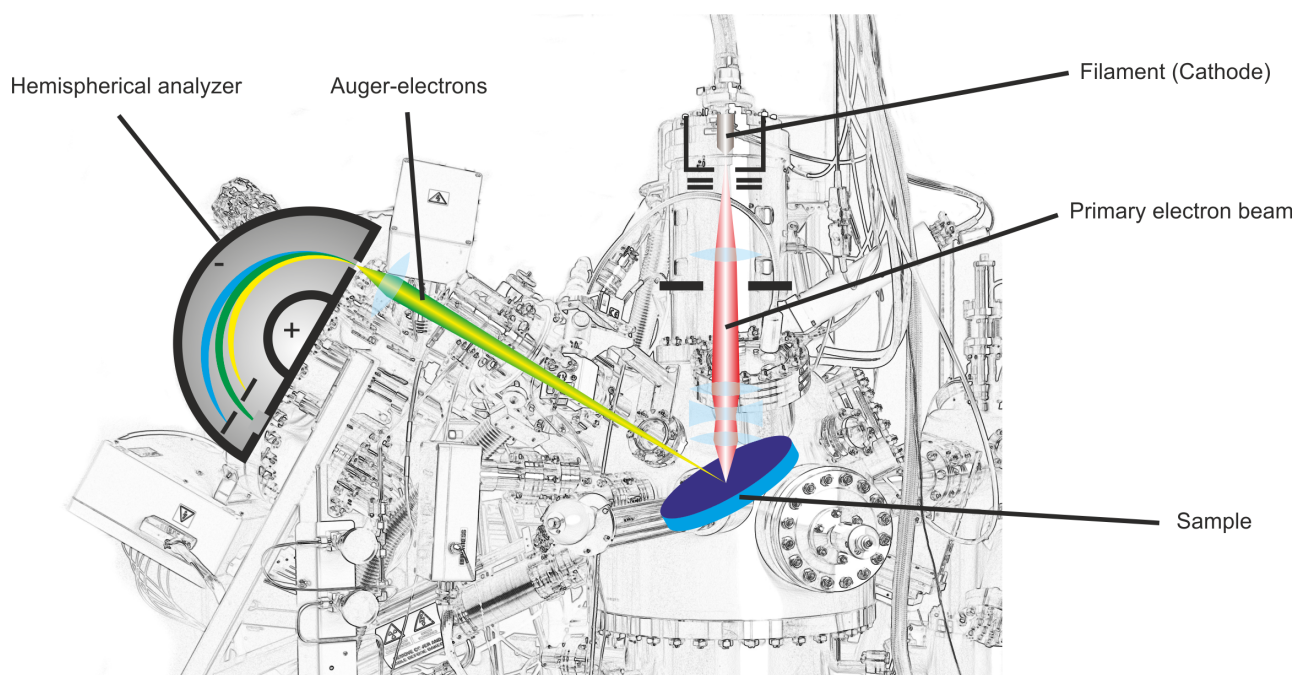


Figure 1: Schematic sketch of the NanoSAM and the relevant electron-paths

2 Samples

You will investigate three different samples. The first sample is a Si(100) wafer with a native surface oxide layer. The second sample consists of a thin film of copper-hexadecafluorophthalocyanine ($\text{=F}_{16}\text{CuPc}$, see Figure 2 for structural formula) deposited on natively oxidized Si(100). The final sample is a 7075 aluminium alloy.

3 Experimental tasks

Sample 1: natively oxidized silicon

1. Measure X-ray photoelectron spectra from 0 eV to 600 eV binding energy for two different sample angles.

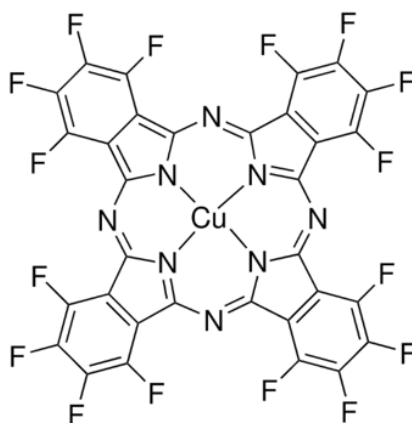


Figure 2: Structural formula of F₁₆CuPc

2. Measure a local Auger electron spectrum up to 600 eV kinetic energy. Remove the oxide layer by argon sputtering. Measure the spectrum again.

Sample 2: F₁₆CuPc on natively oxidized silicon

1. Measure the full X-ray photoelectron spectrum using the magnesium and the aluminium anode.
2. Measure the spectrum of the C 1s emission range.

Sample 3: 7075 aluminium alloy

1. Using the scanning electron microscope, locate dissimilar regions of the sample (e.g. precipitations).
2. Measure Auger electron spectra of these regions.

4 Data analysis and discussion

Sample 1: natively oxidized silicon

1. Calculate the thickness of the oxide layer using the intensities of the Si 2p and the O 1s peak at different angles and the energy-dependent escape depths of the photoelectrons. A database for the inelastic mean free paths will be provided.
2. In which respect does the Auger electron spectrum change after the removal of the oxide layer.

Sample 2: F₁₆CuPc on natively oxidized silicon

1. Assign the peaks in the full X-ray photoelectron spectrum to the respective chemical elements. Compare spectra measured with different anodes to discriminate between photoelectron and Auger electron peaks. Use the F 1s peak to calibrate the energy axis.
2. Estimate the energy resolution of the experiment using the F 1s peak.
3. Analyze the composition of the C 1s detail spectrum.

The following websites are helpful for the assignment of peaks:

<https://srdata.nist.gov/xps/>

<https://www.thermofisher.com/de/de/home/materials-science/learning-center/periodic-table.html>

Sample 3: 7075 aluminium alloy

1. How does the surface composition differ between different regions?
2. *If possible:* Calculate the surface composition of the different regions for a quantitative comparison.