Lab course: X-ray photoelectron spectroscpy (XPS) and Auger electron spectroscopy (AES) with the NanoSAM

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 a 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.

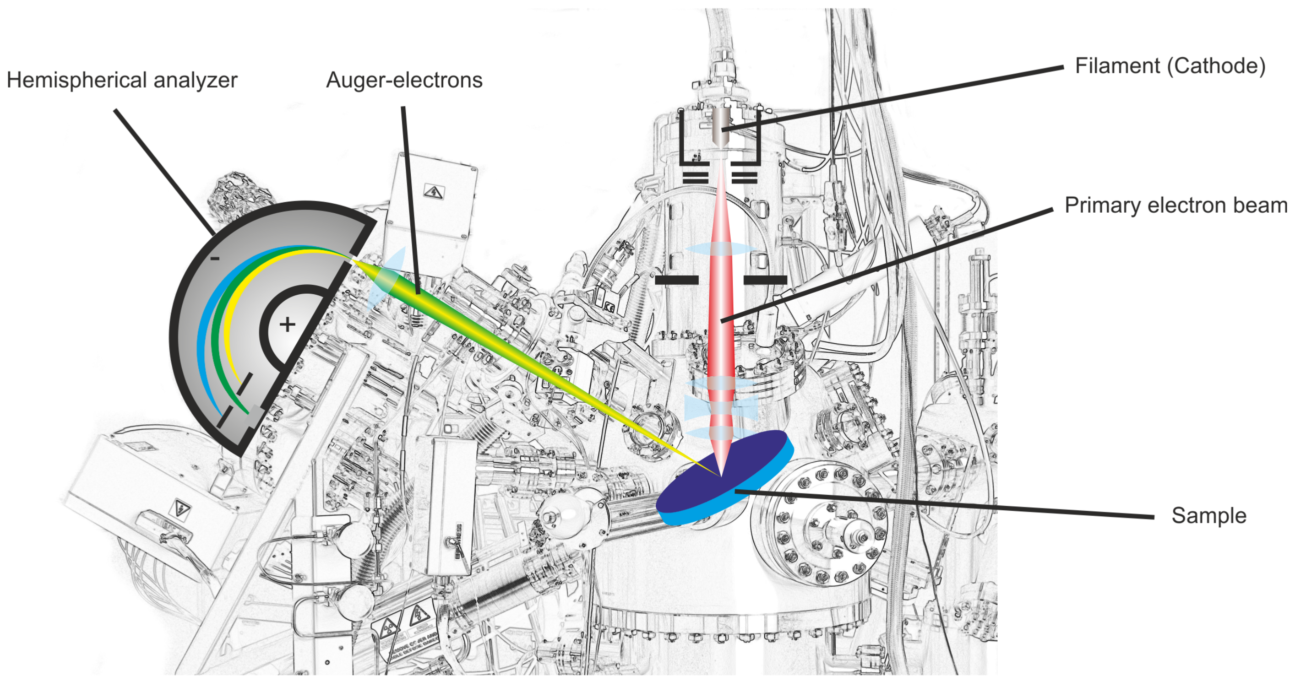

Figure 1: Schematic of the NanoSAM and the relevant electron-paths

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 (=$F_{16}CuPc$, see Figure 2 for structural formula) deposited on natively oxidized Si(100). The third sample is a 7075 aluminium alloy.

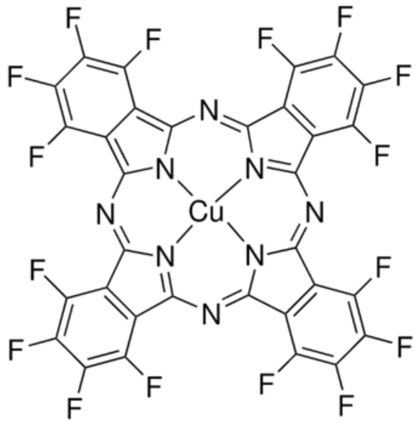

Figure 2: Structural formula of F_{16}CuPc

3 Experimental tasks

Sample 1: natively oxidized silicon

1. Measure a local Auger electron spectrum from 0 eV up to 1800 eV kinetic energy. Remove the oxide layer by argon sputtering. Measure the spectrum again.

Sample 2: F_{16}CuPc on natively oxidized silicon

1. Measure full X-ray photoelectron spectra using the magnesium and the aluminium anode.

2. Measure detail spectra over both the F 1s and 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. Compare the Auger electron spectra before and after argon sputtering. Discuss the observed changes in intensity, energy, and line shape.

2. Calculate the thickness of the oxide layer from the intensities before and after sputtering of the Si LVV transition at 96 eV (which corresponds to elemental Si).

Sample 2: F_{16}CuPc 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. *Hint: shake-up satellites might be present in the spectrum.*

3. Analyze the composition of the C 1s detail spectrum: Deconvolve the spectrum into several peaks which correspond to the different carbon species in $F_{16}CuPc$. Compare their relative intensities with your expectation. *Hint: shake-up satellites might be present in the 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.