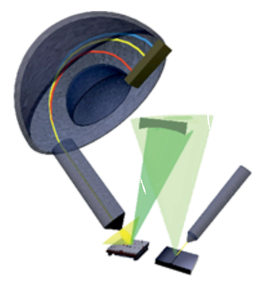


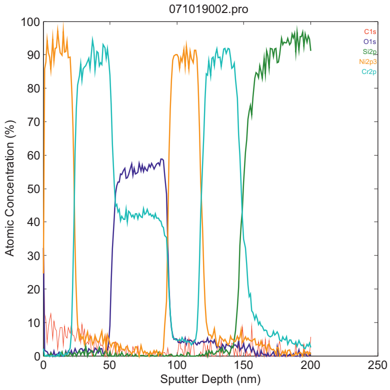
XPS DEPTH PROFILE

Destructive use of XPS, measuring the contents of thin layer surfaces;
DEPTH PROFILING

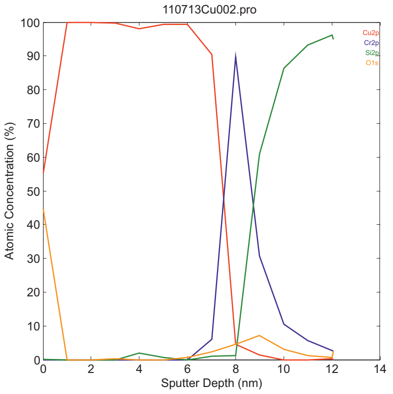
If thin films are made of compound material, it is often the question as to how the elements are distributed as a function of the depth. If the surface is allowed to be destroyed during the measurements and the chemistry is not very important, XPS can give a part of the answer.

An argon source with a controlled energy of between 100 V and 5000 V, a steady ion current and a controllable sputter area of between 1x1 and 3x3 mm^2 is used to remove material. The speed that can be achieved with this source is between 0.1 and 200 nm/minute. During an acquisition the profile can be split up into sections to obtain more precise information at one depth rather than at other depths.

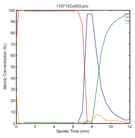
The depth resolution at the start needs not be much better than 3 nm. The information depth of a 45° exit angle measurement is around this size. If the sputtering continues, the information depth will increase to higher values and the resolution decreases due to surface roughening.



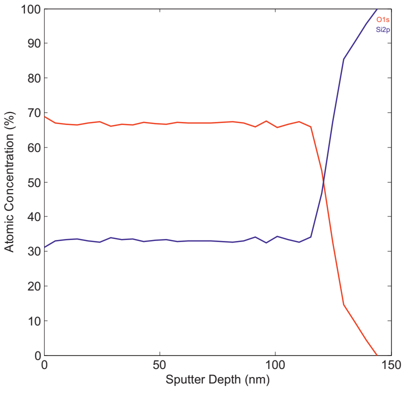
A typical sputter profile is taken on the reference sample consisting of $Ni/Cr/Cr_2O_3/Ni/Cr/Si$ where the first 5 layers have an approximate size of 30 nm each. These kinds of layers can be studied quite reliably. If the layer thickness grows towards the micrometer regime the interfaces become wider and wider. However it still holds that a line of constant height indicates a constant atomic concentration of an element. The noise indicates the precision of the contents which is around 3 at.% for these middle atomic number of mass elements.



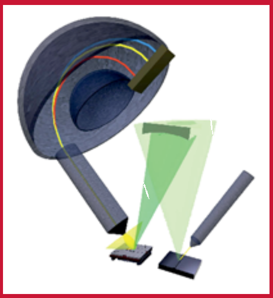
A thin layer of copper placed on silicon with a thin chromium layer in between can be studied. The copper is oxidized at the surface and the silicon substrate seems to have a small oxygen content at its surface too.



A repeated measurement with a better depth resolution shows that the first measurements conclusions agree. Two surfaces show an excess of oxygen.



A depth profile showing a 100 nm thick SiO_2 layer on silicon. The interface has a width of approximately 10 nm.

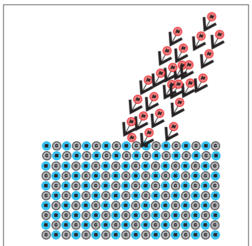


XPS DEPTH PROFILE

Areas of concern:

- Sputter speed is element dependent.

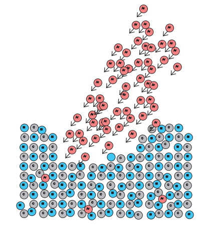
Argon ions are used in order to remove atoms from the surface under study. Standard mechanics show that atoms with atomic mass close to the argon atomic mass will be sputtered with higher speed than atoms with very different masses. Carbon has a low sputter speed compared to potassium and calcium.



Undisturbed crystal lattice

- Preferential sputtering of elements in a compound.

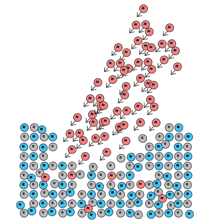
This is in fact a result of the previous point. In a compound material like SiC, the silicon is more rapidly removed than the carbon. This can result in a measurement of the C / Si ratio to be 3:1 in stead of the expected 1:1. In the nanolab cleanroom we work a lot with Si_3N_4 but if we sputter this material, the Si:N ratio will definitely not be 3:4 as expected.



Atoms are removed and roughness increases

- Creation of a damaged layer

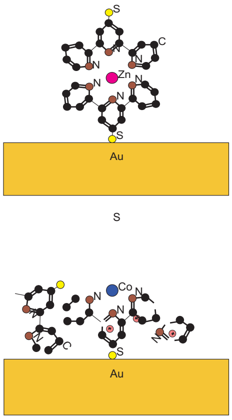
In the depth profiling upon sputtering we not only remove material from the surface, we also displace atoms in the surface, create empty spaces and implant argon in the top layer. The damaged part of the surface can be a few nanometers thick depending on the surface material, the density and energy of the argon ions . One must remember that XPS only gets its information from a layer of about 3 nm. The information can come from a completely damaged surface.



More silicon is removed than carbon, interstitial argon gets implanted

- Destruction of the chemical bonds

Removal of the so-called advantageous carbon, which in fact a small contamination layer, on top of any surface, will result in the destruction of the chemistry of the organic layer under study. Specific bonds like the C-O, C=O and COOH become invisible. The carbon C1s spectrum broadens and looses its details.



A monolayer will be severely damaged during sputtering with a standard argon ion source. Bonds will be broken, but material will still be there. The elemental content ratios could be the same as before sputtering.