LEIS

Low Energy Ion Scattering (LEIS; also known as Ion Scattering Spectroscopy (ISS)) is an analytical method for the chemical characterisation of solid surfaces. It yields the atomic composition of the outermost atomic layer and can be used on conductive and insulating surfaces. The detection limit depends on the element: for heavy elements it is in the ppm range while for light elements it leis in the order of a few %. LEIS is the most surface sensitive analytical technique.

LEIS is based on simple principles: the laws of mechanics. The surface under investigation is targeted by light noble gas ions (often He^+). When such an ion collides with a surface atom, momentum and energy are transferred, depending on the mass of the surface atom and the collision angle (see fig. 1). The light ion is scattered backwards with a high energy after a collision with a heavy surface atom and with a low energy after a collision with a light atom. By measuring the energy of the backscattered ions in an energy spectrum while keeping the angle fixed (see fig. 2) the mass of the surface atoms can be determined and this leads to the elucidation of the atomic composition of the surface.

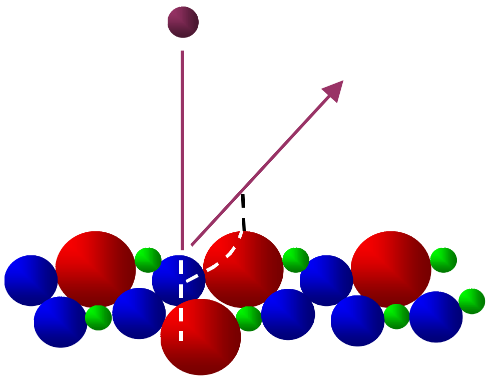

Figure 1: The principle behind LEIS: The primary ion collides with a surface atom and is scattered back. The energy of the backscattered ion depends on the mass of the surface atom.

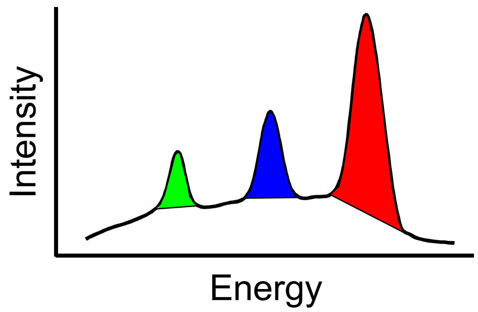

Figure 2: LEIS spectrum for the experiment in fig. 1. The energy after a collision with a heavy atom (red) is high whereas the energy after a collision with a light atom (green) is low.

The use of noble gas ions is the reason why LEIS is so surface sensitive. When these ions are penetrating the material they are neutralised and cannot be detected anymore. Because of its extreme surface sensitivity LEIS is used in catalysis, coatings, ALD growth, study of diffusion barriers, fibres, fuel cells, implants, etc. Figure 3 shows an example of an application of LEIS in catalysis. It shows how platinum in an automotive exhaust catalyst is covered with coke during use. This deteriorates the performance of the catalyst. The coke can be removed in a regeneration step and the platinum is available for the reaction.

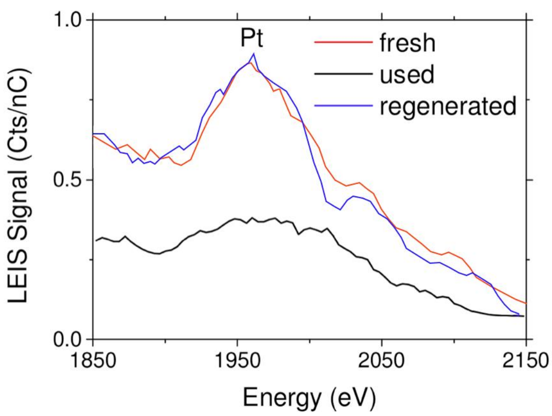

Figure 3: The platinum peak in the LEIS spectrum of an exhaust catalyst. The surface of the fresh catalyst (—) contains Pt. The Pt on the used catalyst (—) is partially covered by coke which leads to deactivation of the catalyst. After removal of the coke the catalyst is active again and the Pt can be seen at the surface (—).

LEIS doesn't suffer from matrix effects as long as the experimental conditions are chosen with care. This means that LEIS results can be quantified easily by comparing with a reference sample with a known surface composition. In some cases reference samples aren't even needed. This is particularly true for larger sample sets with few elements. When the intensities for the different elements are plotted against each other they form a straight line that can be used to calibrate the set of samples.

Apart from giving the composition of the outermost atomic layer LEIS also yields information about deeper layers, up to about 10 nm. As mentioned before, the noble gas ions are neutralised when they penetrate the material. But after that they can still collide with an atom in a deeper layer. On the path to the atom and on the way back to the surface this noble gas atom loses energy: the longer the path, the more energy is lost. A small fraction of these noble gas atoms is reionised at the surface and can therefore still be detected. Because of the energy loss they show up in the spectrum as a background on the low energy side of the surface peak. Figure 4 shows an example of barium diffusion into a polymer (poly(phenylenevinylene), PPV).

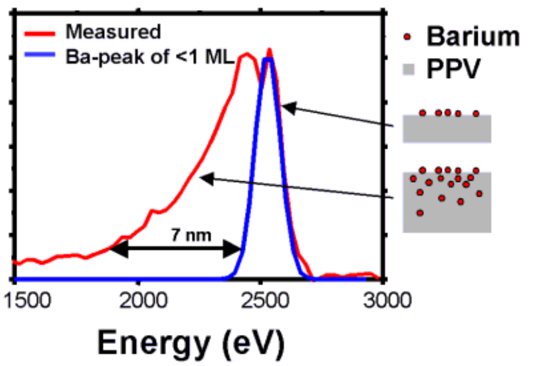

Figure 4: Barium LEIS signal for PPV with barium (—) compared to a LEIS signal for barium that is only present in the outermost atomic layer (—). The LEIS spectrum shows that the barium has diffused approximately 7 nm into the polymer.

This so called static depth profiling is often used for the study of diffusion or the growth of thin layers, e.g. for the semiconductor industry. Figure 5 shows an example where LEIS was used to study an ALD (Atomic Layer Deposition) process. In ALD a layer (in this case tungsten on silicon) is deposited on a surface in cycles. Figure 5 shows that with an increase in the number of ALD cycles the height of the tungsten peak increases while the height of the silicon peak decreases. This shows that the tungsten layer is closing continuously. At the same time the background on the left side of the tungsten peak is increasing which indicates how the layer is getting thicker.

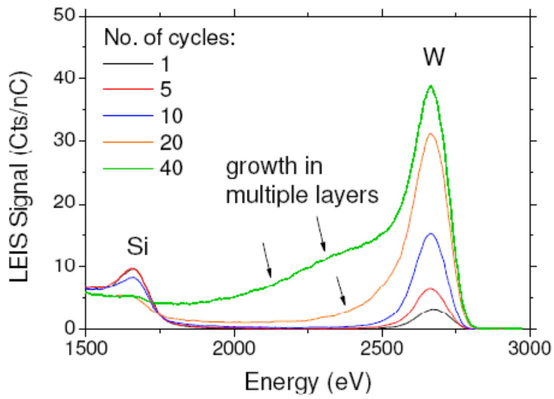

Figure 5: The growth of a tungsten containing layer on silicon followed with LEIS. With an increasing number of cycles the layer is closing (de silicon surface peak decreases and the tungsten surface peak increases). At the same time the tungsten containing layer is getting thicker (the background left of the tungsten peak is expanding towards the left).

LEIS is used to determine the surface concentration and layer thickness as a function of the number of ALD cycles. These data are important in the formulation of kinetic models that can predict ALD growth under different conditions. These models can then be used to optimise the ALD processes.