

Design and Construction of an Ultra High Vacuum Integral Low Energy Electron Mössbauer Spectrometer for Surface Analysis

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Mössbauer spectroscopy is regarded generally a bulk technique. However, it is known from many years [1] that it can be converted into a surface sensitive technique by means of the detection of the conversion electrons emitted subsequently to the nuclear de-excitation that follows the nuclear resonant absorption. Both the theoretical and experimental aspects of this variant of the technique (known as CEMS, Conversion Mössbauer Electron Spectroscopy) are well established and it has been successfully applied to the study of many different problems. One of the limitations of CEMS from the point of view of the surface analyst is that its surface sensitivity is lower than that of other standard analytical techniques such as XPS or AES. While the depth probe in these latter techniques is around 3-5 nm, the depth probe in CEMS is around 300 nm. This is related to the much higher energy of the conversion electrons (7.3 keV in the case of ^{57}Fe), which, obviously, can travel much longer distances in the solid.

After the emission of the conversion electrons a complex reorganisation of the different atomic shells takes place bringing about the emission of Auger electrons of different energies, fluorescent X-rays and shake-off electrons. Monte Carlo calculations show that, in iron, every single Mössbauer event is accompanied by 6.5 ionizations. From those, 1.5 correspond to resonant electrons of high energy (K and L conversion electrons and KLL, KLM y KMM Auger electrons) and 5.0 to LMM, MMM and MMN Auger electrons with energies lower than 100 eV and shake-off electrons with energies lower than 15 eV. It has been suggested [2] that the surface sensitivity of Mössbauer spectroscopy could be enhanced by detecting these low energy electrons since it is thought that they represent more than 50% of the total emitted electrons and their range in the solid is within a few nanometers. This variant of Mössbauer spectroscopy is known as ILEEMS (Integral Low Energy Electron Mössbauer Spectroscopy).

From the experimental point of view, ILEEMS is a technique which needs to be operated at least under high vacuum conditions using a channeltron as electron detector. Since most of the channeltrons have a low efficiency for the detection of low energy (<1 keV) electrons, a positive bias has to be applied to the cone entrance of the channeltron in order to accelerate the less energetic electrons increasing in that way the efficiency in their detection.

We report here on the design and construction of an ultra high vacuum ILEEM spectrometer aimed at the chemical, structural and magnetic characterization of iron-containing surfaces and nanostructures. The design allows the operation of the spectrometer as a stand alone device for the characterisation of samples produced ex-situ or as an accessory instrument coupled to an UHV chamber for the study of samples produced in UHV conditions.

We have carried out experiments to optimize the operational conditions of the spectrometer and we are currently involved in the study of carefully prepared samples with a well-defined layer structure to quantify the surface sensitivity of the technique. The results are compared with those obtained from XPS.

References:

- [1] J.R. Gancedo, M. Gracia y J.F. Marco, *Hyperfine Interact.* **66** (1991) 83.
[2] E. De Grave, R.E. Vandenberghe y C. Dauwe, *Hyperfine Interactions* **161** (2005) 147.

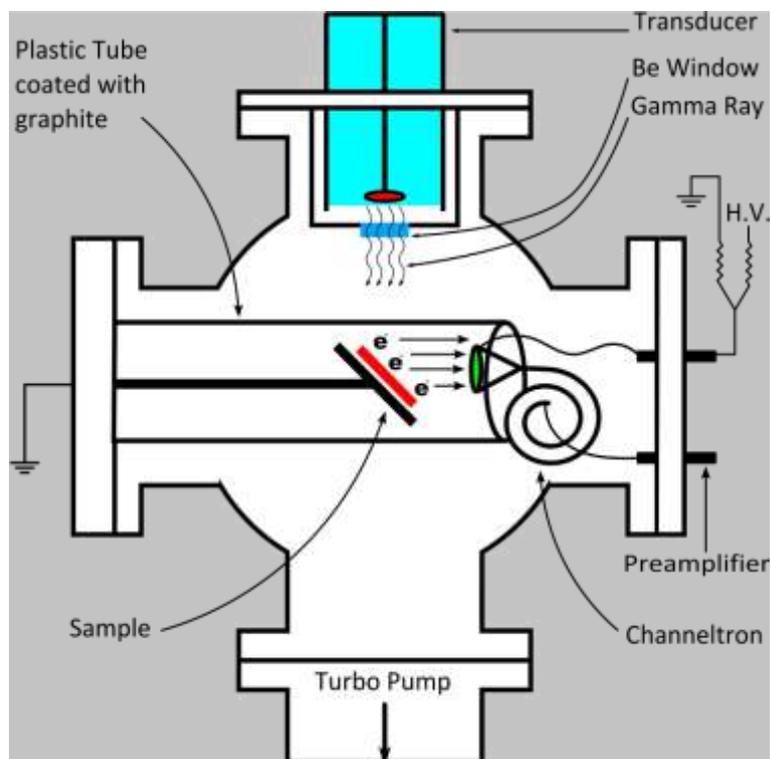
Figure:

Fig.1 Schematic drawing of ILEEMS system.