Integral Low-Energy Electron Mössbauer Spectroscopy (ILEEMS): Methodology and Recent Applications in Material Research

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ILEEMS is a variant of Mössbauer spectroscopy (MS) in which predominantly the low-energy electrons, $E < \sim 10$ eV, emitted by the probe nuclei in the absorber are counted as a function of source velocity. These low-energy electrons are, among others, Auger and "shake-off" electrons that are created in the decay process of the probe nucleus immediately after resonant absorption and excitation by an incident γ-quantum. As a consequence of their low energy, these electrons' origin lies within a very thin surface layer with thickness of an estimated five nanometers. ILEEMS using ^{57}Fe is as sensitive as transmission MS and the more common emission variants such as CEMS. Consequently, ILEEMS, in combination with conventional transmission MS, is a technique that provides information about the surface of Fe-containing substances.

In a first part of this contribution, we describe the design of a home-made ILEEMS instrument allowing the temperature of the investigated sample to be varied continuously between 77 K and room temperature. In essence, the instrument is the same as a spectrometer for transmission, except that detector and "absorber" are contained within a same vacuum (~ 5.10^{-5} mbar) chamber. The electrons are counted by a single channel electron multiplier ("channeltron"). The efficiency for detecting the low-energy electrons is optimized by applying a bias voltage of ~ 150 V between the sample and the detector input. The ILEEMS instrument has been successfully applied in a few earlier studies reported by the present authors.

The second part of this contribution will deal with a selection of results obtained recently. In particular, the following items will be covered:

- Thin films of hematite, α-Fe_2O_3, grown by RF sputtering on glass substrates in Ar plasma; ILEEMS measurements, carried out in the temperature range between 80 to 330 K for a number of α-Fe_2O_3 films with different thickness, will be presented and discussed; this research is focussed on the behaviour of the Morin transition, *i.e.*, the reorientation of the Fe^{3+} spins from the [111] crystallographic direction at low temperature, to the (111) basal plane at high temperature; the influence of thickness, sputtering parameters and of post-deposition annealing is being examined.
- Carbon nanotubes grown by CCVD method in various Fe-containing oxide matrices; the results have shown that the characteristics of the top surface layers as far as the presence of α-Fe, γ-Fe-C and Fe_3C nanoparticles is concerned, are very often significantly different as compared to what is observed for the bulk by transmission MS.
- Freshly synthesized 2 XRD-line and 6 XRD-line ferrihydrite, $5Fe_2O_3{\cdot}9H_20$, to compare the results with those obtained earlier for similar samples that had aged for a very long time.

Interesting findings concerning the surface properties of mentioned systems were obtained. ILEEMS studies so far have remained of a rather exploring nature, but the results encourage more systematic research in this and related fields.