ESRF

Experiment title:

Depth selective phase analysis of corroded ^{57}Fe films by SMR (Synchrotron Mossbauer Reflectometry): Demonstration of the feasibility of a new method

Experiment number:

SI-282

Beamline:

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Local contact(s):

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Report:

Samples of a 20 nm thick ^{57}Fe film grown onto float glass substrate were exposed to annealing in air at various temperatures up to 285 °C for 4 hours. Grazing incidence prompt and delayed time integral Θ–2Θ scans as well as time spectra at selected angles ($0.03 \text{ deg} \leq \Theta \leq 0.41 \text{ deg}$) corresponding to the penetration depth of 1.3 nm to 62 nm were measured at the ID18 nuclear resonance beamline (E = 14.413 keV, λ = 0.0860 nm) in an external magnetic field B_{ext} = 0.37 T) perpendicular to the scattering plane.

The prompt x-ray reflectivity curves show damped Kiessig oscillations corresponding to the total film thickness that is increasing with increasing degree of oxidisation. The delayed nuclear scattering, in contrast, reveals the depth profile of the hyperfine interaction characteristic of the chemical and magnetic state of the sublayers within the thin film. The quantum beat patterns (examples in Fig. 1.) from the uppermost few nanometers show the presence of non-magnetic or superparamagnetic phases (probably β–FeOOH and superparamagnetic α–Fe_2O_3) in case of all but the most strongly oxidised sample (Fig. 1, right side) the whole cross-section of which is magnetic. Time spectra from deeper regions of the film show different magnetic interactions stemming from a-Fe and from various iron oxides (Fe_3O_4 and antiferromagnetic α–Fe_2O_3). Detailed analysis of the spectra (simultaneous fitting of all time spectra measured on the same sample at different angles in terms of a model consisting of different iron, oxide and oxyhydroxide layers) is in progress.

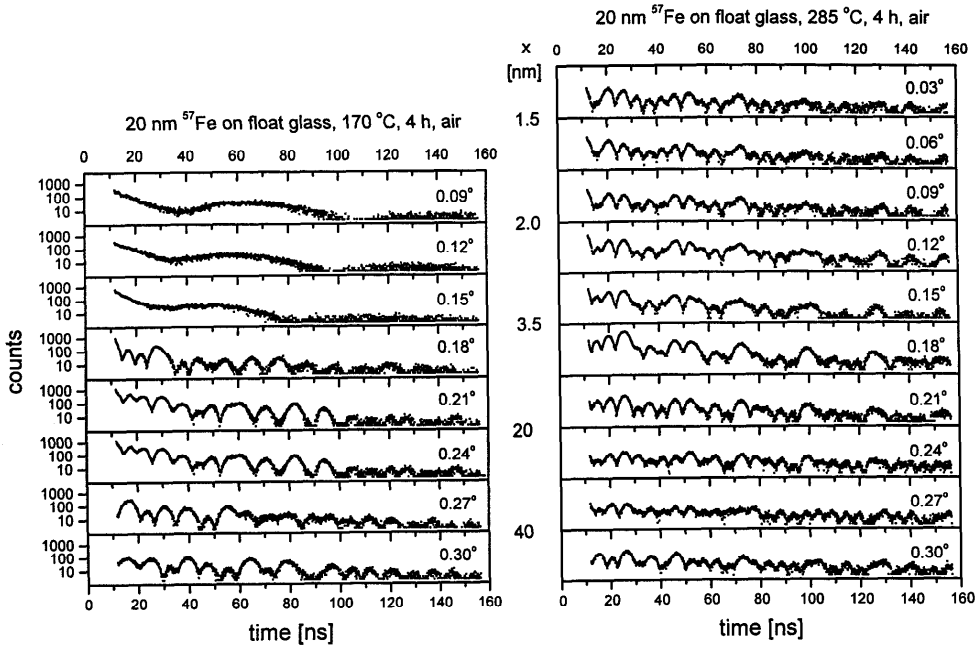

Fig 1. SMR time spectra of a 20 nm thick ^{57}Fe film grown onto float glass substrate and exposed to annealing in air at 170 °C (left) and 285 °C (right) for 4 hours measured at various angles of grazing incidence. The numbers between the two ladders approximately show the respective penetration depth x of the 14.4 keV photons. The uppermost ≈ 4 nanometers of the less oxidised sample (left side) is non-magnetic the rest consisting mainly of Fe_3O_4. The strongly oxidised sample (right side) is almost pure α–Fe_2O_3.