ESRF	**Experiment title:** Evolution of the magnetic structure during growth of native Fe oxides	**Experiment number:** SI 1259
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Report:

The aim of the experiment was the determination of the magnetic structure of buried native oxides at different stages of their formation. For that purpose a dedicated transportable UHV chamber was built and installed at the beamline. The chamber is equipped with two magnetron sputter sources and an ellipsometer for in-situ and on-line control of the preparation. Since nuclear resonant scattering (NRS) is an isotope selective method and has a pronounced sensitivity to the orientation of the magnetic hyperfine fields, this experimental method was chosen. A permanent magnetic field of about 70 mT is acting on the samples during preparation and measurement. As a substrate we used superpolished Si wafers, which in part have been covered with a palladium layer of about 200 Å to increase the signal by using the effect of standing waves. The samples investigated here were produced by deposition of a thin layer of natural iron (~ 32 Å) followed by a very thin ^{57}Fe layer (~ 6 Å). Subsequent oxidation at room temperature in an O_2 atmosphere led to a 10 Å thick layer of native ^{57}Fe oxide. To analyze the evolution of the magnetic structure of the buried native oxide, the thickness of the covering natural iron layer was increased stepwise. After each step the sample was analyzed in-situ by recording a NRS time spectrum. A selected set of time spectra is shown in Fig. 1. The solid lines in the time spectra are theoretical simulations based on the hyperfine field distribution shown in the second column of Fig. 1. With increasing Fe coverage the magnetic properties of the native oxide changes dramatically. For the pure oxide phase an rather wide distribution of the hyperfine field around $B_h = 4.3$ T has been found. This can be assigned to a magnetically frustrated phase or superparamagnetic α-Fe_2O_3.

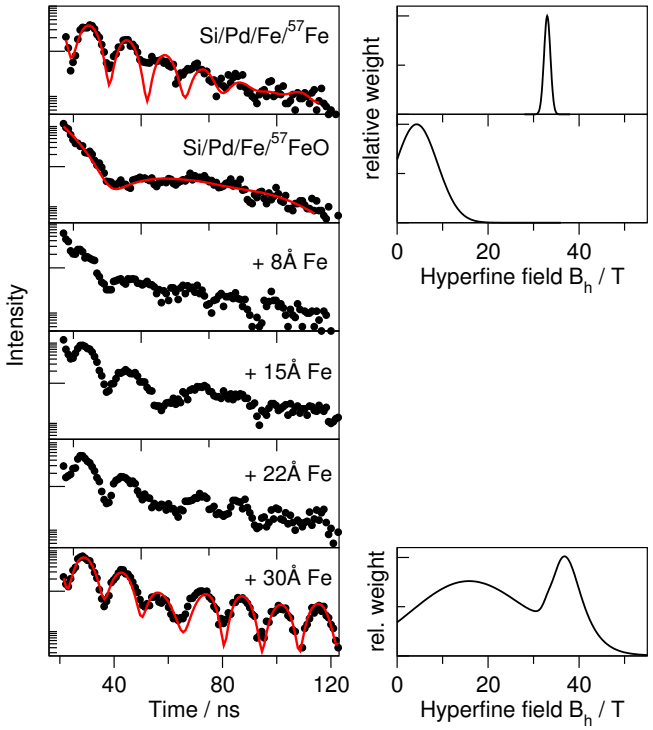

Fig. 1: Time spectra for grazing incidence reflection of Fe/FeO layers of the given thicknesses. Assumed hyperfine field distribution (right).

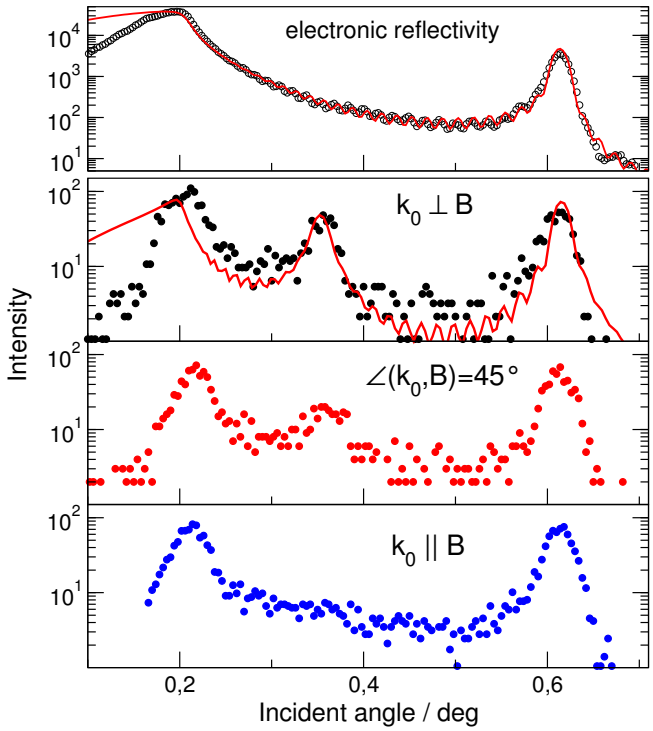

Fig. 2: Reflectivity curves taken at 14.4 keV of a Fe/FeO multilayer showing antiferromagnetic order of the layers. Solid lines are simulations.

Upon covering the oxide layer with iron the magnetic structure changes gradually to a phase with different components. According to the simulation the high field components are ferromagnetically aligned with the Fe metal. The simulation of the intermediate steps is especially difficult because of additional changes in the isomer shift and quadrupole splitting. It should be noted that the influence of the ^{57}Fe in the natural Fe is not negligible. To obtain unambiguous results it would be highly desirable to further reduce the amount of metallic ^{57}Fe by using ^{56}Fe for the metallic layers.

Since the motivation for our experiment has been a work on Fe/FeO multilayers [1] such a multilayer has been prepared and analyzed. The sample has the structure: Si/[Fe/^{57}FeO]$_{30}$/Fe and was measured in an external magnetic field of 70 mT. The angular dependence of the nuclear and electronic reflectivity is shown in Fig. 2. The chemical periodicity of the multilayer with a period of 43 Å results in the first-order electronic Bragg reflection. The same peak is also seen in the nuclear reflectivity. However, the nuclear reflectivity shows also a peak at the position of the half-order reflection, which correspond to a period of 86 Å. This is the period of a magnetic superstructure that most probably results from an antiferromagnetic (AFM) coupling of neighbouring iron oxide layers [2]. The AFM peak is most prominent if the angle between the incident photon beam and the external magnetic field is 90°. Changing this angle to 45° reduces the peak to about half its intensity and for 0° the AFM peak has vanished. From that we conclude that the coupling angle between the metallic iron and that component in the oxide, which is responsible for the AFM coupling is about 90°. In upcoming experiments we want to analyze the origin of this phenomenon in more detail.

[1] G. Beach et al., Phys. Rev. Lett. 91, 267201 (2003).
[2] Th. Diederich, R. Röhlsberger et al., in preparation for Phys. Rev. Lett.