



Experiment title: Investigation of interdiffusion processes at an Fe/FeO interface	Experiment number: SI 1614	
Beamline: ID18	Date of experiment: from: 09.09.2008 to: 17.09.2008	Date of report: 16.01.2009
Shifts: 24	Local contact(s): S. Stankov	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): S. Couet*, HASYLAB at DESY, Notkestr. 85, D-22607 Hamburg, Germany K. Schlage*, HASYLAB at DESY, Notkestr. 85, D-22607 Hamburg, Germany R. Röhlberger*, HASYLAB at DESY, Notkestr. 85, D-22607 Hamburg, Germany		

Report:

The aim of the experiment was to study in-situ the interdiffusion process occurring at a Fe/FeO interface with highest spatial resolution. For that purpose, we use the isotope sensitive technique of nuclear resonant scattering (NRS) of synchrotron radiation on a $^{56}\text{Fe}(4\text{ nm})/^{57}\text{Fe-O}(1\text{ nm})/^{56}\text{Fe}(4\text{ nm})$ trilayer. This technique allows us to monitor, as a function of temperature, magnetic and chemical changes in the ^{57}Fe layer by studying the time-differential de-excitation of the ^{57}Fe nucleus. To increase the depth sensitivity, the system was placed between two Ta layers, effectively creating a x-ray waveguide structure. This way, the nuclear signal was increased by more than one order of magnitude, effectively allowing in-situ experiments. At the same time x-ray and nuclear reflectivity (the time-integrated nuclear signal) were recorded. The first allows to follow the diffusion of the oxygen atoms by monitoring the density depth profile while the second, uniquely sensitive to ^{57}Fe , allows to monitor the diffusion of the ^{57}Fe . It should be noted that the waveguide nature of the system increases the depth sensitivity of the NRS signal to the position at the probe layer. By recording the three datasets (electronic + nuclear reflectivity and timespectra), one is able to monitor the diffusion and the induced structural changes in-situ.

The sample was placed in a vacuum oven equipped with x-ray windows for grazing incidence geometry experiments. First, the temperature was raised stepwise to find the exact temperature at which diffusion processes start to be visible. Selected reflectivity and timespectra are shown in Fig.1. No changes are observed in the electronic and nuclear reflectivity curve up to 530 K, meaning that the ^{57}Fe does not diffuse, and that the multilayered structure is preserved.

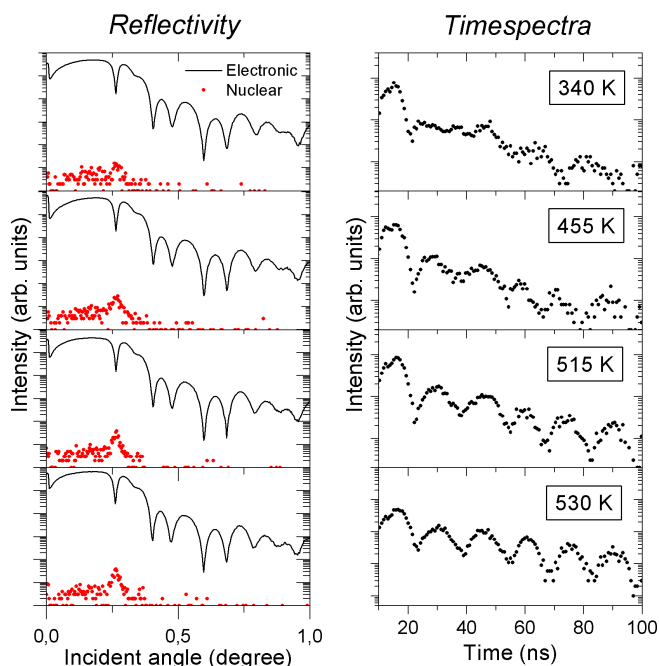


Fig. 1: Temperature dependent NRS experiment on a Fe/ ^{57}Fe -oxide/Fe trilayer. (left) Electronic and Nuclear reflectivity curves recorded at various temperature. (right) Corresponding time-spectra showing a transition from oxide to metal for the probe layer.

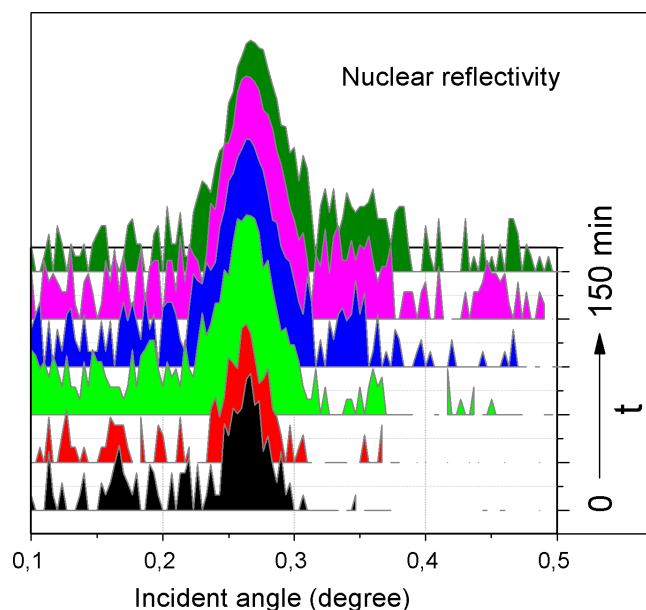


Fig. 2: Evolution of the Nuclear reflectivity curve with time at the constant temperature of 530 K. The progressive appearance of kiessig fringes indicates the progressive diffusion of the ^{57}Fe layer in the whole metal layer.

However, drastic changes are seen in the timespectra at each steps. While at low temperature they can be attributed to change in the magnetic structure of the oxide, the spectrum recorded at 530 K shows the typical beating of ferromagnetic metallic Fe. Since we know from the nuclear reflectivity measurements that the ^{57}Fe did not diffuse, the transformation of the ^{57}Fe -oxide to metallic Fe can only mean that the oxygen alone has diffused to the edges of the trilayer.

After this measurement series, we stayed at 530 K and recorded stepwise reflectivity and timespectra. No changes are observed in the timespectra anymore but we observe a slow change in the nuclear reflectivity. Subsequent nuclear reflectivity curves, covering a timespan of 150 minutes, are shown on Fig.2. At the later times, kiessig fringes are distinguishable at higher angles. Although the data analysis is still in progress, this feature is a sign of the progressive diffusion of the central ^{57}Fe layer in Fe.

Afterwards, we performed similar experiments with the ^{57}Fe placed at different positions in the metal part of the sample, still with the oxide nanolayer in the central position (an ^{56}Fe -oxide layer). The hope was to detect in the timespectra the eventual formation of an oxide at the Ta/Fe interface. Those experiments were unsuccessful, as the recorded timespectra show pure metallic behavior throughout the temperature range.

Overall, the information gained is quite important. We have seen that oxygen diffusion in Fe starts to occur at the relatively low temperature of 515 K (240°C). Knowing the potential importance of iron oxide interfaces in contact with metals in the upcoming field of spin-electronics, one should eventually take these information into consideration for practical applications.