ESRF	**Experiment title:** Magnetic interparticle coupling of ^{57}Fe nanoparticles in a polymer waveguide matrix	**Experiment number**: MA-196
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Shifts: 18	**Local contact(s)**: Alexandr Chumakov, Rudolf Rüffer	*Received at ESRF:*

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Report:

The aim of this experiment was to test if an x-ray thin film waveguide structure can be applied to enhance the nuclear resonant scattering signal from smallest amounts of ^{57}Fe Moessbauer atoms to characterize the thermal magnetic stability of ^{57}Fe nanoparticles embedded in a polymer matrix. The experimental technique is time resolved coherent nuclear scattering of synchrotron radiation in grazing incidence geometry and was carried out at the nuclear resonance beamline ID18 during the 16bunch mode.

The waveguide structure consists of two sputtered Pd layers which are separated by a thin polymer layer which was prepared with the spin coating technique. Due to the low thickness of the capping Pd layer x-rays can couple in grazing incidence into waveguide modes of the polymer guiding layer. The high difference in the refractive index of this waveguide structure and the low absorption coefficient of the polymer guiding layer cause a very strong enhancement of the x-ray field intensity inside the polymer layer.

In the first part of the beamtime we focused on the investigation of the quality of the Pd/polymer/Pd waveguide structure or rather on the enhancement factor of the nuclear resonant scattering signal from tiniest amounts of ^{57}Fe atoms embedded in the polymer due to the waveguide effect. For this we prepared a waveguide sample with a monolayer of ^{57}Fe in the central position of the polymer layer. As a main result from this investigation we detected a remarkable intensity of the nuclear signal of up to 2000Hz (!) in the first waveguide mode which is 2-3 orders of magnitude larger than the nuclear signal from a single ^{57}Fe monolayer directly placed onto a silicon wafer.

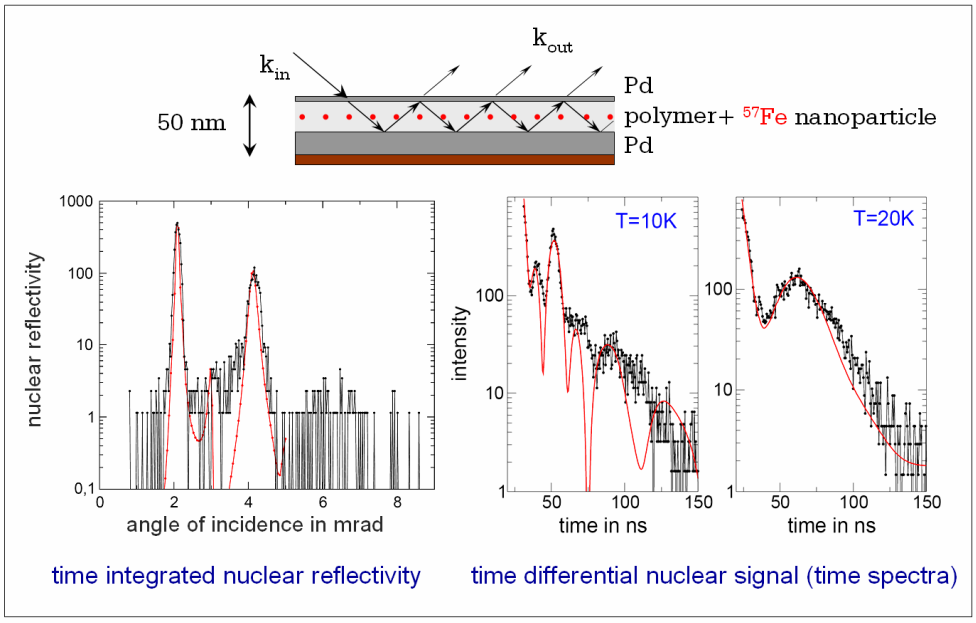

Figure 1:
Schematic drawing of the Pd/polymer/Pd waveguide structure with ^{57}Fe nanoparticles embedded in the central position of the polymer X-ray guiding layer. The nuclear (resonant) reflectivity curve of this sample (on the left side) shows two sharp peaks where created waveguide modes in the guiding layer lead to an extreme enhancement of the nuclear (magnetic) signal from the Fe nanoparticles. The time differential nuclear signal was used to investigate the magnetic stability of the particles depending on the particle diameter and on the temperature. The two time spectra on the right side show the magnetic transition from the ferromagnetic to superparamagnetic state of the particles between 10K and 20K.

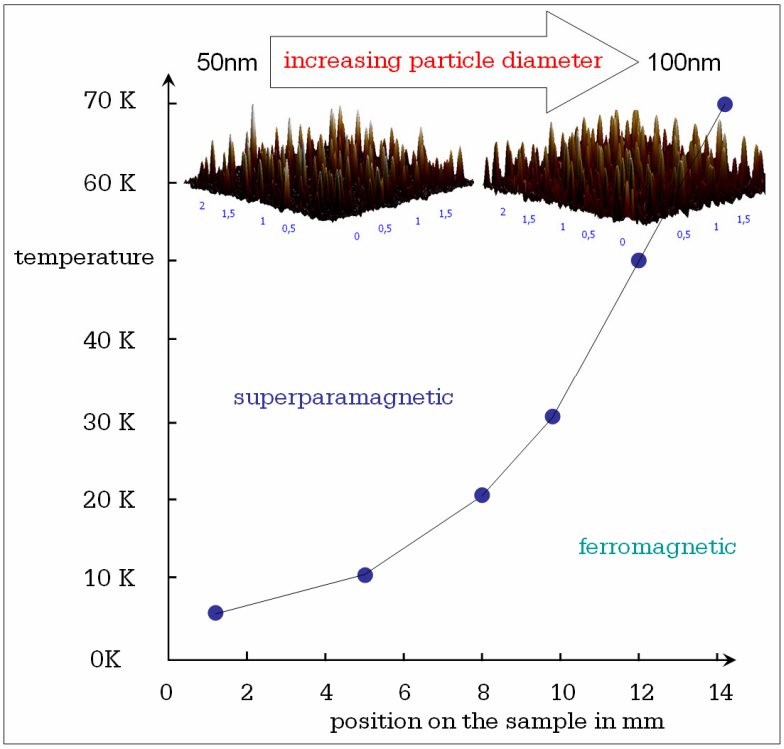

Figure 2:
Variation of the transition temperature from ferromagnetic to superparamagnetic state (blocking temperature) with increasing particle diameter. The phase diagram was determined from a waveguide sample with a lateral gradient of the diameter of the embedded particles which varies from 50nm to 100nm from the left to right side of the sample (see AFM pictures in the inset). The height of the particles remains constant at around 8nm.

This extreme boosting of the nuclear signal is caused by the strong enhancement of the normalized field intensity inside the guiding layer which was determined to around 50 from the simulation of the electronic reflectivity curve. A second reason is given by the coherent nature of the nuclear scattering process in the reflectivity channel which causes a quadratically scaling of the nuclear signal with the normalized fieldintensity at the position of the ^{57}Fe monolayer [1].

We applied this strong waveguide effect in the second part of the beamtime to investigate the thermal magnetic stability (superparamagnetic effect) of sputtered ^{57}Fe nanoparticles embedded in the central position of the polymer layer. To investigate the dependence of the blocking temperature on the particle diameter we prepared a sample with a lateral gradient of the particle diameter which varies from 50 to 100nm. The height of the partially oxidized, oblate shaped nanoparticles is around 8nm and the in plane particle coverage increases from around 2% to 12%.

To determine the temperature dependant magnetization of the ^{57}Fe particles we mounted the sample during the experiment in a He cooled cryomagnetic system. The time resolved signal of the nuclear reflectivity was used to characterize the orientation and strength of the particle magnetization at different temperatures and different lateral positions on the sample. Figure 1 shows two time spectra were the loss of the beat pattern in the temporal nuclear signal determines the thermal induced transition from the ferromagnetic to superparamagnetic state between 10K and 20K. The simulation of the data (red curves) yield an isotropic in plane magnetization of the particles below the blocking temperature.

A fascinating count rate of up to 4000Hz allowed us to determine the complete blocking temperature phase diagram of this sample which is described in Figure 2. We found a very strong increase of the magnetic transition temperature with increasing particle diameter. Whereas the iron nanoislands of 50nm get ferromagnetic only below 5K the particles with a lateral diameter of around 100nm remain ferromagnetic up to 70K. Further experiments should clarify if this unexpected strong increase of the blocking temperature is only due to an increase of the particle volume or additionally due to an interparticle coupling effect.

In conclusion, we have shown that a Pd/polymer/Pd thin film x-ray waveguide structure is an excellent tool to increase the scattering signal from smallest amounts of material placed in the polymer x-ray guiding layer. Especially for coherent scattering processes like nuclear resonant forward scattering of synchrotron radiation the waveguide effect results in an extreme boosting of the scattering signal. This waveguide effect allowed us to carry out a systematic study on the superparamagnetic effect of isolated iron nanoparticles embedded in a polymer matrix which showed an unexpected strong dependence of the blocking temperature on the particle diameter.

[1] Coherent x-ray scattering from ultrathin probe layers
R. Röhlsberger, T. Klein, K. Schlage, O. Leupold, R.Rüffer
PRB 69, 235412 (2004)