ESRF	**Experiment title:** Hard x-ray-detected electron paramagnetic resonance on Gd_2O_3 nanoparticles	**Experiment number:** HE-3293
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Shifts: 18	**Local contact(s):** A. Rogalev	*Received at ESRF:*

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Report:

The aim of this experiment was to detect electron paramagnetic resonance (EPR) using x-ray magnetic circular dichroism (XMCD) at the Gd L_3–edge on Gd_2O_3 nanoparticles. For the beamtime other Gd containing samples were brought as backup, namely 16% Gd:ZnO, Gd doped Permalloy, and $GdSO_4 \cdot 8H_2O$ pressed into a pellet. All samples exhibit a clear conventional EPR signal with linewidths ranging from 1 to 3 kOe, therefore deemed to be suitable for XEPR measurements. This experiment requires lowest possible temperatures for maxmimum possible EPR signal. The lowest achievable temperature was about 40 K and all four samples were tested in the XEPR setup to determine to maximum achievable XMCD signal size at the given experimental conditions, i.e. ~40 K and an external field around 5 kOe to decide to most suitable sample for XEPR. The resulting XMCD spectra are shown in Fig. 1 and Fig. 2 for two samples each.

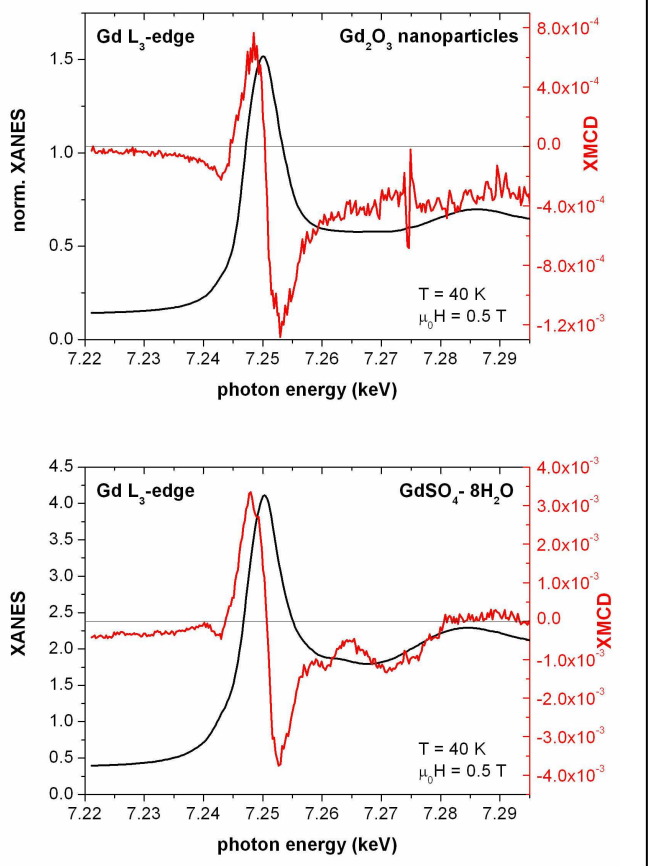

Fig 1: XMCD spectra of Gd nanoparticles and $GdSO_4 8H_2O$.

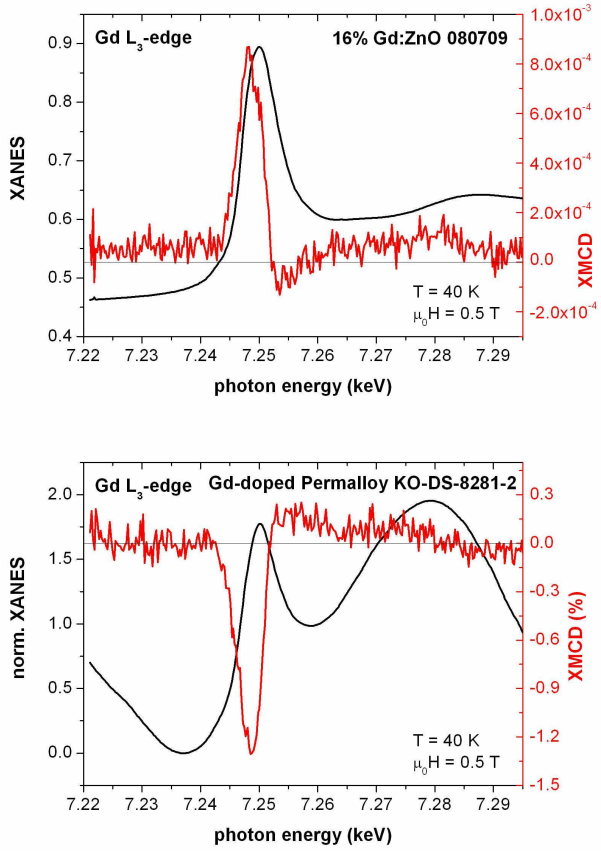

Fig 2: XMCD spectra of Gd:ZnO and Gd doped Permalloy.

From Fig. 1 it should become clear that the maximum XMCD signal for the given temperature and magnetic field is too low, i.e. of the order of 10^{-3}, to detect the small XEPR effect of the order of 10^{-6}. In addition, both samples showed signs of radiation damage after the above experiment; i.e. the color of the Gd nanoparticles and the $GdSO_4 \cdot 8H_2O$ pellet changed the latter presumably due to release of some of the crystal water. Fig. 2 shows the two other samples, which were stable against the x-rays. However, for the Gd:ZnO sample the XMCD signal was the smallest of all, where as the Gd doped Permalloy showed two other problems: (i) the Gd L_3-edge sits on top of some EXAFS wiggles of the lower lying Fe K-edge; whil the XMCD can be easily recorded, it has a strong background signal which makes the useful signal a very small fraction of the overall signal for the XMCD already; XEPR would not be possible. (ii) The sign of the XMCD of the Gd:Py compared to the Gd:ZnO suggests that the Gd moments are aligned antiparallel to the external field which drives the Py parallel; this observation is interesting by itself, however, it makes useful XEPR measurements at the Gd L_3-edge difficult.

Thus it was decided to test for another sample for the feasibility of XFMR. For that a 600 nm thick Fe_3Si layer was used and first measured conventionally in the XFMR setup; the outcome is shown in Fig. 3. The FMR line would be sufficiently narrow to measure XFMR in principle. However, despite many efforts any XFMR signal could be recorded in transversal geometry which was check at various power levels up to that high powers, that the resulting Fe K-edge XANES signal was reduced, i.e. part of the film evaporated. The impossibility to record any XFMR signal could be related to so electrical problems of the used photo diode which however could not be resolved during the beamtime itself, because there is no obvious other physical explanation as to why the XFMR of this sample could not be measured.

Since during this beamtime the XFRM setup could only be operated by the beamline staff and not many experiments with long averaging times were required, some of the overnight beamtime was used to measure some other backup samples. A few Co:ZnO layers were brought as well containing for the first time a high Co concentration of nominally 20%, i.e. above the coalescence threshold. Their XMCD signals were measured overnight, for two samples even with grazing and normal incidence, respectively, which also can yield the XLD signature by subtracting the XANES recorded with circular polarized light under grazing and normal incidence. The result is exemplarily shown for a paramagnetic 20% Co:ZnO sample and the spectroscopic features corroborate earlier findings. The same results were also recorded for increased O content in the sputtergas. These findings will be included in a future publication.

In summary, it turned out to be experimentally very difficult to record XEPR spectra because of lack of XMCD signal as well as sample damage upon x-ray irradiation. The found antiparallel alignment of Gd to Py is interesting. Further it turned out to be not possible to record XFMR on Fe_3Si, presumably due to detector problems. Some otherwise unused night-shifts were used for XMCD and XLD measurements of Co:ZnO samples to complement earlier work.

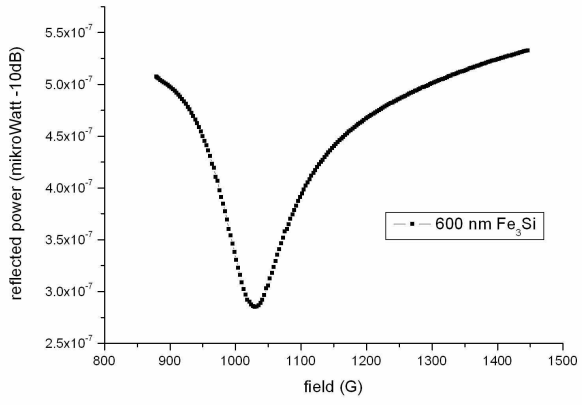

Fig. 3: Conventional FMR of Fe_3Si in the XFMR setup.

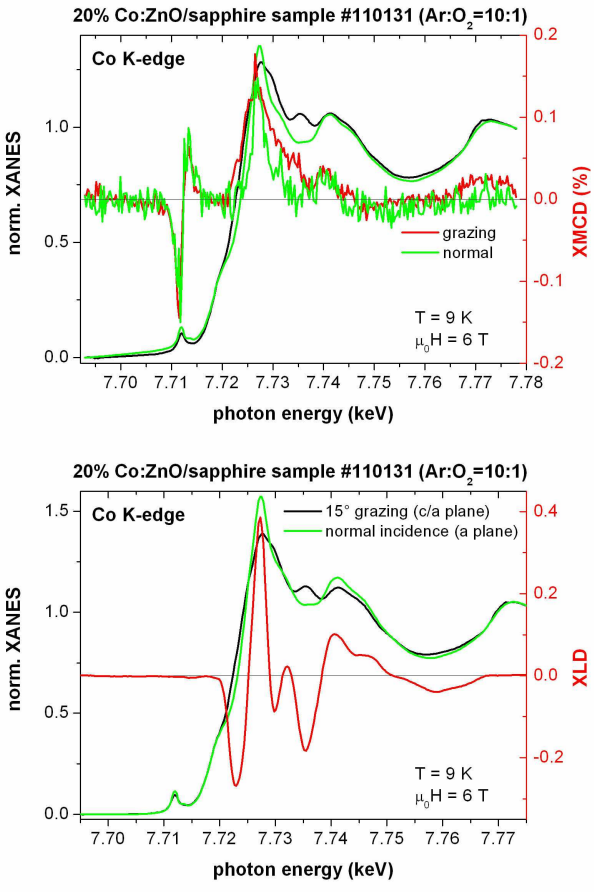

Fig 4: XMCD spectra under grazing an normal incidence repsectively. The resulting XLD is shown in addition.