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ESRF

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- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **Influence of the FeAg interface on the magnetic behaviour of FexAg100-x granular thin films above the percolation limits (x>28%) by EXAFS spectroscopy**	**Experiment number**: 25-01-0689
Beamline: BM25A	**Date of experiment**: from: November 7 to: November 11	**Date of report**: 28-11-2008
Shifts: 12	**Local contact(s)**: Iván da Silva	*Received at ESRF:*

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Report:

The study of the magnetic behaviour of assemblies of interacting nanoparticles has attracted great attention from material scientists for many years. The understanding of these interactions is important not only from a theoretical point of view, but also for various technological applications.[1] If the interparticle interactions are significant one can obtain very different collective magnetic behaviours, from an interacting superparamagnet (ISPM)[2] at low enough concentrations to a superferromagnet (SFM)[3] or a correlated

susperspin glass (CSSG)[4] at higher volume fractions. Binary Fe-Ag alloys are ideal systems to study such phenomena since Fe and Ag are highly inmiscible,[5] and so, it is viable to produce samples consisting of Fe nanoparticles embedded in a diamagnetic Ag matrix. In our case, we have focused on the study of Fe-Ag thin films with Fe concentrations above the percolation limit, which has been estimated about 28 at. % for these granular systems,[6] prepared both by pulsed laser deposition (PLD) and sputtering techniques. In this range, we have found a rich variety of collective magnetic states modulated mainly by the competition between interparticle magnetic interactions, of dipolar and direct exchange character, the intraparticle anisotropies and the nature of the Fe-Ag interface.

In this experiment we started to perform EXAFS measurements at the Fe K-edge in a series of PLD thin films. What we pretended with this analysis was to obtain information about the microstructure of our samples (spatial and size distribution of the Fe nanoparticles) and about the Fe-Ag interface. Due to the high energies reached in the PLD some kind of intermixing in the interface is possible for these samples. In any case, even collecting the data 6 times and increasing the integration time in order to obtain better statistics and to reduce the noise appearing in the spectra, we observed that the spectra were too noisy to be able to obtain some reasonable result from the EXAFS analysis, as can be clearly seen in Fig. 1. The problem is that since our thin films (100-200 nm) are deposited onto a Si substratre (~500 μm), the only available configuration for the measurements is in fluorescence and unfortunately the fluorescence detector resolution was heavily limited by the dead time, which didn´t allow us to obtain better results.

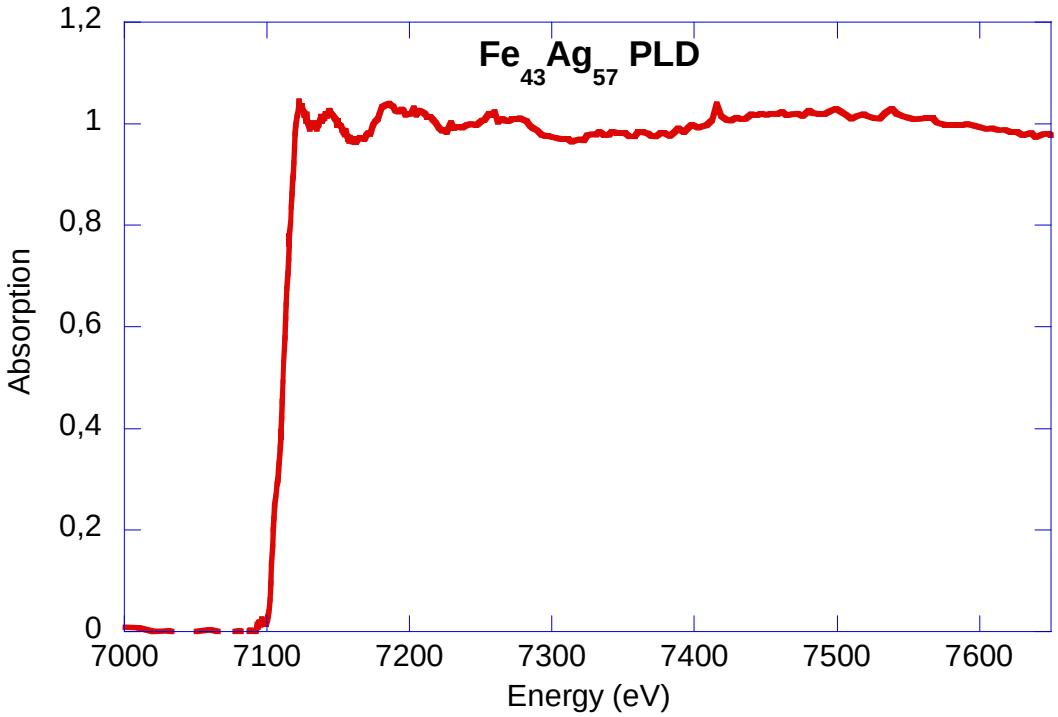

Figure 1. Fe K-edge EXAFS spectra of a $Fe_{43}Ag_{57}$ thin film deposited onto Si.

Therefore, after several unsuccessful trials to improve the quality of the spectra, we opted for changing the samples. These new samples were composed of Fe nanoparticles synthetised using different concentrations of surfactant (see Table 1), and could be easily measured in transmission, thus avoiding the previously cited problems with the fluorescence configuration. The main objective in these new measurements was to analyse the overall quality of the nanoparticles, trying to discard the presence of unwanted oxides. The data were collected at room temperature, at least 3 times in order to obtain better statistics. As can be seen in Fig. 2 and 3, the normalized XANES spectra of all the samples showed clear indication of the presence of oxides, except for the sample TXE66. This indicates that the conditions for the preparation of this sample are the best ones in order to obtain less oxide, and therefore, they have to be carefully analysed for future samples.

sample	[Surfactant] (M)	g $FeCl_2$	g $HAuCl_4$	g H_2O	T (ºC)
TXE 046	0,3	0,4	0,4	6	
TXE 052	0,3	0,4		6	
TXE 056	0,1	0,4		2	
TXE 058	0,1	0,3	0,3	3	
TXE 059	0,1	0,3	0,3	2	
TXE 061	0,1	0,3	0,5	4	
TXE 065	0,3	0,4		6	
TXE 066	0,15	0,4		6	
TXE 067	0,3	0,4		6	
TXE 068	0,3	0,4		6	30
TXE 069	0,3	0,4		6	35
TXE 070	0,3	0,4		6	40
TXE 071	0,3	0,4		6	45
TXE 072	0,2	0,4		6	

Table 1. Samples composed of Fe nanoparticles synthetised using different oncentrations of surfactant, $FeCl_2$ and H_2O. If the preparation temperature is different than room temperature, it is also stated.

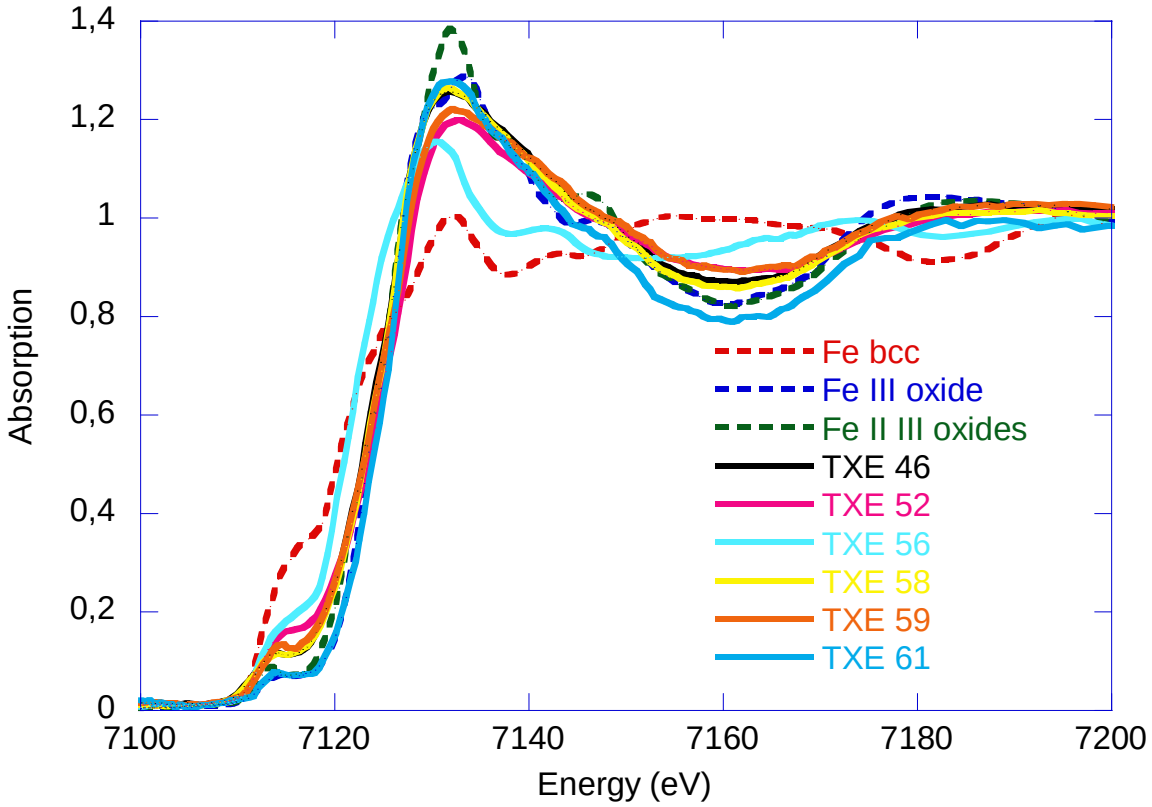

Figure 2. Fe K-edge EXAFS spectra of several samples composed of Fe nanoparticles inside an emulsion. Fe bcc and two iron oxides spectra are also included for comparison.

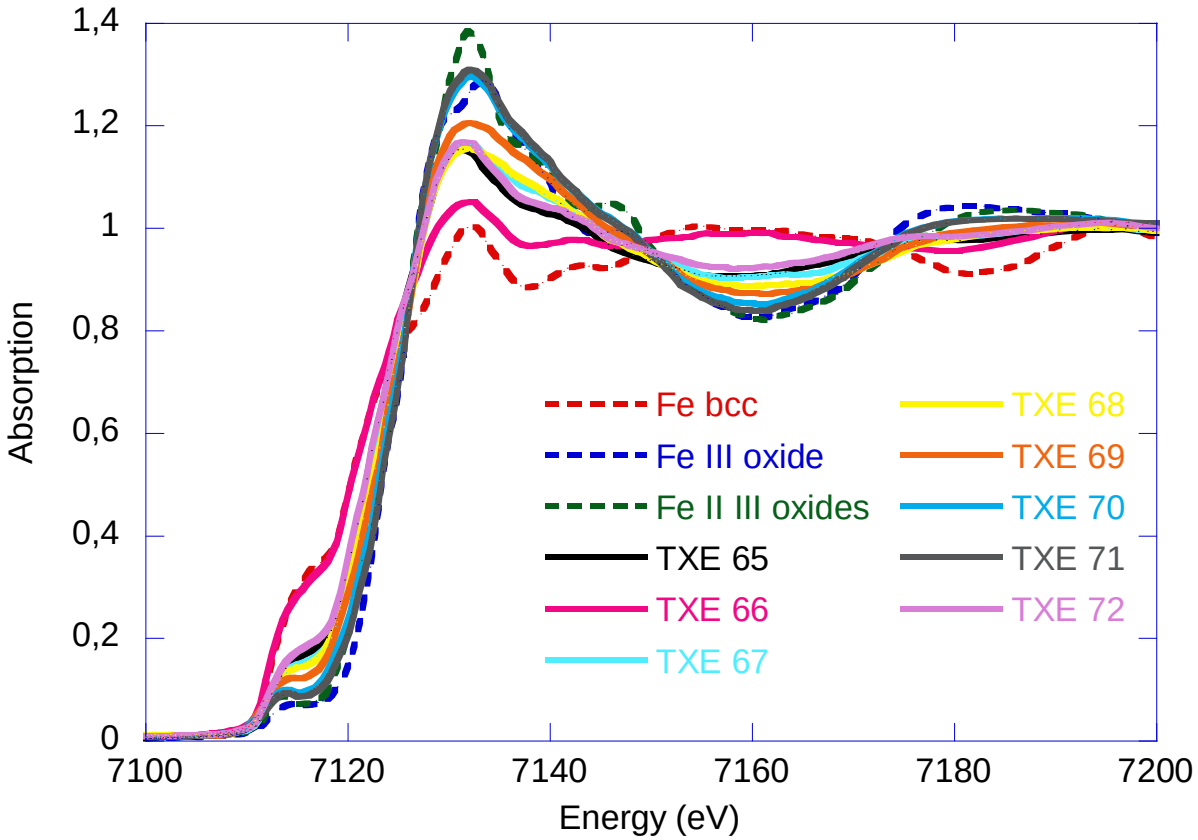

Figure 3. Fe K-edge EXAFS spectra of several samples composed of Fe nanoparticles inside an emulsion. Fe bcc and two iron oxides spectra are also included for comparison.

Finally, we would like to thank the SPline team for competent support and help all through our experiments and before and after our arrival to the ESRF, as well as the personnel at the facility for their assist in all the aspects of their competence.

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