ESRF	**Experiment title: Analysis of the influence of the microstructure and the intergrain region on the magnetic behaviour of FeAg granular thin films by EXAFS spectroscopy**	**Experiment number**: 25-01-750
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Report:

Two different systems have been measured in this experiment. Firstly, FeAg nanogranular thin films, and secondly, Ni-doped Fe_3O_4 nanoparticles.

Room temperature EXAFS on Fe_xAg_{100-x} granular thin films (x= 20, 39, 50) has been performed in fluorescence configuration on the Fe-K edge. The aim of this experiment was to correlate the observed changes in the magnetic behavior with changes in the microstructure, especially in the intergrain region. The obtained EXAFS signals and their Fourier transforms are shown in figure 1 for the 3 samples together with the Fe bcc foil.

The data has been fitted up to the fourth shell using the FEFFIT package. Results of the interatomic distances and Debye-Waller factors are displayed in table 1. Only the values corresponding to the first shell are shown because the interatomic distances for the other 3 shells have been restrained to maintain the geometric relation corresponding to a bcc lattice, and the Debye-Waller factors of the shells are related by the correlated Debye model. As can be seen, the fact that the interatomic distances remain nearly unaltered across the composition range ($20 < x < 50$), is consistent with a nanostructure of Fe nanoparticles embedded in the Ag matrix. In comparison with the sputtered bcc Fe film, there is a slight increase of the interatomic distances (~0.5 %), which can be related to the atoms in the surface of the nanoparticles, since due to the small size of the Fe nanoparticles (3-6 nm) their proportion is very important, and are affected by the presence of nearby Ag nanoparticles (with higher lattice size). Although we cannot completely rule out the presence of certain alloy formation at the surface of the Fe nanoparticles, these measurements indicate that its effect is not important enough to modify the predominant bcc structure of the Fe. Moreover, from the quality of the fitting to a bcc Fe structure, we can conclude that the proportion of subnanometer clusters diluted in the Ag matrix, which, if present, could affect the magnetic interactions between the nanoparticles, is not appreciable.

However, no clear changes have been observed in the evolution of these parameters with the Fe concentration, thereby indicating that the observed magnetic changes have no direct correspondence with the microstructure and are more related to the exchange correlation lengths which depend mainly on the interactions.

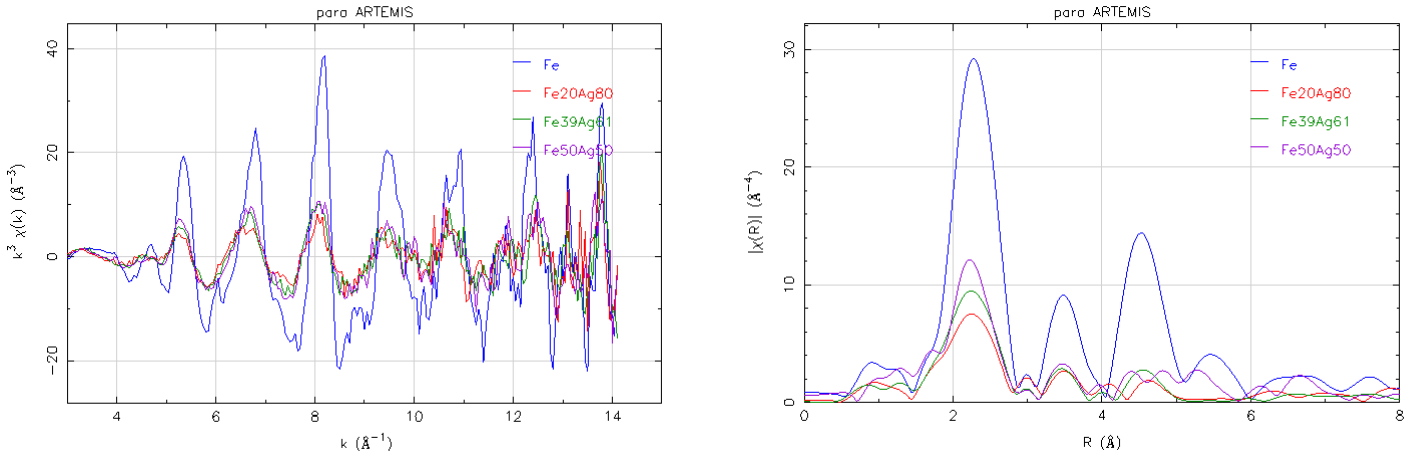

Fig. 1 EXAFS spectra and corresponding Fourier transforms for three Fe_xAg_{100-x} samples with different compositions (x = 20, 39 and 50) and a Fe film.

	Fe film	**Fe20Ag80**	**Fe39Ag61**	**Fe50Ag50**
r_1 (Å)	2.483	2.512	2.509	2.501
σ_1^2	0.0069	0.0186	0.0157	0.0148

Table 1: Fitting parameters obtained from the fits of the EXAFS spectra.

On the other hand, XANES measurements were performed on Ni-doped Fe_3O_4 nanoparticles. The aim of the experiment was to determine the position of the Ni atoms in the spinel structure as a function of the Ni content, and correlate it with the magnetic properties of the nanoparticles. Due to the low content of Ni in the samples, measurements were performed in fluorescence for the Ni-K edge and in absorption for the Fe-K edge, both at room temperature. XANES spectra performed at the Ni-K edge are shown in figure 2. Changes of the pre-edge and the white line of the ferrite doped with 0.65 at Ni with respect to a foil of NiO and the stechiometric $NiOFe_2O_3$ suggest a partial substitution of Fe^{+3} atoms with Ni^{+3}.

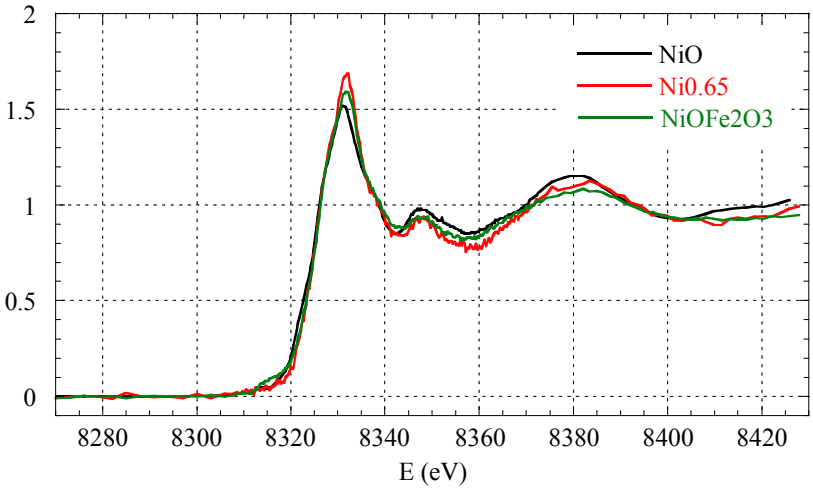

Figure 3: Ni-K XANES spectra for NiO foil, Ni0.65Fe2.35O4 and the stechiometric composition (NiOFe2O3).