ESRF	**Experiment title:** SAXS/WAXS on FeAg nanogranular thin films	**Experiment number:** 16-02-40
Beamline: BM16	**Date of experiment:** from: 24 Sept 2008 to: 27 Sept 2008	**Date of report:** December 5, 2008 *Received at ESRF:*
Shifts: 9	**Local contact(s):** Dr. Francois Fauth	

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Report:

Granular alloys composed of magnetic nanoparticles embedded in a metallic non-magnetic matrix show a rich variety of magnetic phenomena dominated by the magnetic interactions inside the particles and in between them. By varying the volume fraction one can obtain interesting magnetic collective behaviours, from an interacting superparamagnet (ISPM) [1] at low enough concentrations to a superferromagnet [2] or a correlated susperspin glass (CSSG) [3] at higher volume fractions. Additionally, these collective behaviours are combined with the individual magnetic properties of the nanoparticles: from superparamagnetic to ferromagnetic single- or multi-domain as the particle size increases. The combination of collective and individual magnetic interactions gives rise to very complex magnetic phenomena that will be closely correlated to the microstructure of the samples, in particular the nanoparticles size and size distribution, distance between particles and interfacial roughness.

We have prepared Fe_xAg_{100-x} thin films in concentrations close to the percolation limit ($30 \leq x \leq 55$). Due to Fe and Ag not being miscible, both elements precipitate and the samples consist of a Ag matrix in which Fe clusters are embedded. We have prepared samples of similar compositions by two different preparation techniques, namely DC magnetron sputtering and pulsed laser deposition (PLD). From SAXS/GISAXS measurements we expected to obtain microstructural information necessary to explain the complex magnetic behaviour described in the above paragraph.

In BM16 we performed SAXS measurements in transmission and in grazing incidence (GISAXS) configurations. We measured 4 PLD and 3 DC sputtered FeAg thin films, Fe and Ag foils and 3 bare substrates. The CCD detector was fixed at 2 m from the sample and consequently, for an incident energy close to the Fe K-edge, the Q-range was approximately $0.05 \leq Q \leq 1$ nm^{-1}. Measurements in transmission were taken in anomalous conditions. Therefore, each sample was measured well-before the Fe K-edge (7.08 keV) and on the edge (7.112 keV). However, no differences were observed between the two spectra and as a consequence the GISAXS experiment that followed was performed only at 7.08 keV.

Measurements performed in transmission are currently under study. Their analysis is complex because the thin films are deposited onto a Si substrate of several hundred microns of thickness, which absorbs most of the incident beam.

GISAXS measurements were performed at 10 different incident angles between 0.3 and 0.75°. Figure 1 shows the GISAXS spectra of a PLD sample (left) and a sputtered one (right) for an incident angle of 0.70°. Simply by direct inspection of the figures one can appreciate a drastic difference in the microstructure of the samples depending on the preparation method. Knowing that the image can be thought of as a plane perpendicular to the film surface, in the PLD film a typical structure of frustrated multilayers is observed, while for the sputtered one the clusters are pilled up, forming frustrated columns. This result can be attributed to the way the films are deposited in each case: in the PLD films the laser beam impacts on a target that is a rotating disc with sectors of Ag and Fe which area corresponds to the desired composition of the final thin film. In the sputtered case a similar target is used, but unlike in PLD, it does not rotate, therefore the columnar stacking of the Fe clusters.

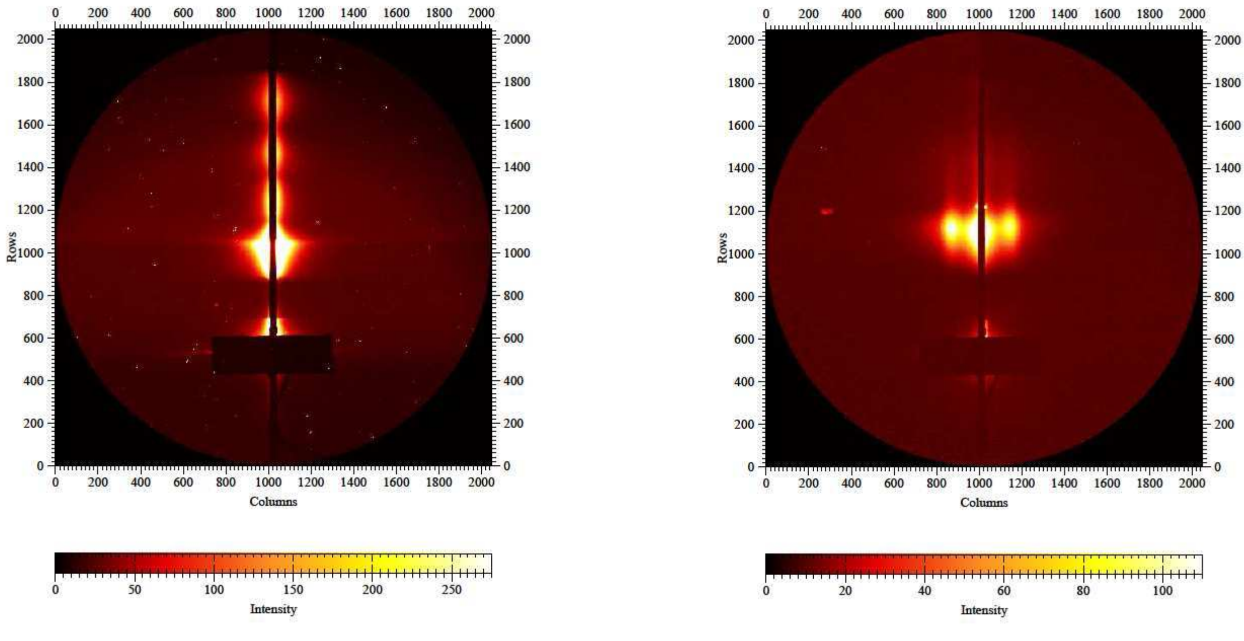

Figure 1: GISAXS spectra of a $Fe_{51}Ag_{49}$ PLD thin film (left) and a $Fe_{35}Ag_{65}$ thin film prepared by DC magnetron sputtering (right).