



	<b>Experiment title:</b> <b>In-Situ GISAXS and GIWAXS of C/FePt multilayers</b>	<b>Experiment number:</b> 32-02-613
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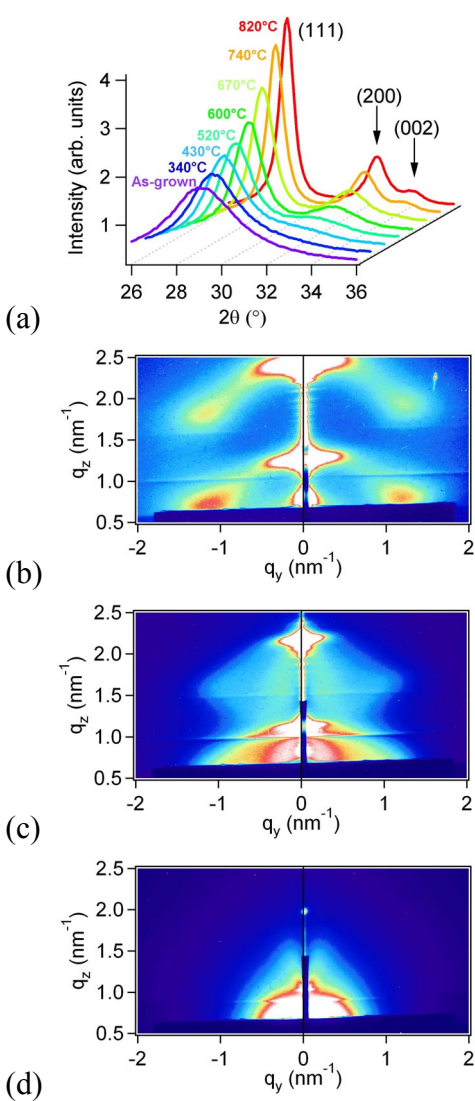
### Report:

The initial aim of this project was to study the structural modifications at both the atomic and nanometric scales induced in C/FePt multilayers under annealing, by means of coupled *in-situ* grazing incidence x-ray diffraction (GIXRD) and small-angle x-ray scattering (GISAXS) experiments. 2 (C  $t_C$ /FePt  $t_{FePt}$ ) multilayers have been prepared at room temperature by ion-beam sputtering deposition, with the thickness of carbon  $t_C = 4$  nm and the thickness of FePt  $t_{FePt} = 1$  and 2 nm respectively. The number of bilayers was adjusted so that the total thickness of the multilayers was around 200 nm, with the first and last layers being carbon layers. In order to study the influence of the matrix and the surfactant effect of silver on the FePt growth, 3 additional multilayers have been prepared with the following characteristics: (C 4 nm/FePtAg 1 nm), (BN 4 nm/FePt 1 nm), and (TiN 4 nm/FePt 1 nm). All the samples were annealed *ex-situ* at 600 °C for 1h and ion-irradiated with  $2 \times 10^{16}$  He<sup>+</sup>.cm<sup>-2</sup> at 600 °C. GISAXS measurements have been carried out at 11270 eV using a 2D CCD detector fixed in front of the direct beam. The angle of incidence ( $\sim 0.3^\circ$ ) was adjusted so that the whole multilayers were fully irradiated. XRD measurements have been performed in both  $\theta$ - $2\theta$  and grazing incidence ( $\omega = 1^\circ$ ) configurations using a NaI detector with  $2\theta$  being varied from  $10^\circ$  to  $62^\circ$ .

*Ex-situ* GISAXS and (GI)XRD data clearly show that (C 4 nm/FePt 1 nm), (C 4 nm/FePtAg 1 nm), and (BN 4 nm/FePt 1 nm) multilayers contain self-organized nanometric FePt clusters whereas (C 4 nm/FePt 2 nm) and (TiN 4 nm/FePt 1 nm) multilayers consist of a stacking of continuous C (or TiN) and FePt layers. Furthermore, after annealing

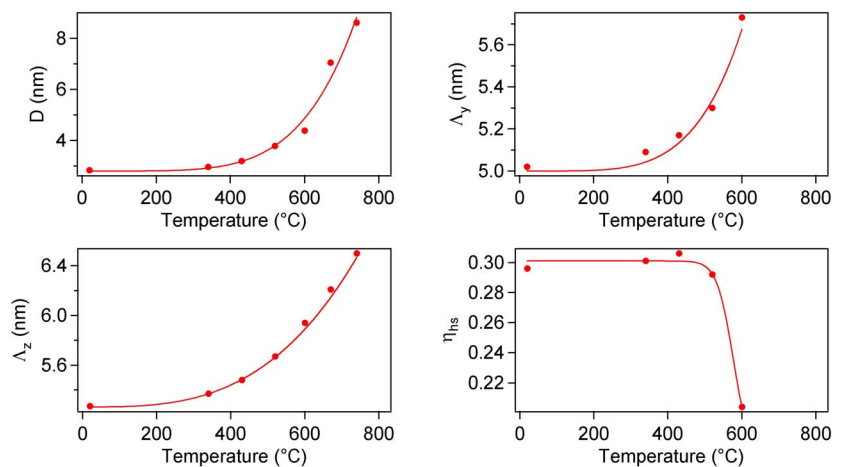
or irradiation, all the multilayers undergo a phase transition from a disordered Fe-Pt fcc structure (magnetically soft) to an ordered fct structure ( $L1_0$  phase, magnetically hard). The quantitative analysis of the experimental data, which is still in progress, will permit the determination of the clusters morphology (shape, size, size distribution, organization) and of the fraction of ordered FePt in the multilayers as a function of the growth conditions and of the post-growth treatments.

To follow in real time the structural evolution of a multilayer containing FePt clusters, the (C 4 nm/FePt 1 nm) multilayer has been subjected to annealing under vacuum at  $10^{-6}$  mbar. *In-situ* GISAXS and GIXRD acquisitions has been completed successively at room temperature, 340 °C, 430 °C, 520 °C, 600 °C, 670 °C, 740 °C, and 820 °C. As shown in Figure 1(a), the transition from the disordered fcc phase to the  $L1_0$  phase is characterized by the presence of the (002) superlattice peak in the GIXRD scans obtained at high temperatures. The quantitative analysis of these scans will permit the determination of the crystallographic parameter order as a function of the annealing temperature. Moreover, the crystallographic phase transition comes with strong modifications of the clusters morphology as demonstrated by GISAXS measurements presented in Figure 1(b) to 1(d). The thermal evolution of the structural parameters (cluster size  $D$ , lateral interparticle distance  $\Lambda_y$ , vertical



**Figure 1:** *In-situ* GIXRD scans (a) and GISAXS patterns at room temperature (b), 600 °C (c), and 820 °C (d) of the (C 4 nm/FePt 1 nm) multilayer.

interparticle distance  $\Lambda_z$ , and structural parameter order  $\eta_{hs}$ ) obtained from the analysis of the corresponding GISAXS patterns is displayed in Figure 2. The data clearly show an increase of  $D$ ,  $\Lambda_y$ , and  $\Lambda_z$  with the annealing temperature whereas  $\eta_{hs}$  strongly decreases from 600 °C. It is worth noting that these results are in agreement with transmission electron microscopy observations and magnetic measurements performed *ex-situ*. In conclusion, the obtained results constitute a crucial information for the understanding and the control of the nucleation, growth, ordering and magnetic properties in FePt-based clusters systems and for the optimization of their potentiality as recording media.



**Figure 2:** Evolution of the structural parameters as a function of the annealing temperature deduced from the *in-situ* GISAXS measurements of the (C 4 nm/FePt 1 nm) multilayer.