Experimental Report (14/11/2016) Proposal 32-02-729

"Behavior of preformed size selected FePt nanoparticles soft landed on the moiré pattern of graphene/Ir(111)."

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Number of shifts: 18 Local contact: Gilles Renaud

Objective and expected results:

The FePt alloy, when chemically ordered in the $L1_0$ phase, is among the magnetic materials displaying the highest magnetic anisotropy constant (Keff around 7 MJ/m^3). Therefore it is a perfect candidate for ultra-high density magnetic storage applications, provided nanoparticles can be prepared in such a high anisotropy phase[1]. Another requirement for applications, as well as for fundamental studies, is to organize the magnetic nanoparticles in a regular 2D array. In parallel to investigations on chemically synthesized systems, a great effort is devoted to the bottom-up elaboration of nanomagnet arrays following a physical route. In this context, one widely used path consists in using template surfaces with specific sites regularly distributed. Such a 2D lattice can be obtained with the moiré phenomenon, which appears when two crystalline structures of slightly different cell parameters are stacked. Thus, a graphene layer epitaxially grown on a Ir(111) surface displays a 2D spatial modulation corresponding to a hexagonal lattice of 2.5 nm cell parameter. This moiré has already been successfully used for the growth of nanoparticle arrays (islands) by atomic deposition[2]. Recently, we have followed an original approach where preformed and size-selected clusters (pure Pt of 1.5 nm diameter) are deposited on a graphene/Ir(111) surface displaying a moiré lattice. By using grazing incidence x-ray scattering measurements (GISAXS technique), performed at the ESRF, the organization of particles on specific sites of the moiré lattice has been put into evidence[3,4]. This indicates that the nanoparticles are sensitive to the moiré pattern and we have found that the resulting organization can be preserved up to temperatures around 700 K[4]. Note also that such organized arrays of FePt alloyed particles cannot be obtained by atomic deposition, which makes preformed cluster deposition highly valuable.

We want to extend this approach to FePt nanoparticles and expect similar results for the organization of FePt nanoparticles of size 2.2 nm (size below the cell parameter) and 4 nm (larger than the cell parameter) as function of the temperature.

Results and preliminary conclusions:

Size-selected FePt particles (2.2 nm and 4 nm) are deposited on epitaxially grown graphene on a prepared Ir(111) surface. We have made 4 samples with density of $3x10^4$ nanoparticles/μm^2 (occupation of the moiré sites up to 16%) with different deposition temperatures. The following table gives a summary of our samples.

	S1 (2.2 nm)	S2 (2.2 nm)	S3 (4 nm)	S4 (2.2 nm)
$T_{deposition}$	RT	150°C	150°C	300°C
Info	Amorphous carbon capping	Amorphous carbon capping	No capping	Amorphous carbon capping

Tableau 1. Summary on the preparation of our 4 samples, with 2 different sizes for nanoparticles : 2 nm and 4 nm and temperatures of the substrate during the deposition of the nanoparticles.

The Ir(111) surface is prepared in the UHV chamber of the BM32 beamline using ion bombardments and annealing. The layer of graphene is grown in the same chamber following a well-known process[3]. The samples are then transfered to Lyon in UHV conditions at the PLYRA for the deposition process of FePt NPs and back to Grenoble in the same conditions. Our cluster deposition setup can select pre-formed NPs in size with a relative diameter dispersion < 10%. 2D assemblies of FePt NPs are prepared on the moiré (2.5 nm periodicity) of Gr/Ir(111). The samples are protected by a thin amorphous carbon layer (around 5 nm thick), enabling a transfer in air without oxidation and annealing without particle diffusion. Such a procedure is well mastered.

On the figure 1a and 1b. We can distinguish the correlation peak as well as the form factor indicating an organization, at least partial, of 2.2 nm FePt nanoparticles on the Moiré sites. This implies that they have been able to diffuse on the surface and self-organize. The increase of the correlation peak indicates that the deposition temperature has an influence on the organization of the FePt nanoparticles ($T_{deposition}$ for 1a is room temperature and 1b is 150°C). Both images have been taken at RT. Out of azimuth, the correlation peak is not visible (figure 1c), this suggests a hexagonal organization corresponding to the moiré lattice.

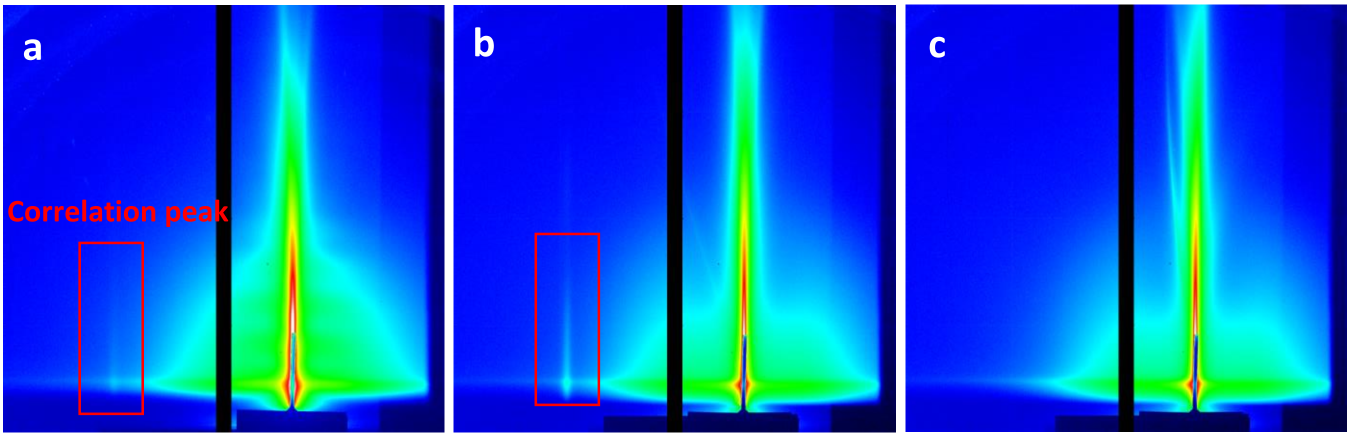

Figure 1.(a) (b) GISAXS images in the <100> direction showing the influence of the temperature of the deposition respectively at RT (S1) and at 150°C (S2). (c) GISAXS image out of azimuth of the sample S2.

As function of the temperature, for the sample 2, the correlation peak is evolving indicating a possible modification of the organization as well as a small change of the form factor (figure 2c). The GISAXS image 2a has been taken at 700°C and an organization can still be seen after the expected $L1_0$

phase transition temperature (~600°C). The image 2b is showing the evolution of the correlation peak as function of the temperature (from RT to 700°C).

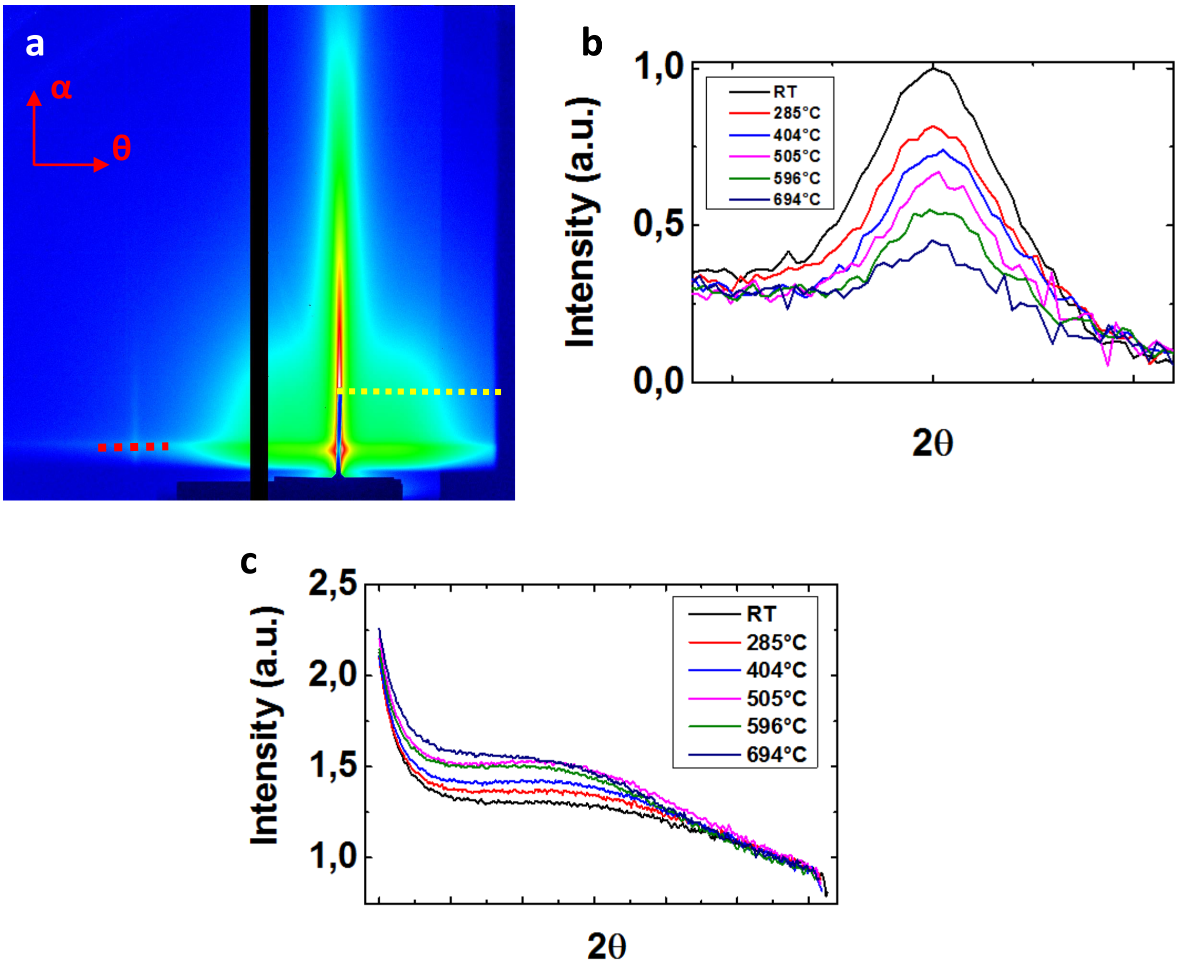

Figure 2. (a) GISAXS image in the <100> direction showing an organization at high temperature (700°C – S2). (b) Evolution of the correlation peak as function of the temperature taken at the base of the correlation peak as illustrated by the red dashed line in the figure 2a. (c) Evolution of the form factor as function of the temperature taken from the middle of the central intensity as illustrated by the yellow dashed line in the figure 2a.

The figures 3a presents a GISAXS image of the 4 nm nanoparticles taken at RT. Similar to the previous sample, we observe an organization of the nanoparticles. The figure 3b shows the GIXD curves as function of the temperature of the 2nd order of the iridium peak surrounded by the moiré peak (1.88) on the 4 nm nanoparticles sample. The moiré peak disappear after an annealing at 500°C. This could come from the modification of the nanoparticle layer (nanoparticles moving away from the moiré sites, coalescence, nanoparticles intercalating between graphene and the substrate…). The figure 3c indicates us that the nanoparticles do not undergo a shape modification. We could assume that the nanoparticles are just moving away from their moiré sites. The moiré peak on the GIXD measurements has not been seen on the 2 nm nanoparticles samples and needs to be interpreted. The shift of the moiré peak originates from the dilatation of the lattice due to the increase of the temperature.

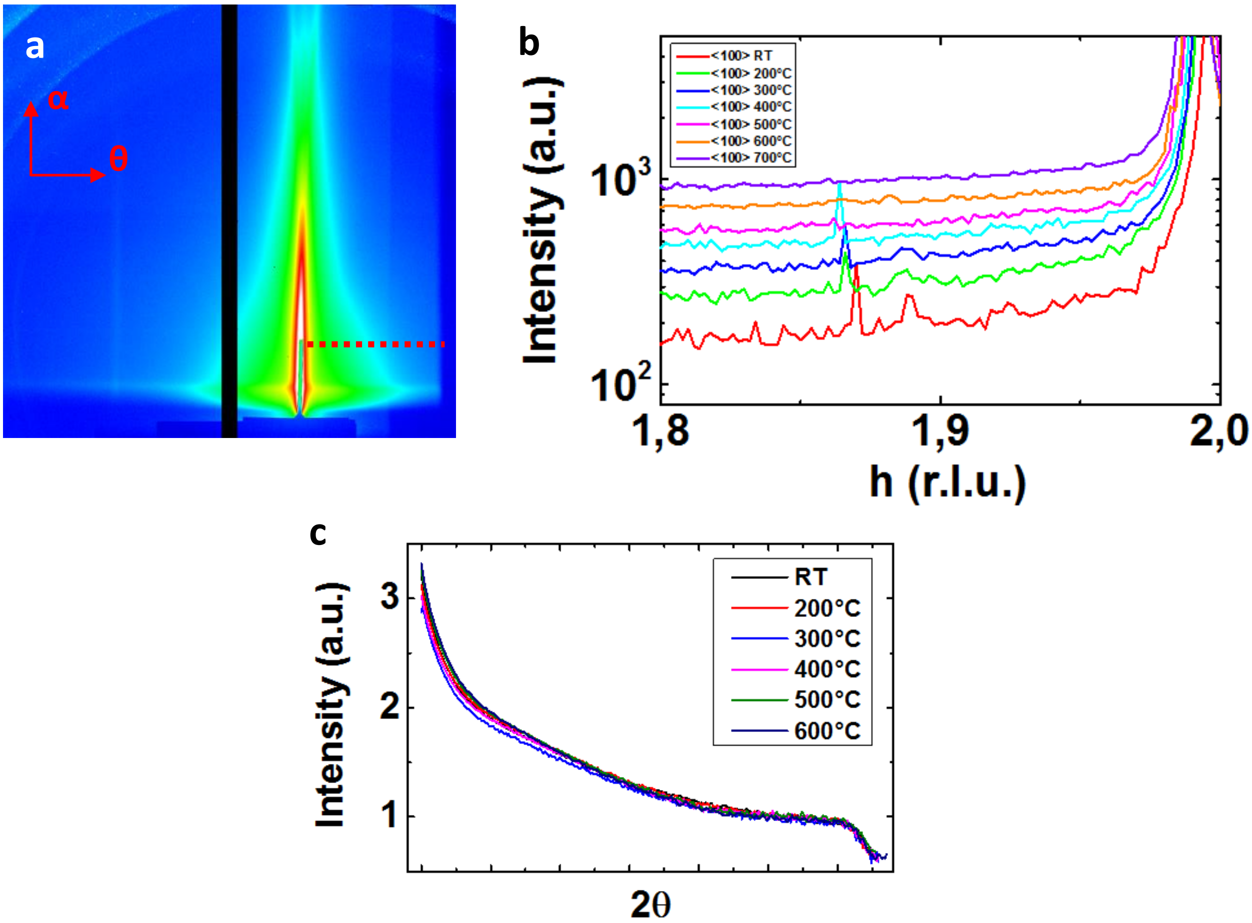

Figure 3. (a) GISAXS image in the <100> direction showing an organization with particles larger than the moiré lattice at room temperature. (b) GIXD scans as function of the temperature of the 2nd order iridium peak and its vicinity. The moiré peak can be visible until 500°C. Curves have been shifted for clarity. (c) Evolution of the form factor as function of the temperature for the sample 3, the cross sections have been taken from the central intensity as highlighted by the red dashed line on the image 3a.

<u>References</u>

[1] P. Andreazza et al., Surf. Sci. Rep. 70, 188 (2015).

[2] Q. Liao et al., Nanotech. 22, 125303 (2011) ; A. T. N'Diaye et al., New J. Phys. 11, 103045 (2009).

[3] S. Linas et al., Sci. Rep. 5, 13053 (2015).

[4] Experimental report of ESRF proposal MA2099.