Experimental Report (30/08/2013)

Proposal 32-03-717

"Structure, morphology and ordering of preformed size-selected Pt-based nanoclusters deposited on graphene/Ir moire patterns"

Beamline: IF-INS BM32
Number of shifts: 18

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Objective and expected results:

We aimed at determining the structure, epitaxial relationships, lattice relaxation, shape, organization and possibly locations of preformed Pt-based nanoparticles deposited on the moiré pattern of graphene/Ir(111). We had planned to consider 1.5 nm diameter Pt and CoPt particles, for two different surface coverages, and follow the evolution of the system with the annealing temperature (up to 300°C). The purpose was to gain insight on both the cluster/cluster and cluster/surface interactions (diffusion, pinning, ordering, coalescence). This proposal is part of the NMGEM ANR project which aims to explore nanomagnetism on graphene/metals. Due to the original behaviour of size-selected Pt clusters deposited on graphite (where a local hexagonal self-organization is observed with a ~1 nm interparticle separation), [1] we expected to observe a self-organization of the Pt nanoparticles on the moiré lattice (periodicity of 2.5 nm).

Results and preliminary conclusions:

Three samples have been studied. For each sample, a graphene monolayer has been grown on the (111) surface of an Ir single crystal following the procedure described in ref. 2, and characterized by x-ray diffraction. The sample has then been transferred, under UHV, to the PLYRA platform of Lyon university for cluster deposition (LECBD technique). Size-selected Pt clusters, with a diameter around 1.45 nm have been soft-landed (random deposition) on the moiré pattern. The three samples correspond to different coverages: ~6% for S1, ~25% for S2 and ~20% for S3. It should be noted, that given the particle size and the moiré lattice parameter (2.5 nm), a full occupation of each site corresponds to a coverage ~30%. Samples S1 and S2 have unfortunately been exposed to air **after** deposition (problem of sample fixation) while S3 has been prepared and characterized fully in UHV conditions. However, at this point, no major differences were detected between S2 and S3. S1 has been annealed during the x-ray measurements, at a moderate temperature (~200°C), while S3 has been annealed up to 600°C.

As shown in Fig. 1 (sample S1), the GISAXS pattern indicates that the Pt particles are located on the moiré hexagonal lattice. This implies that they have been able to diffuse on the surface and self-organize at room temperature. Some particles must however be almost isolated (no neighbours on the nearest moiré lattice sites) so that the form factor of the Pt clusters is apparent on the GISAXS pattern. This is due to the low coverage used for S1. The GIXRD measurements also indicate that the Pt particles could be in a (111) epitaxy over the Ir crystal (see Fig. 1): this is visible as a shoulder at the bottom of the Ir 110 and 111 diffraction peaks. However, the difference between Pt and Ir lattice parameter is very small (~2%).

Another indication of the location of Pt particles on the moiré lattice is visible on the GIXRD measurements. As shown in Fig. 2, the moiré peaks are more pronounced when Pt particles have been deposited on top of the graphene/Ir substrate. This effect has also been observed in the case of Pt particles obtained by atomic deposition [2]. Interestingly, the intensity of these moiré peaks decreases upon annealing and vanishes between 220°C and 300°C (see Fig. 2). At the same time, the Pt nanoparticle organization evolution has been followed, up to 600°C. As shown in Fig. 3, while the particle coalescence is limited, the lattice organization remains visible up to around 500°C. The results still need to be analyzed in details and further experiments with bi-metallic particles should be very fruitful!

References:

[1] D. Tainoff *et al.*, J. Phys. Chem. C **112**, 6842 (2008).
[2] N. Blanc *et al.*, Phys. Rev. B **86**, 235439 (2012).

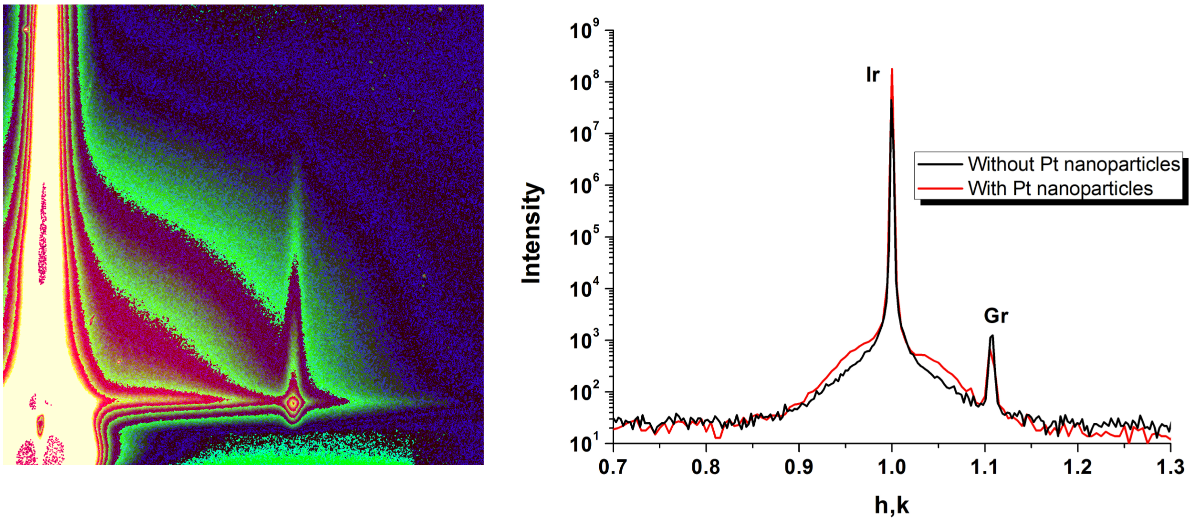

Fig. 1: (Left) GISAXS pattern at room temperature for sample S1. The peak corresponding to the moiré superlattice is clearly visible. (Right) GIXRD measurements for sample S1 around the Ir (110) peak. A shoulder is visible after Pt nanoparticle deposition.

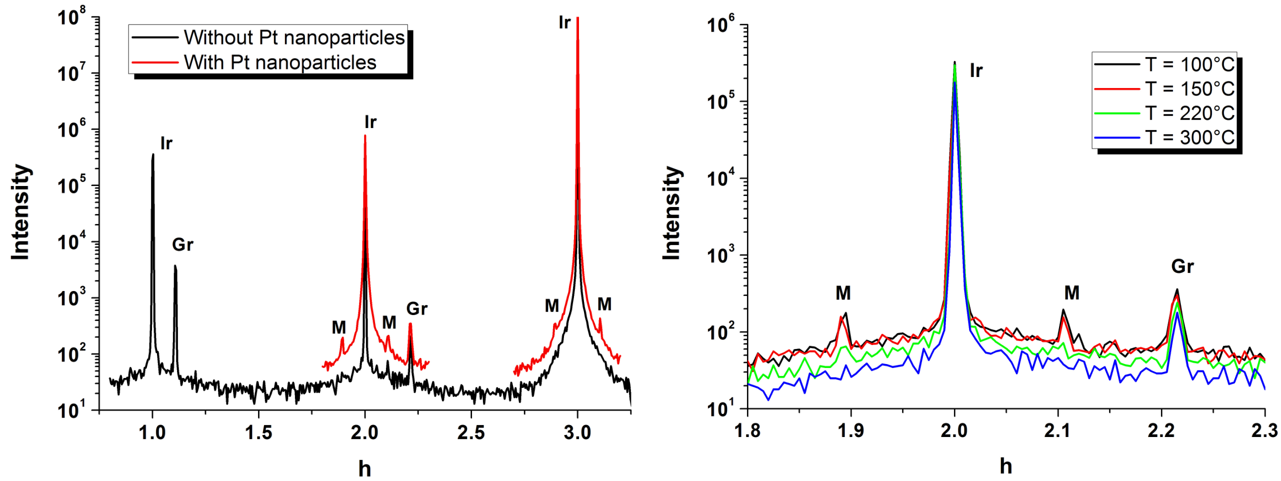

Fig. 2: (Left) Comparison of the GIXRD peak intensities, along the (h00) direction, before and after Pt nanoparticle deposition (sample S3). The moiré peaks are labelled by the letter "M". (Right) Evolution upon annealing of the GIXRD peak intensities, in the (h00) direction, for sample S3.

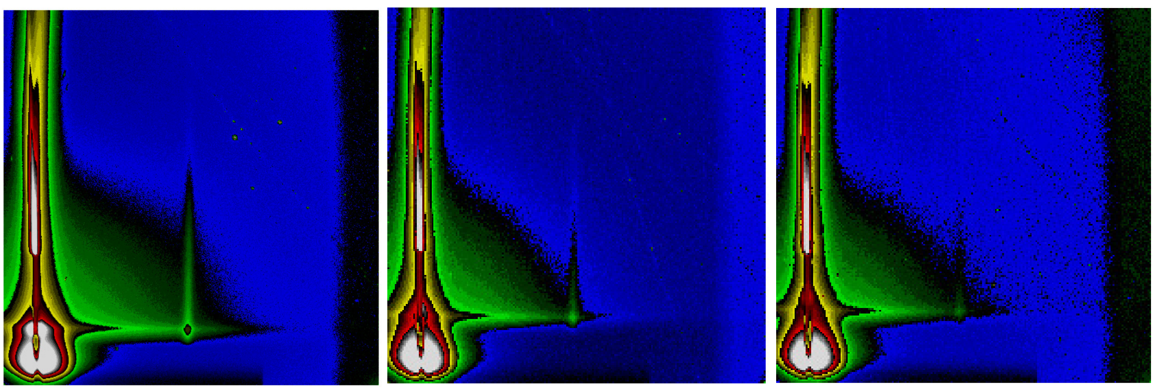

Fig. 3: Evolution of the GISAXS pattern for sample S3 upon annealing. From left to right, the temperature is room temperature, 300°C and 490°C. The peak corresponding to the moiré lattice organization decreases.