ESRF	Experiment title: STUDY OF FIVEFOLD SYMMETRY NANOMETRIC COBALT CLUSTERS	Experiment number:
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6		
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Report:

The aim of the proposed experiments was to:

- 1) Resolve the structure of the intrinsically strained icosahedral cobalt core
- 2) Gain some structural information on the structure of the CoO shell in its early stage of growth

This information will be used as inputs or guidelines for an in-going program on the micromagnetic simulation of the exchange coupling involving clusters.

One experimental difficulty comes from the fact that the amount of deposited clusters is low, usually below two monolayers, in order to avoid sintering. It is thus necessary to use grazing incidence diffraction (GID) to minimize substrate signal. The measurements were carried on ID01 beamline with the energy set at 17 keV, far from the Co K edge, at a value which allows scan up to Q=10.

During our run we looked at six samples:

 2 monolayers of 5 nm diameter clusters, deposited on Si(100), covered with a 30 nm alumina layer (to prevent oxidation) (sample C06);

- 2 monolayers of 1.3 nm diameter clusters, deposited on Si(100), covered with 10 nm of alumina (sample C07);
- 0.1 monolayer of 6 nm diameter clusters, deposited on Si(100), covered with 20 nm of alumina (sample C13);
- 4) 0.5 monolayer of 5 nm diameter clusters, deposited on Si(100), covered with 10 nm of alumina layer. In this case the clusters were intentionally exposed to 2000 L of O₂ gas, under high vacuum, to form an oxide shell around Co cores (sample B62);
- 40 nm thick layer of 5 nm diameter clusters deposited on glass, with no protection against oxidation (sample B31);
- 6) one bare Si(100) substrate.

In each case the measurements began by locating the critical angle, by measuring the reflectivity, from which the grazing angle was determined. After this we had to find out the good scan conditions and directions – in the silicon reciprocal space – in order to avoid the strong substrate peaks. The best choice turned out to combine two scans: the first one in the Si(220) direction and a second one in the Si(400) direction. By clipping the two scans it is possible to obtain a full scan with minimal Si background (Fig. 1 and Fig. 2). In addition, we also performed diffraction scans in an intermediate direction relative to the substrate's orientation, as far as possible from Si peaks.

Results for the different samples are shown in Fig. 2 – 4. Figure 2 shows diffraction pattern obtained with clusters of 4.7 and 1.3 nm diameter. Diffraction spectra for clusters covered with a shell of Co oxide are compared with pure cobalt clusters in Fig. 3. One can see the difference in the peaks in the 25° - 40° region. Finally, Fig. 4 shows the diffracted intensity for a thick layer of cobalt clusters deposited on glass. The different curves correspond to different values in the incidence angle. A broad nearest neighbour peak appears at low angle as the angle is increased. Although the cobalt signal is higher due to the increased thickness of clusters, it turns out that the substrate signal is possibly less annoying with an amorphous material.

Work is now under progress to compare all these results with structural numerical simulations. It should be possible to make the difference between the fcc and the icosahedral structure, and possibly to identify the nature of the oxide that is formed around the Co cores.



Figure 1: Bare silicon substrate along the (220) and (400) directions.



Figure 2: Cobalt clusters diffraction spectra obtained by data clipping from two Si direction scans.



Figure 3: Diffraction spectra for oxidized (B62) and un-oxidized (C06) clusters.



Figure 4: Diffraction spectra for cobalt clusters deposited on glass.