

A look inside epitaxial cobalt nanoparticles with a 3D reciprocal space imaging

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1 Introduction

In recent years intense research efforts have been devoted to the heterostructures with ferro- and antiferromagnetic layers. Particular effects observed in these systems are of great importance as being related to the design of modern high density magnetic media [1]. Cobalt nanoparticles on Si produced by growth of Co on nonmagnetic CaF₂ (used as a buffer) or antiferromagnetic MnF₂, provide a suitable framework to study properties of magnetically ordered nanosystems. The current work presents an extensive diffraction study of such a system with Co epitaxially grown on CaF₂ / Si.

2 Experiment

Cobalt nanoparticles were grown at 600°C on CaF₂ / Si by molecular beam epitaxy (MBE) at Ioffe institute. A 0.1 nm low temperature Co seeding layer was used to ensure nucleation of regular arrays of nanoparticle with high crystalline quality. Nanoparticles with three major orientations of cobalt lattice (111, 110 and 001) were produced by appropriate choice of Si substrate face and CaF₂ growth parameters.

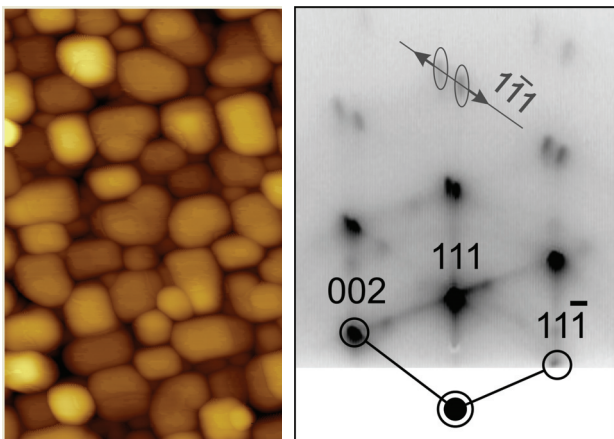


Fig. 1: AFM image of Co(001) nanoparticles (left). Image size 1000 nm × 600 nm × 80 nm. RHEED pattern from Co(111) nanoparticles (right) showing streaking and Bragg reflection splitting in the (1-10) zone.

The structures have been pre-characterized by atomic force microscopy and electron diffraction prior to the synchrotron studies (Fig. 1). Interestingly, RHEED patterns exhibit bright (111) and (11-1) streaks showing up as lines normal to the sample surface and inclined by 70.5° correspondingly. Presence of the other two streaks: (1-11) and (-111) is expected due to the symmetry reasons.

These streaks indeed appear in RHEED images causing Bragg reflections to split when the sample is rocked. In order to study streaking and reflection shape in general the samples were investigated by GIXD at BL3A beamline at Photon Factory using $\lambda=1.033$ Å radiation. The measurements were partly carried out with a Hamamatsu 2D CCD detector. Finally GIASXS measurements have been carried out at the same beamline to study particle faceting.

3 3D mapping by GIXD

The electron and X-ray diffraction studies confirm that on either face of CaF₂ cobalt grows in fcc phase with its lattice cooriented with the underlying fluorite layer. Interestingly bright and long {111} streaks have been observed passing through Bragg reflections of Co by recording 3D intensity maps around several Bragg reflections of cobalt (Fig. 2 top).

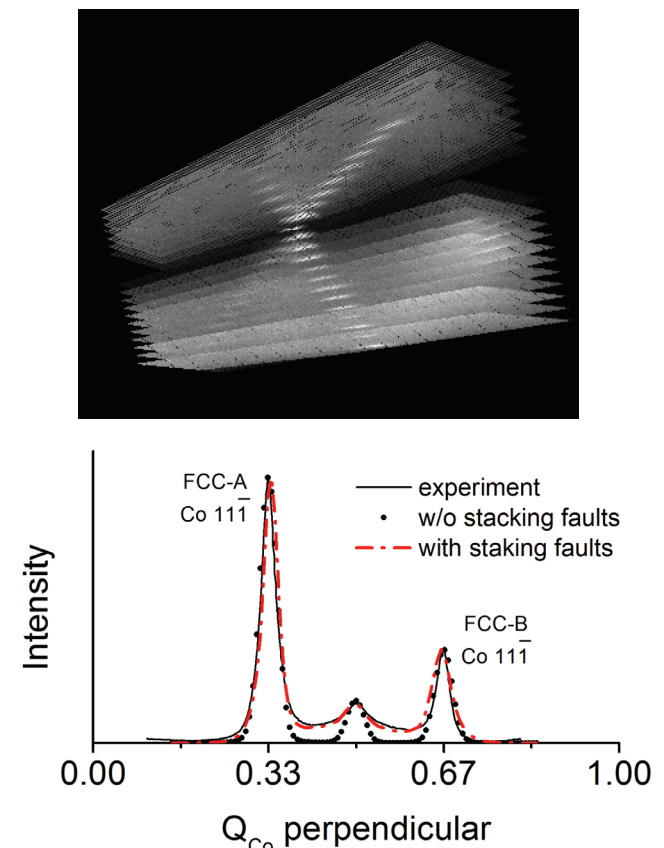


Fig. 2: GIXD data obtained from Co(111) nanoparticles. 3D intensity distribution around Co(111) reflection

showing {111} streaks (top). Experimental and simulated intensity profiles along the Co [111] streaks (bottom).

Intensity profiles along the streaks have been recorded and then modeled assuming that islands are faceted with {111} planes (Fig. 2 bottom). It was shown that although faceting does produce streaks, the intensity along these streaks drops much faster ($\sim q^{-2}$) compared to the experiment. The other hypothesis checked was that the streaks are due to the stacking faults. The latter may naturally occur in cobalt that is stable as hcp at normal conditions and grows as fcc at elevated temperatures. The length of the stacking fault related streaks is defined by the correlation length in the faulted structure. A calculation was carried out suggesting three types of stacking order existing within a cobalt island. The calculation involved random insertion of stacking faults and averaging over few thousands of islands. A reasonable fit was achieved for a fault density of $\sim 0.06 \text{ \AA}^{-1}$ (Fig. 2 bottom). Although the presented data show data for Co (111) islands, similar 3D intensity distribution maps have been also measured for Co (001) and Co(110) islands to show presence of {111} streaks with similar intensity profiles.

3 GISAXS studies

Since the stacking fault streaks mask any existing facet streaks, information about faceting can hardly be extracted from the Bragg reflection shape. For this reason Co nanoparticles were further examined with GISAXS that is not influenced by lattice defects and at the same time is sensitive to the particle shape. GISAXS patterns from Co(111) islands (Fig. 3) clearly show streaks perpendicular to Co(111) planes. In case when cobalt was grown on CaF_2 (110) surface, streaks from (001) facets were also present.

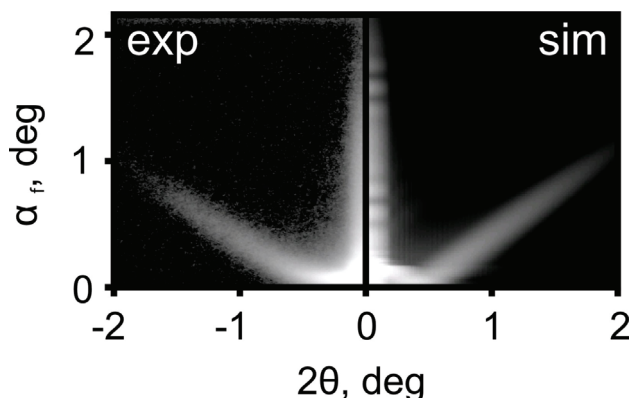


Fig. 3: Comparison of experimental (left) and simulated (right) GISAXS patterns from Co(001) islands.

The intensity was found to drop fast along the streaks following the q^{-n} behavior where n ranged between 2.4 and 3.6. This is faster than expected from a parallelepiped (q^{-2}) and slower than for a polyhedron (q^{-4}). The GISAXS patterns were then analyzed using the “simulation annealing” algorithm implemented in the IsGISAXS

software [2]. A reasonable fit was achieved for a model in which Co(001) islands were assumed to be truncated pyramids. The obtained island parameters appeared to be in good agreement with the AFM results allowing a better evaluation of the island aspect ratio.

Conclusion

Epitaxial Co islands of three different substrate induced orientations were extensively studied from inside - to reveal an unstrained fcc lattice with the presence of {111} stacking faults, and from outside - to show that the islands are faceted with {111} and {001} crystal planes. The usage of 3D Bragg reflection imaging combined with GISAXS proved effective to separate different contributions to streaking. Various parameters were obtained from fitting the XRD and GISAXS data. These are statistically averaged over a large sample area that is of particular importance when it comes to understanding magnetic properties of the samples with Co nanoparticles. Simultaneous study of all three cobalt orientations (111, 001 and 110) made it possible to get a more comprehensive understanding of the particle shapes, size distributions and crystal structure. The summarized data obtained for Co on CaF_2 is believed to be useful in predicting the properties of Co nanoparticles on antiferromagnetic MnF_2 .

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