Report on Experiment SI 1935 (November 2009)
Structure and magnetic properties of ultrathin magnetite films grown on (001) surfaces of Fe whiskers.

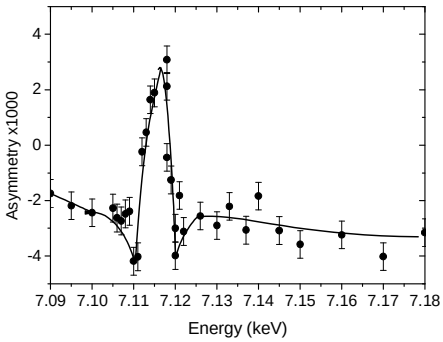
A whisker sample of dimensions 0.5 x0.5 mm (top surface) x 4 mm (length) was selected (on he basis of the flatness of the top surface) from a batch of samples observed by SEM. The sample was mounted on a *ad hoc* sample holder and installed on the "new reactor chamber" of ID3 in which the sample is surrounded by a hemispherical Be dome. The long dimension of the sample was sitting vertical and the top (001) surface in a horizontal plane. Thus, the side faces of the whisker (100) and (010) were vertical.
We concentrated our interest right from the beginning on magnetic measurements. A magnetic coil with an iron core was installed on top of the chamber and powered with a bipolar current amplifier to generate at the sample position magnetic fields up to +/- 600 Oe as determined by preliminary measurements with a Hall probe.
Based on previous experience at the beamline, a Maxipix detector was installed at the detector arm of the vertical diffractometer in Hutch 1. To filter the fluorescence from Fe K edge de-excitation, a well ordered graphite crystal (HOPG) was installed before the detector in a 2 circle goniometer.
Radiation from all three undulators was vertically focused with the meridional mirror of the KB pair to achieve at the sample position a vertical dimension (FWHM) of the beam of about 5-10 μm.
The monochromator was tuned to the K absorption edge (7.112 keV) of Fe and the diffractometer and sample were aligned to collect the (002) reflection of one of the side faces of the whisker.
We encountered a number of experimental difficulties that we summarize:

1.- The Medipix detector has no high pass discriminator and therefore all the photons from the Si (333) reflection (at ~21 keV) that were not rejected by the mirrors or by the monochromator slightly detuned were impinging on the active area since they were not filtered out by the graphite analyzer crystal. A measurement of the harmonic contamination gave around 1% or somewhat less. The consequence was a relatively high background on the pixel detector producing saturation (~11 000 photons per pixel) of several pixels at the detector. Attenuation of the diffracted intensity produced some improvement since the number of pixels reaching saturation events was reduced. However, this did not results in better statistics given the fact the signal that was expected to be measured was pretty small and the diffracted peak was extremely sharp intercepting a small number of pixels. The graphite analyzer crystal was substituted by Ge (111) but not significant improvements were achieved.
After several tests of different procedures we decided to install a scintillation detector with low and high level electronic discrimination. To give an idea of the intensity levels. the Fe Bragg reflection above the absorption edge had an intensity of 2E9 ph/sec , a FWHM of a rocking theta scan of 0.014 degrees and a signal to background ratio evaluated as the peak intensity divided by the intensity at 1.5 times the FWHM of the theta scan equal to 30. Below the K edge the peak intensity was about 7 times higher and the signal to background about twice.
2.- Sample motion

The magnetic field induced a motion of the sample which originated unphysical hysteresis curves at resonant Bragg conditions. This was by far the most difficult problem to localize and quantify since previsions were made on the sample holder to try to avoid this field effect. After many measured cycles done at different conditions aimed to understand the origin of the incorrect shape of the cycles, the motion of the sample was directly measured with optical means. The chamber was opened and a focused laser bean was reflected on a side whisker face. An angular displacement of less than 0.1 mrad could be determined when the magnetic field was applied. As this value (6 mdeg) is comparable to the angular width of the reflection (14 mdeg) we concluded that even a tiny motion may affect the measurements in very sharp reflections.
The sample was mounted on a new holder very rigidly and the possibility of sample heating was sacrificed at the expenses of solid mounting.

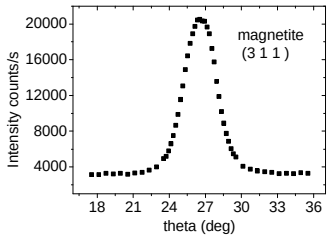
3.- Magnetic results.

The asymmetry ratio R= (I↑- I↓)/(I↑ +I↓) was measured as a function of the energy of the photons across the K absorption edge of Fe on a (200) reflection from a side face.
Each data point in R is the result of about 20-30 minutes data acquisition



The amplitude of the magnetic effect is about 0.007 as expected by a non dipolar electronic transition (1s-3d). The base line of the curve is centered at approx. -3 E-3 instead at 0. The origin of this phenomenon is not understood at the moment.
The crystallographic structure of the sample was then investigated with conventional

diffraction and it was found out that the surfaces consisted on Fe3O4 epitaxial with the metallic Fe substrate. No hematite and maghemite were found, neither FeO. The magnetite film has a thickness of ca. 2 nm as obtained by doing reflectivity measurements and measuring the fringe spacing.
Resonant magnetic measurements were performed at two reflections form magnetite: The specular reflection from the side face of the whisker at the position of the (002) reflection of the magnetite exhibited a too high background to acquire asymmetry data with the required accuracy. On the contrary , the (3 –1 1) reflection from the top surface measured at grazing angles (1 degree) was quite acceptable as shown in figure:



The peak intensity was 17 kc/s on a background of about 3 kc/s . The FWHM was 3.1 degrees.
Resonant magnetic measurements as a function of photon energy across the K absorption edge did not generate significant signals although the conditions of the measurement were judged to be correct.
We suspected that the intensity of the magnetic field was insufficient to polarize the magnetite film.