	Experiment title: The effect of hydrostatic pressure on magnetostriction measured by differential EXAFS	**Experiment number:** IH HC 474
Beamline: ID24	**Date of experiment:** from: 20/02/04 to: 23/02/04	**Date of report:** 25/02/04
Shifts: 9	**Local contact(s):** S. Pascarelli	*Received at ESRF:*

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Report:

We have recently assessed the feasibility of performing X-ray linear dichroism measurements of anisotropic (Joule) magnetostriction at high pressure on a test sample of polycrystalline FeCo. The method we used, based on the measurement of a differential EXAFS signal, was initially suggested by R. Pettifer (Univ. of Warwick) and has been developed in the past 2 years on the dispersive XAS beamline ID24 through a collaborative effort between the beamline staff, the Warwick group and M. Gibbs (Univ. of Sheffield) who provided the samples (proposals MI510, MI588 and HS2102).

For these first tests at high pressure, a 50 x 100 μm^2 chip of annealed polycrystalline FeCo film (7 μm thick) was inserted into a CuBe Diamond Anvil Cell (provided by A. Barla, ID18) which was then placed at the center of a rotating magnetic field device. The device is based on a prototype developed for MI510 but redesigned for the high pressure applications. The gap is larger to fit the 22 mm diameter DAC, the magnetic field intensity was also maximized by using different magnets, and the sample holder was redesigned based on the geometry of the DAC. The new device, shown in Figure 1, is composed of 8 elements of U42 undulator (furnished by J. Chavanne, Insertion Device Group, ESRF) and has been assembled by the Sample Environment Lab (P. Van Der Linden) and the beamline technician S. Pasternak. The system allows the rotation of the magnetic field on a plane perpendicular to the propagation of the X-rays. The calculated intensity of the magnetic field in the center of the 24 mm x 26 mm gap is 0.53 T. The field intensity distribution is relatively flat in the center of the gap, remaining above 99% of its maximum value over the central 2 mm in both directions.

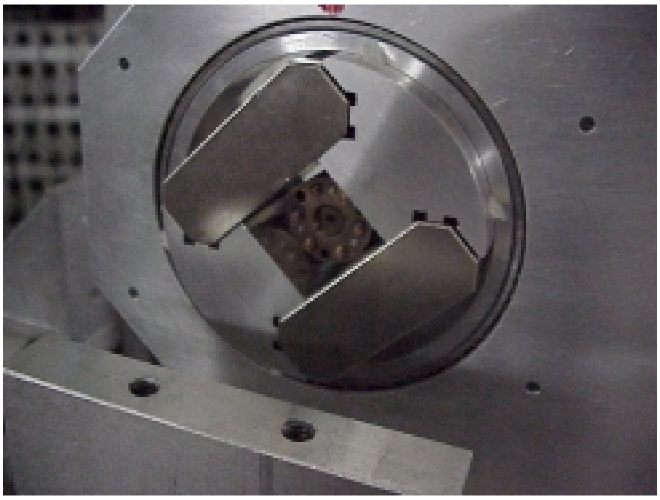

Figure 1. Rotating magnet device with the CuBe DAC positioned within the gap (not supported). The calculated value of the field at the center of the gap is ~ 0.53 T.

EXAFS measurements at the Fe K edge were acquired with the magnetic field parallel ($\mu\|$) and perpendicular ($\mu\perp$) to the polarization of the electric field of the X-rays emitted by one of the planar undulators of the ID24 straight section. The normalized differential EXAFS signal is defined as: $(\mu\| - \mu\perp)/(\mu\| + \mu\perp)$. The signal was averaged over N number of consecutive pairs ($\mu\|$, $\mu\perp$) using a summing procedure that was developed on ID24 for the XMCD applications and eliminates linear drifts.

For each value of pressure, we performed 3 kinds of measurements that we denominated “90”, “0” and “45”:

90: the first acquisition is $\mu\perp$ (magnets on top and bottom of DAC)

0: the first acquisition is $\mu\|$ (magnets on left and right of DAC)

45: the first acquisition is with H at 45° to E – halfway between $\mu\perp$ and $\mu\|$

Initially we tried to measure the differential EXAFS signal on the sample without the DAC. This turned out to be impossible, due to the difficulty in fixing the sample rigidly enough so that it would not move as the field rotated around it. We then passed to the sample in the DAC.

We used a pair of 320 μm culet, 1.5 mm thick diamonds. We started with a CuBe gasket indented to 55 μm and with a 150 μm hole. Silicone oil was used as pressure transmitting media. The pressure was measured using the ruby fluorescence method.

We first performed 6 series of acquisitions at ambient P (XFeCo_12 – XFeCo_17), shown in Figure 2 together with a spectrum of FeCo taken out of the DAC. The differential signal obtained through the DAC is identical (in shape and intensity) to that obtained in previous runs on the same sample out of the cell. The maximum P-P amplitude (around pixel 800) is about $5\ 10^{-4}$.

We then increased the pressure very slightly to 0.055 GPa and performed 3 series of acquisitions (XFeCo_18 – XFeCo_20). The signal was equivalent, with a slightly smaller amplitude ($4.5\ 10^{-4}$). When we tried to increase the pressure further, the gasket hole closed, crushing the sample. No differential signal could be measured (XFeCo_21-22). When pressure was released, the signal appeared again (XFeCo_23) but the noise level was high. Air had penetrated within the sample chamber, and the sample moved with the magnetic field.

We performed a second loading with a CuBe gasket indented to 20 μm (same hole diameter). The pressure rose rapidly to 7.5 ± 0.5 GPa, but the gasket hole closed in again and the sample was crushed. Differential EXAFS scans (XFeCo_25-30) show no signal, or a very weak one, within the noise level.

We then tried to use different gaskets (non-magnetic stainless steel and Rhenium). The same effect of hole closing in occurred. This was a clear indication that the problem was not to be attributed to the softness of the gasket metal, but rather to the charging method (the sample chamber not leak tight) or to an intrinsic problem of the cell (misalignment of the diamonds).

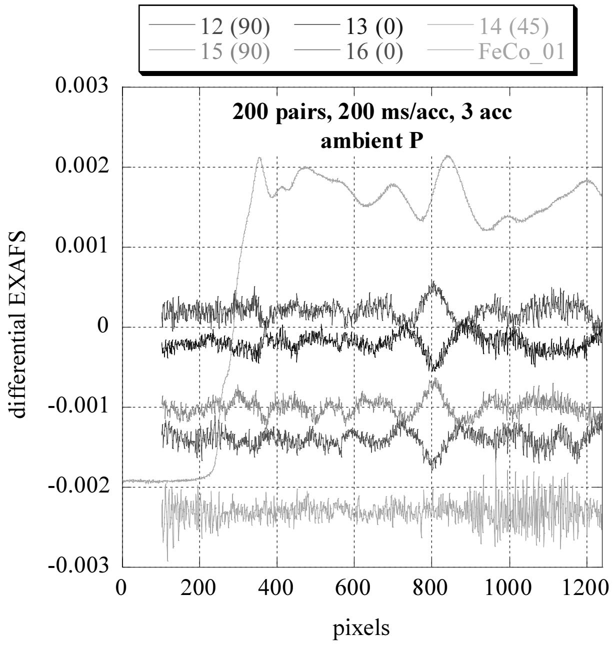

Figure 2 : Ambient P measurements on the first loading.

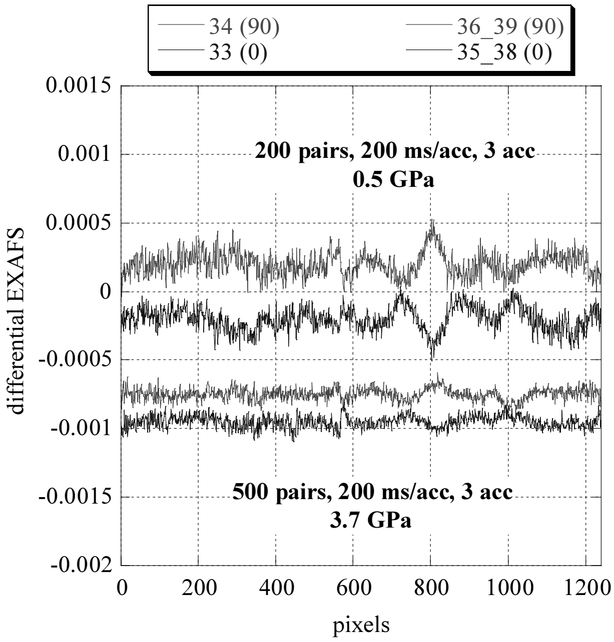

Figure 3 : Differential EXAFS at P=0.5 GPa and P=3.7 GPa on the third loading.

So we went back to CuBe gaskets and used a different sample loading procedure(third loading). This time the hole did not close in and we performed measurements at 0.50 ± 0.05 GPa (XFeCo_33-34) and at 3.7 ± 0.2 GPa (XFeCo_35-39), shown in Figure 3. A clear reduction of the amplitude is observed as pressure is increased, the signal at 0.5 GPa and 3.7 GPa being respectively of about 3.5 10^{-4} and 1.0 10^{-4}. The reduction in amplitude of the differential EXAFS as function of pressure is shown in Figure 4, while absorption spectra as a function of pressure up to 7.5 GPa are shown in Figure 5 (a big diamond glitch is present below the edge on FeCo_08). The ambient pressure bcc structure is preserved in this pressure range, and the reduction in frequency of the oscillations indicates that the lattice contracts uniformly.

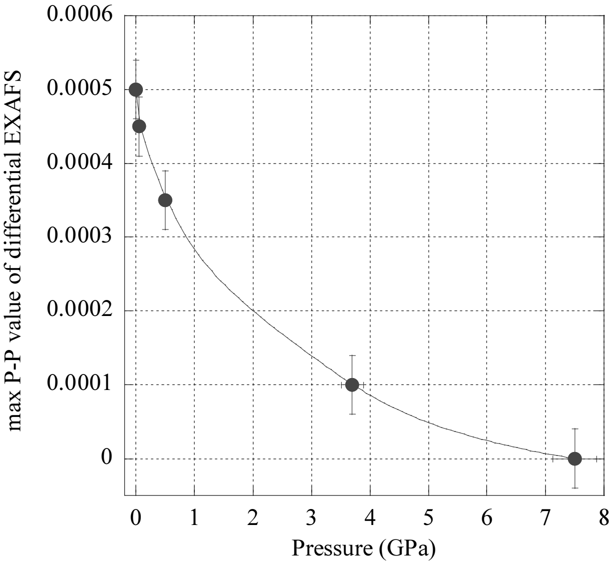

Figure 4: Variation of amplitude of differential EXAFS as a function of P.

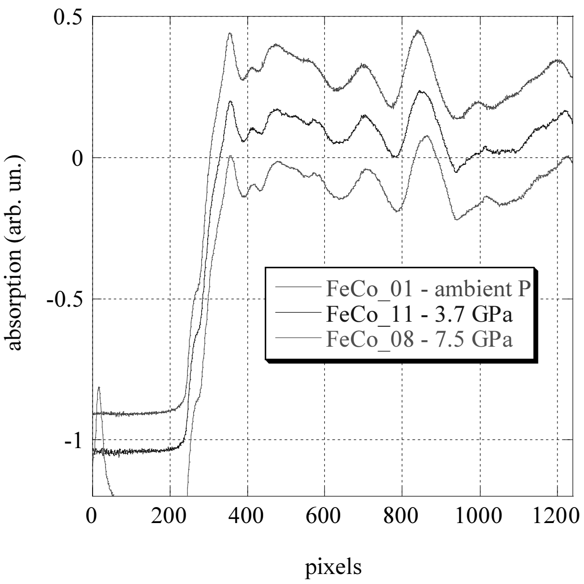

Figure 5: Fe K edge absorption spectra as a function of P.

We have not unloaded the DAC in order to acquire XRD patterns from this sample (at 3.7 GPa), that exhibits a reduction of the differential EXAFS of about a factor 5 with respect to the ambient pressure value. By comparing the XRD patterns of this sample with an ambient pressure one, we will know whether this reduction has to be attributed to a lower degree of preferential orientation or strain, or to some other origin.