



	Experiment title: Differential EXAFS and Magnetostrictive properties of FeCoB alloy	Experiment number: HD-44
Beamline:	Date of experiment: from: 13 April 2006 to: 18 April 2006	Date of report: 27 February 2007
Shifts:	Local contact(s): Sakura Pascarelli	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): *Javier Diaz, *Carlos Quiros, *Diego Martinez Universidad de Oviedo Robert Pettiffer		

REPORT:

Abstract

The magnetostrictive properties of $(\text{Fe}_{80}\text{Co}_{20})_{80}\text{B}_{20}$ films deposited by magnetron sputtering were investigated by differential EXAFS. Their EXAFS spectra showed that they were polycrystalline, using up to three shells in the fits. Fe and Co were arranged in a bcc structure. However, bond lengths were sensitively longer in Fe. Coordination was 20% higher in Co than in Fe. TM-B environments were not needed to fit the spectra. This suggests that B was preferentially bonded to Fe, and that it formed an amorphous structure with undetectable contribution to EXAFS. Differential EXAFS (DEXAFS) observed atom displacements induced by intense applied magnetic fields in the Fe K edge only. The bond length between Fe-Fe atoms that yielded such atom displacements was 2.59 (1) Å, 0.10(1) Å larger than the bond length measured to first neighbors by dispersive EXAFS. Fe-Fe displacements were of about 350 ppm, i. e., their magnetostriction coefficient was of 134 ppm.

Experiment

$(\text{Fe}_{80}\text{Co}_{20})_{80}\text{B}_{20}$ films 6 µm thick were prepared by magnetron sputtering, with an evaporation rate of 46 nm/min (8 Å/s). The films measured were those peeled off from the sample holder in the preparation chamber. They were encapsulated in a specially designed sample holder for differential EXAFS. To reduce external strain, the film was fix to it with a bit of vacuum grease.

EXAFS spectra simulation was done using the FEFF8 code. Background subtraction and fit of the EXAFS spectra was done using the IFEFFIT application.

The differential EXAFS spectra were fit in k space. The fitting functions were the EXAFS contribution for each scattering path (χ) differentiated with respect to R with the parameters deduced from FEFF8. The fit of the differential spectra was done using the application "Igor Wavemetrics". This allowed varying the coefficients of the

differentiated χ paths, proportional to the magnetostriction coefficients, and other parameters like interatomic distances and Debye-Waller factors.

Results

EXAFS spectra

Figure 1 shows that the absorbed intensity of Fe and Co at their respective K edges was consistent with their relative concentration in the alloy.

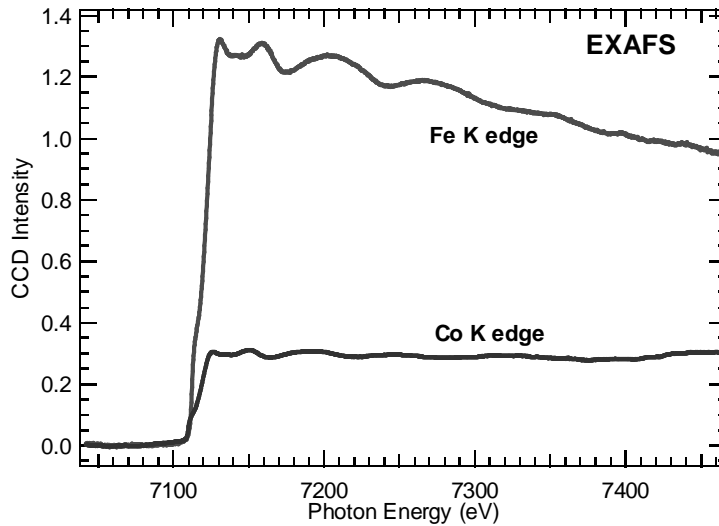


Figure 1. EXAFS spectra at the Fe K edge and Co K edge. Raw data without normalization. (Energy scale for Co edge was downward shifted 700 eV)

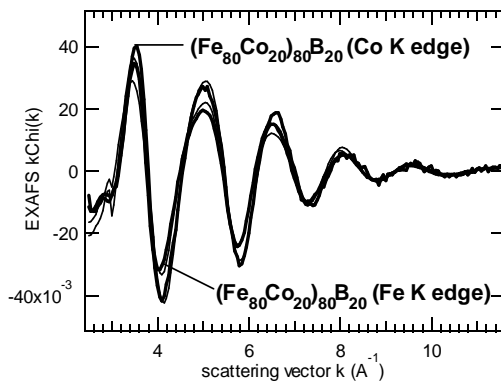


Figure 2. EXAFS spectra in k-space of the FeCoB film at the Co and Fe K edges. Thin line corresponds to the respective fit to the spectra.

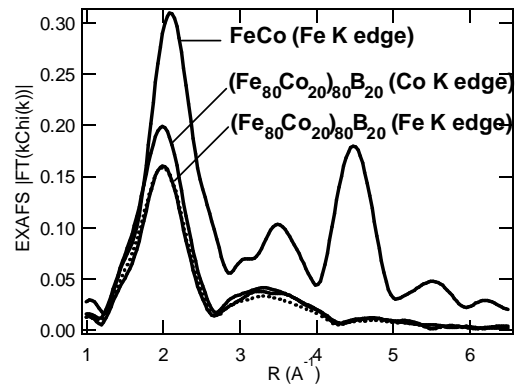


Figure 3. EXAFS spectra in R space of the FeCoB film at the Co and Fe K edge, and of an FeCo polycrystalline film taken at the Fe K edge. Dotted line is the FeCoB spectrum multiplied by 0.8.

Figure 2 and 3 shows EXAFS spectra of the same films taken in BM29. EXAFS amplitude was 20% higher in Co than in Fe. The same figures show the fit of the spectrum in the Fe K-edge. A FeCo polycrystalline film was used as a reference spectra to obtain the S_0^2 factor, which was 0.8. Three shells related to a bcc lattice were used in the fits of the FeCoB films. The free parameters were the Debye-Waller factor and radius of each shell. Coordination numbers were initially fixed to those of the bcc lattice ($N_1=8, N_2=6$ and $N_3=12$).

The TM-B environment was not detected in the fits, indicating that its contribution to the EXAFS spectra was small. This was probably due to disorder, and the low scattering cross section of B. The loss in amplitude in the spectra taken at the Fe K edge with respect to that at the Co K edge should be related to those undetected atom environments. The loss in amplitude was of 20%, equivalent to the B concentration in the films. Then, Fe would be preferentially bonded to B, as it has been already observed [1]. The amplitude of the total EXAFS oscillations after normalization of the spectra to the intensity of the jump at the Fe K edge should be lower than if Fe were bonded only to other TM atoms, since Fe atoms bonded to B participate in the absorbed intensity at the Fe K edge but no in the EXAFS oscillations.

The spectra analysis yield sensitively longer bond lengths for Fe than for Co: first neighbours were at 2.47 (0.01) Å in Co and at 2.49 (0.01) Å in Fe.

Differential EXAFS analysis

Figure 4 shows the differential spectra of the FeCoB alloy at the Fe K edge (3 spectra) and at the Co K edge (1 spectrum). Its energy scale was shifted downwards in energy 600 eV). The spectrum obtained flipping the magnets 180° each turn was added to the figure.

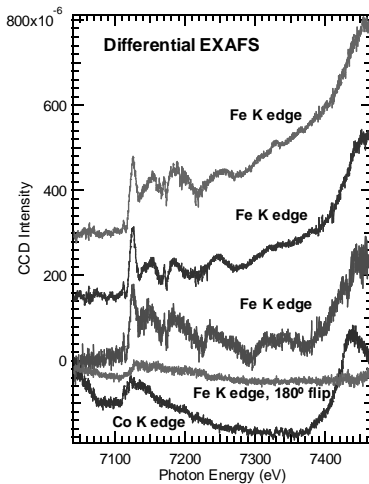


Figure 4. DiEXAFS spectra of the FeCoB alloy at the Fe and Co K edges. Co K edge spectrum was shifted downwards in energy 600 eV.

Oscillations are clearly visible in the Fe K edge but they are almost negligible in the Co K edge. This could be due to the 4 times lower concentration of Co than Fe in the alloy. Figure 5 compares the DiEXAFS obtained at the Co K edge with that obtained at the Fe K edge divided its intensity by 4. The noise level is similar in both spectra. DiEXAFS oscillations are still visible at the Fe K edge, indicating that if the amplitude of the same oscillations was similar in Co they should be seen. This might suggest that Co atoms remain fixed under the externally applied fields, i.e., only pairs of Fe atoms participate in the magnetostriction effect.

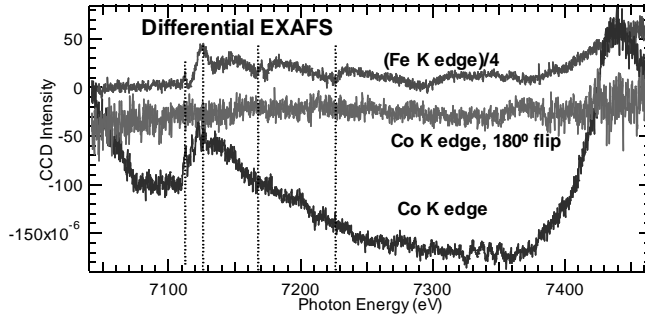


Figure 5. DiEXAFS of the FeCoB alloy at the Fe K edge divided in intensity by 4 (red), and at the Co K edge (energy 600 eV downward shifted) obtained with 90° magnets flip (dark red) and 180° magnets flip (blue).

All the DiEXAFS spectra at the Co and Fe K edges seem to have a jump at the edge that decreases with increasing photon energy. Such a jump in intensity does not appear in the Co edge when magnets were flipped 180° each turn (see figure 5). It appears, but with a lower intensity, in the Fe K edge (see figure 4). Therefore, it does not seem to be an artefact but something real. It might be attributed to changes in the electronic structure due to the magnetic field. Published magnetic linear dichroism observation at a TM K edge are very rare due to its extraordinary small intensity, which it has been reported to be of the order of 0.0006 times the intensity at the edge [2].

Figure 6 displays the three Fe K edge DiEXAFS spectra displayed in figure 4 in k-space after background subtraction using the AUTOBK application. Figure 7 compares one of these spectra with the EXAFS spectra of the alloy in R space.

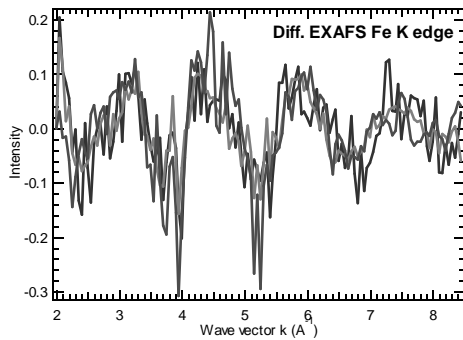


Figure 6. DiEXAFS spectra of the FeCoB alloy at the Fe k edge in k-space.

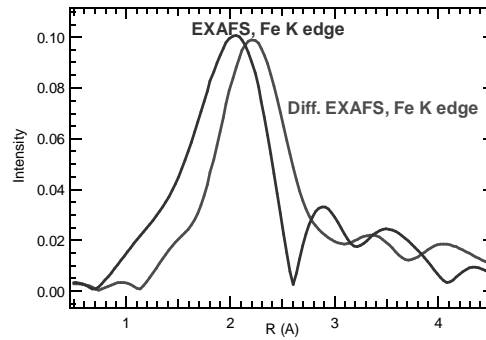


Figure 7. DiEXAFS spectrum of the FeCoB Alloy at the Fe k edge Compared to its corresponding EXAFS spectrum.

The DiEXAFS spectrum is clearly shifted to a higher bond length respect to its EXAFS spectrum. That is what it was observed in the fits: the spectra were better fitted if the length of the first shell was 0.10(1) Å larger than the observed in the EXAFS spectra. Moreover, the DiEXAFS spectra were better fitted using one single shell (first neighbors) and not three as in the case of the EXAFS spectra. Figures 8, 9, 10 and 11 show the fit with different constraints. In Figure 8, three shells were used for the fits, the same as the used in EXAFS. The first and second shells correspond, respectively, to the 111 and 100 directions in the bcc crystal. The third shell was a linear combination of these two shells. Its coefficients were 1.25 in the 111 direction and 1.5 in the 100 direction. It was assumed that the magnetostriction coefficients λ_{111} and λ_{100} were different. Then, the trial fitting function was:

$$\begin{aligned}\chi'_{\text{tot}} &= \alpha_{111} \chi'_{111} + \alpha_{100} \chi'_{100} + (1.25\alpha_{111} + 1.5\alpha_{100})\chi'_3 \\ &= a(R_{111}\lambda_{111}\chi'_{111} + R_{100}\lambda_{100}\chi'_{100} + (1.25R_{111}\lambda_{111} + 1.5R_{100}\lambda_{100}))\chi'_3\end{aligned}$$

α_{111} and α_{100} were the parameters to vary in the fit. The parameter a is equivalent to 1 when the amplitude of the DEXAFS spectra is related to the amplitude of the corresponding EXAFS spectra measured in the same conditions, i.e., the spectra shown in figure 1. Figure 8 shows the trial function with $\lambda_{111}=0.5\cdot\lambda_{100}$, which is similar to the expected ratio between the magnetostriction coefficients in FeCo crystal.

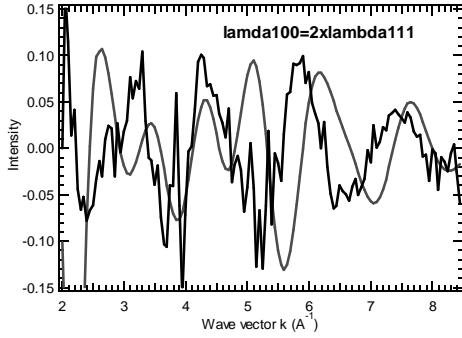


Figure 8. DiEXAFS spectrum at the Fe K edge (black) and the trial function using similar parameters than the used in EXAFS and considering that $\lambda_{111}=0.5\cdot\lambda_{100}$.

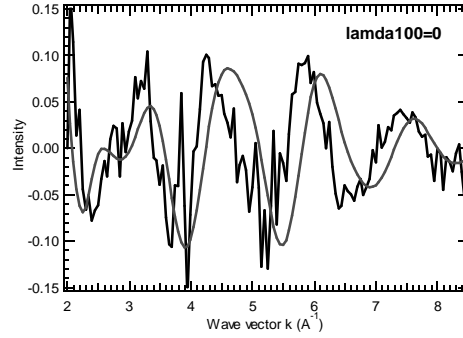


Figure 9. DiEXAFS spectrum at the Fe K edge (black) and the fitted function using similar the trial function of figure 8. λ_{100} was 0 after the fit.

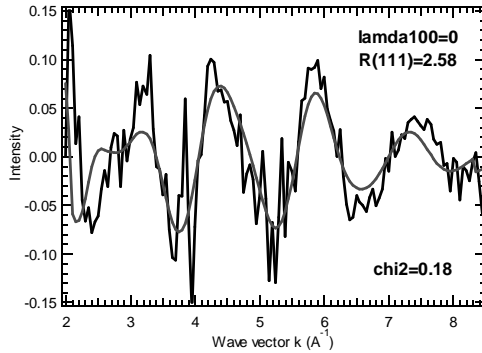


Figure 10. DiEXAFS spectrum at the Fe K edge (black) and the fitted function using the first and third shells, and changing their respective scattering lengths.

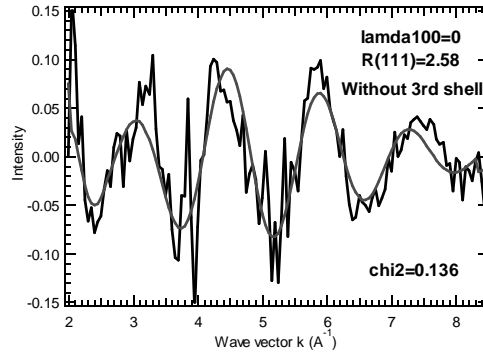


Figure 11. DiEXAFS spectrum at the Fe K edge (black) and the fitted function using only the first shell.

Figure 9 shows the fit after fitting the DiEXAFS spectra using the trial function of figure 8. The result is that α_{100} goes to zero. Figure 10 shows that the fit improves if the lengths of the first and third shells are varied in the fit. Finally, figure 11 shows the best fit which was obtained using only the first shell and changing the bond length from 2.49 Å in EXAFS to 2.59(1) Å in DiEXAFS.

Only the atomic environments involved in the magnetostriction phenomena can give a DiEXAFS signal. Therefore, Fe-Co or Co-Co environments did not contribute to magnetostriction. Only a part of the Fe atoms whose Fe-Fe bond lengths were 2.59 Å participated. The longer bond lengths observed in the EXAFS Fe K edge respect to the

obtained in the Co K edge could be related to those environments. To detect them, the fit of the EXAFS spectra at the Fe K edge was done imposing the following conditions:

- (1) shell coordination was reduced in 20%, i.e., it was assumed that all B were bonded to B and contributed with an undetectable intensity to EXAFS.
- (2) The first shell bond length was fixed to that of Co and another shell was added with its bond length fixed to 2.59 Å, and its Debye-Waller factor equivalent to that of the first shell.

The coordination of the added first shell resulted to be 23% the coordination of the initial first shell, i.e., the proportion of the environments with a bond length of 2.59 Å was similar to the B concentration in this films. Nevertheless, the Debye-Waller factors were too large (0.02 Å^2) to consider this result as definitive.

The Fe-Fe environments with a bond length of 2.59 Å could be those that had one of their Fe atoms bonded to B. The Fe-B bond length (2.1 Å after reference [1]) is smaller than the Fe-Fe bond. Their bonding type and charge transference should be also different. Both facts might cause the Fe-Fe bond length of the Fe bonded to B to be substantially longer.

The magnetostriction coefficient resulted from the fits of the DEXAFS spectra was 134 ppm. If the proportion of Fe-Fe environments was equivalent to the B relative concentration (20%), the resulting macroscopic magnetostriction coefficient should be $134 \times 0.2 = 27 \text{ ppm}$ which is very close to 30 ppm, the value measured in these films by other techniques.

Conclusions

EXAFS analysis showed that Co and Fe atoms were in a disordered bcc lattice. The films were not totally amorphous but likely formed by small crystallites since their diffraction data did not resolved any diffracted peak. No TM-B environments were detected. They might be segregated from the FeCo crystallites forming an amorphous structure in between the grains. B was probably bonded to Fe because the known affinity of B to Fe compared to Co [1] and because the EXAFS amplitude was 20% smaller in the Fe K edge than in the Co K edge, the same proportion than the relative concentration of B in the films. The length of the first shell was longer ($0.02(1) \text{ Å}$) in the Fe K edge than in the Co K edge.

DiEXAFS oscillations were observed only in the Fe K edge. They were missing in the Co K edge, indicating that Co-Co and Co-Fe environments were not involved in the magnetostriction effect. All the DiEXAFS spectra showed a jump in intensity at the edge, which seemed to be related to a linear magnetic dichroism.

The fit of the DiEXAFS in the K edge was done using a single shell. The bond length was larger ($0.10(1) \text{ Å}$) than the obtained in EXAFS. The magnetostriction coefficient obtained from the fits was 27 ppm, similar to the measured in these films (about 30 ppm).

[1] M. L. Fdez-Gubieda, I. Orúe, F. Plazaola, and J. M. Barandiarán. Phys. Rev. B 53 (1996) 620

[2] K. Sato, Y. Ueji, K. Okitsu, T. Matsushita, J. Saito, T. Takayama, and Y. Amemiya. Phys. Rev. B 65 (2002) 134408