

Experimental report on Proposals ID : 20130096 (FAME-BM30B)

Anomalous structural distortion in mass-selected FeCo nanoalloys from X-ray chemical local probe

Scientific Background

Structural distortion of FeCo alloys in chemically ordered B2 phases is theoretically expected to lead to giant magnetic anisotropy energy (MAE) and large saturation magnetic moment as required for future recording media at room temperature. The aim of the present proposal is to observe for the first time local order and structural distortion in FeCo nanoalloys. To reach this purpose, X-ray absorption spectroscopy (XAS) experiments at both Fe and Co K-edges, has been performed on FeCo mass-selected nanoparticles of different diameters ranging between 2-6 nm. On one hand, the chemical ordering has been promoted by high temperature annealing in vacuum without coalescence of such bimetallic clusters assemblies embedded in amorphous carbon matrix. On the other hand, the tetragonal structural distortion has been tuned by taking a profit of finite size effect and specific relaxation previously evidenced in our FePt and CoPt clusters.

Sample preparation and Characterization

The possibility to control size, composition and host element offers a wide possibility of fundamental studies in these nanoparticle assembled films. We have measured FeCo bimetallic nanoparticles embedded in matrices with a weak influence on the interface magnetic anisotropy (Carbon) in order to preserve and investigate the intrinsic cluster surface properties. Using MS-LECBD coupled to an electrostatic quadrupole deflector, we have been able to synthesize mass selected bimetallic nanoparticles embedded in an amorphous carbon matrix. The samples are prepared from nanoparticles pre-formed in the gas phase and then co-deposited in UHV with the matrix. Diluted (~ 1% of nanoparticles in volume) nanoparticles assembled films, with quite negligible magnetic interaction among nanoparticles, are obtained by adjusting independently the deposition rate of the nanoparticle and matrix beams. The relative size dispersion, which is around 40% without mass selection, has been lowered to about 7% with the mass selection.

We prepared six different FeCo samples, three were left as-prepared and three were annealed at 500°C under vacuum. The samples were prepared as couples each of a specific size (150V, 300V, and 600V). The average diameter of the nanoclusters is varied by changing the applied voltage on the quadrupole during sample preparation ($m \propto qV$); thus changing the selection voltage we select a specific size. In addition, we prepared four reference samples of mass selected pure Fe clusters of two different sizes (150V and 300V) two as-prepared and two annealed. The same was prepared for the pure Co reference clusters.

Experimental results

On our allocated beamtime we were able to systematically pass all our samples annealed and as-prepared. The beamtime was well organised. The FeCo samples were all passed at both K-edges (Fe and Co) for all the different samples annealed and as-prepared. The control samples (Fe and Co) were passed at their respective K-edges for all sizes, annealed and as-prepared. In addition, we managed FeAu samples embedded in carbon and another in MgO, as well as, FePt embedded in carbon at the Fe K-edge. Moreover, we also succeeded in measuring Co nanoparticle sample embedded in gold, and two CoAu binary alloy samples one embedded in carbon and the other in LiF at the Co K-edge.

The idea was to study separately the finite cluster size effects (size selection), as well as thermal treatment (annealing compared to as-prepared). A systematic study is underway on the FeCo system.

We are in the process of fitting the rest of the data we obtained. From the preliminary fitting of the as-prepared FeCo samples of all sizes, we seem to have a tendency of obtaining a Co rich core with a Fe-C shell. From a qualitative analysis of the radial distribution of the FeCo 600V samples (annealed and as-prepared) at both Fe and Co K-edges, we observe an evolution after annealing to a BCC structure (Figure 1). The best fit at the Co K-edge for the annealed sample (Figure 2) was achieved by using a B2 phase BCC structure. The fit was performed up to 5Å (3rd nearest neighbour).

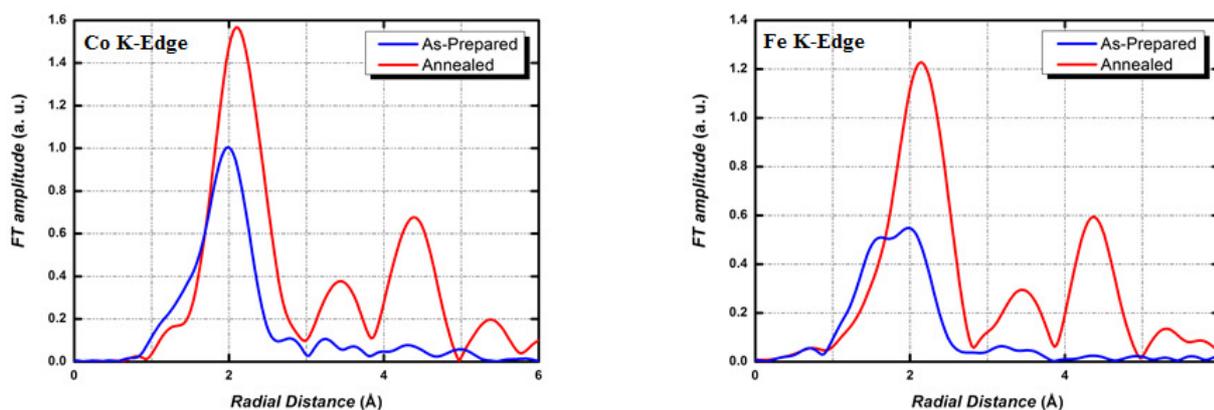


Figure 1 - Radial distribution of as-prepared (blue) and annealed (red) EXAFS signal at the Co K-edge (left) and the Fe K-edge (right).

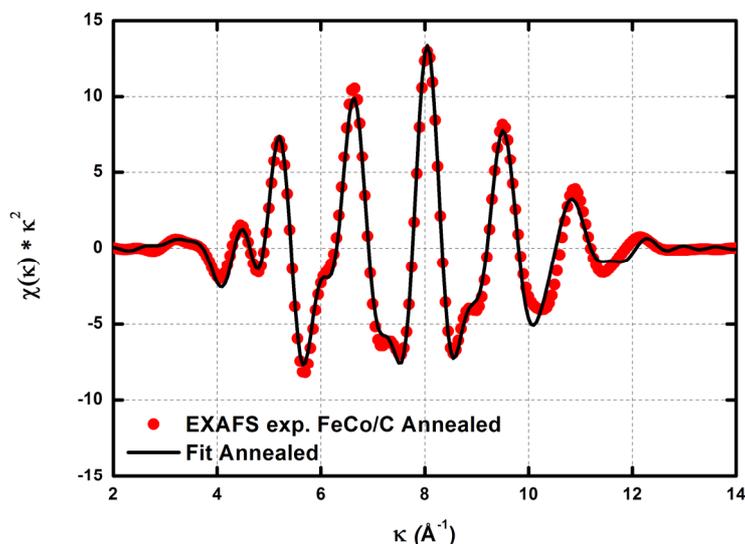


Figure 2 - EXAFS oscillations for FeCo 600V annealed at the Co K-edge (red spots) with best Fit (black line).