

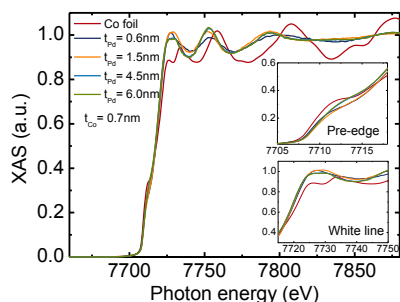


	<b>Experiment title:</b> EXAFS study of Pd-Co alloys in metal-capped Co nanoparticles	<b>Experiment number:</b> HE-3229
<b>Beamline:</b> BM29	<b>Date of experiment:</b> from: December 16 <sup>th</sup> , 2009 to: December 21 <sup>st</sup> , 2009	<b>Date of report:</b> Sept. 28 <sup>th</sup> , 2010
<b>Shifts:</b> 12	<b>Local contact(s):</b> Dr. Matthieu CHORRO	Received at ESRF:
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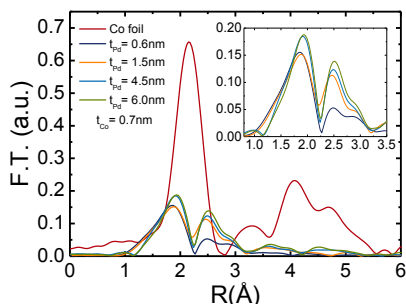
Experiment HE-3229 was performed with the aim of completing the structural analysis of  $(Al_2O_3/Co/M)_N$  multilayer samples  $M = Pt, W$  and  $Pd$  in order to correlate their magnetic properties with their nano-structure. These samples consist of Co nanoparticles with a nearly spherical shape produced by sputtering of Co on amorphous alumina, previously deposited on a Si substrate and subsequently capped with a metallic layer [1]. Structural and magnetic properties of these granular Co systems have been intensely studied by us via conventional SQUID magnetometry, high resolution electron microscopy and synchrotron radiation techniques, such as XMCD and XAFS [2-5]. We have demonstrated that the magnetic properties of these Co nanoparticles are strongly affected by the surrounding matrix so that capping them with a metallic layer modifies magnetic response of the system [3-5]. When capping with noble metals (Cu, Ag and Au) the particles behave as superparamagnetic with anisotropy constants that depend on the metal capping, though, their structural properties remain unaltered by the enclosing noble metal [3].

Instead, capping with W, Pt or Pd has a completely different effect both in the magnetic and structural properties of the particles. In the Pt case, the Co particles are strongly coupled via the polarized Pt [4], and their structure is modified due to the high affinity between Co and Pt, giving rise to the presence of Co-Pt alloys. For the W case, we found evidences of amorphous Co-W alloy in the superparamagnetic Co-W particles, producing a systematic reduction of their saturation magnetization and magnetic anisotropy as the amount of W in the samples increases [5]. Most of this structural characterization in the Pt and W – capped Co nanoparticles was obtained from our last XAFS experiment at the ESRF (HE-2952), where such measurements were performed for samples with formula  $(Al_2O_3/Co/Pt)_{25}$  and  $(Al_2O_3/Co/W)_{25}$  with nominal thickness of Pt and W being  $t_{Pt} = 0.6, 1.5, 4.5$  and  $6.0$  nm and  $t_W = 0.6, 1.5,$  and  $4.5$  nm.

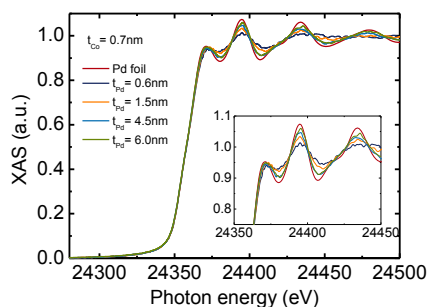
By performing experiment HE-3229 we have completed our XAFS study in this set of Metal – capped Co – nanoparticles samples. Samples with formula  $(Al_2O_3/Co/Pd)_{25}$  with  $t_{Pd} = 0.6, 1.5, 4.5$  and  $6.0$  nm were measured at the Co K and Pd K edges. Reference samples were also measured at respective edges: CoPd, CoPd<sub>3</sub> and Co<sub>3</sub>Pd alloys measured at the Co K and Pd L<sub>3</sub> edges, cobalt film measured at the Co K edge and a palladium film measured at the Pd L<sub>3</sub> edge. Thickness of the Co and Pd foils was of 5µm. For each edge measured on each sample, four spectra were taken in order to have enough statistics in data treatment, following a 4s/point count.



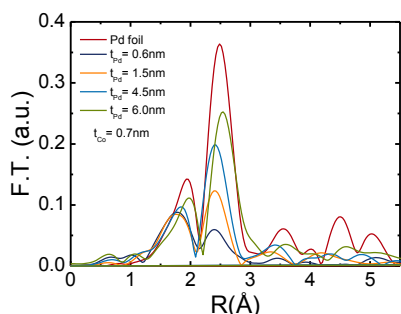
**Fig. 1.** XANES at the Co K edge on different Pd capped Co – NPs with a constant  $t_{Co} = 0.7$  nm and comparison with a Co Foil. Insets: Zoom of the pre-edge and white-line zones.



**Fig. 2.** FT of EXAFS signal at the Co K edge on the samples shown in Fig. 1. Inset: Zoom of the first two coordination shells in the NPs.



**Fig. 3.** XANES at the Pd K edge on different Pd capped Co – NPs with a constant  $t_{Co} = 0.7$  nm and comparison with a Pd Foil. Inset: Zoom of the white-line zone.



**Fig. 4.** FT of EXAFS signal at the Pd K edge on the samples shown in Fig. 3.

## References

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Some representative scans obtained from this experiment are shown in Fig. 1 and 2 for measurements at the Co K edge, and Fig. 3 and 4 for the Pd K edge. In Fig. 1 we compare spectra at the Co K XANES region of the Co-Pd nanoparticles samples with the one recorded for the Co foil. Reduction of the amplitude of the pre-edge line and enhancement of the white line in the nanoparticles systems, as compared to the one in the Co foil, are observed (see insets in Fig 1). These features are evidence of a change in the electronic structure in the particulate systems, possibly given by existence of Co-Pd alloy.

Changes in the extended region (EXAFS) at the Co K edge are also evidenced in Fig. 1, showing further structural changes in the particulate systems. To better illustrate such variations, the Fourier Transform of the EXAFS signal at Co K edge for each sample is shown in Fig 2. We found the expected decrease in amplitude given by reduction of the size scale from the Co bulk to the nanoparticles system. The Co environment in all Co-Pd nanoparticles samples has significantly changed compared to the one in metallic Co. Peak positions and shape of the first two coordination shells in the Co-Pd nanoparticles (see Fig. 2 inset) are close to those found in the alloy-like Co environment identified for Co/Pd multilayer films in Ref. [6].

Pd K edge spectra recorded for the Co-Pd nanoparticles and the Pd foil are shown in Fig. 3. Some changes in the amplitude at the XANES and EXAFS regions of the scan in the particulate systems with respect to the Pd reference foil are identified. A systematic reduction of the Pd signal in the nanoparticles compared to the one of the foil is observed, following a consistent trend according to the decreasing amount of Pd present in the sample.

Fig. 4 shows the FT of the EXAFS signal at the Pd K edge. The systematic decrease in the signal as the amount of Pd is reduced in the sample is also observed in this R-space plot. Changes in the Pd environment in the Co-Pd nanoparticles compared to the Pd foil are less pronounced than those identified in the measurements at the Co K edge and are more evident in the first coordination shell.

Quantitative analysis of these EXAFS measurements is still in progress.

The proposal program was fully accomplished during the experiment.