ESRF	**Experiment title:** Investigation of the structure of iron-carbide/iron-oxide core-shell nanoparticles with additional carbon shells for magnetic hyperthermia	**Experiment number:** **MD-1068**
Beamline: BM23	**Date of experiment:** from: 25 Oct 2017 to: 28 Oct 2017	**Date of report:** 27 Feb 2018
Shifts: 9	**Local contact(s):** Olivier Mathon (email: mathon@esrf.fr)	*Received at ESRF:*

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Report:

The two series of iron/iron oxide (R-samples) and iron/iron carbide (A-samples) nanopartilces with additional carbon shell were synthesized and studied by XAS (XANES and EXAFS) experiemnts at BM23. The variation of conditions of pyrolysis should allow to vary the ratio of iron oxide (carbide) and metal iron in the nanoparticles. Indeed, the spectra show distinguishable differencies between samples, as it illustrated on Figures 1 and 2. However, the changes are not monotonic. The fourier-transforms of EXAFS functions are not monotonicaly either, but this can be caused by interference between contributions from different states of iron. The evidence can be shown by simple FT analysis of EXAFS using two scattering contribtions Fe-light atom (O or C) and Fe-Fe presented in Tables 1 and 2.

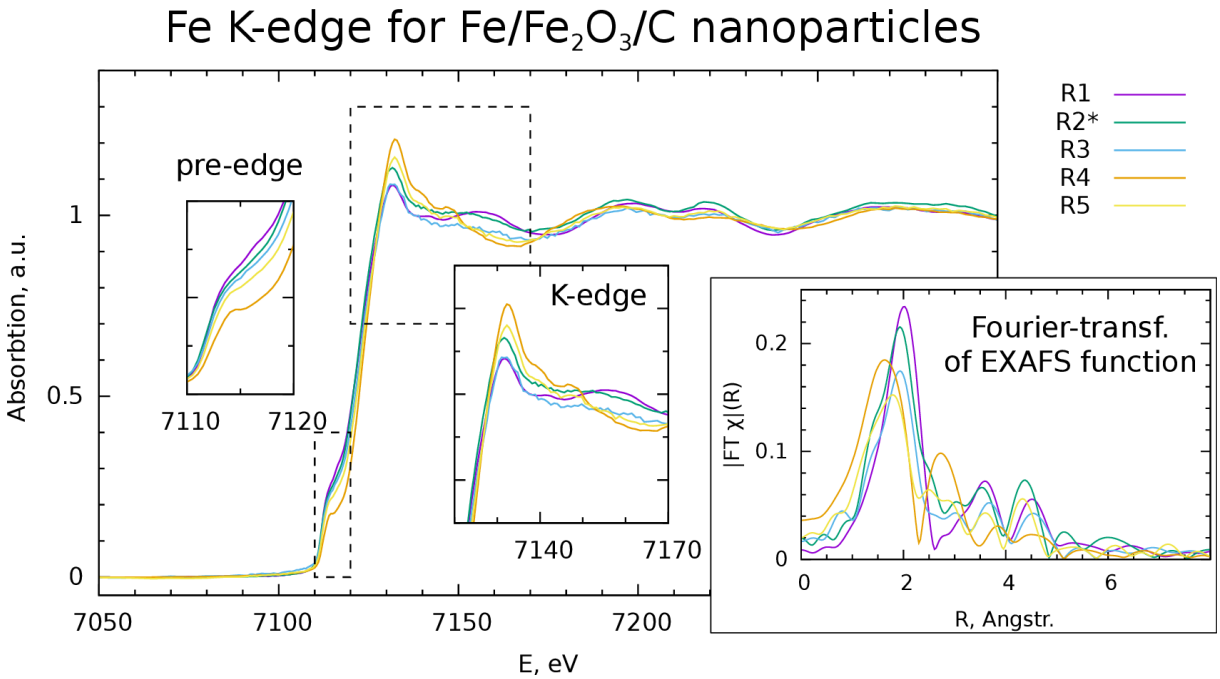

Figure 1. Measured experimental spectra of iron/iron oxide (R-samples) nanoparticles.

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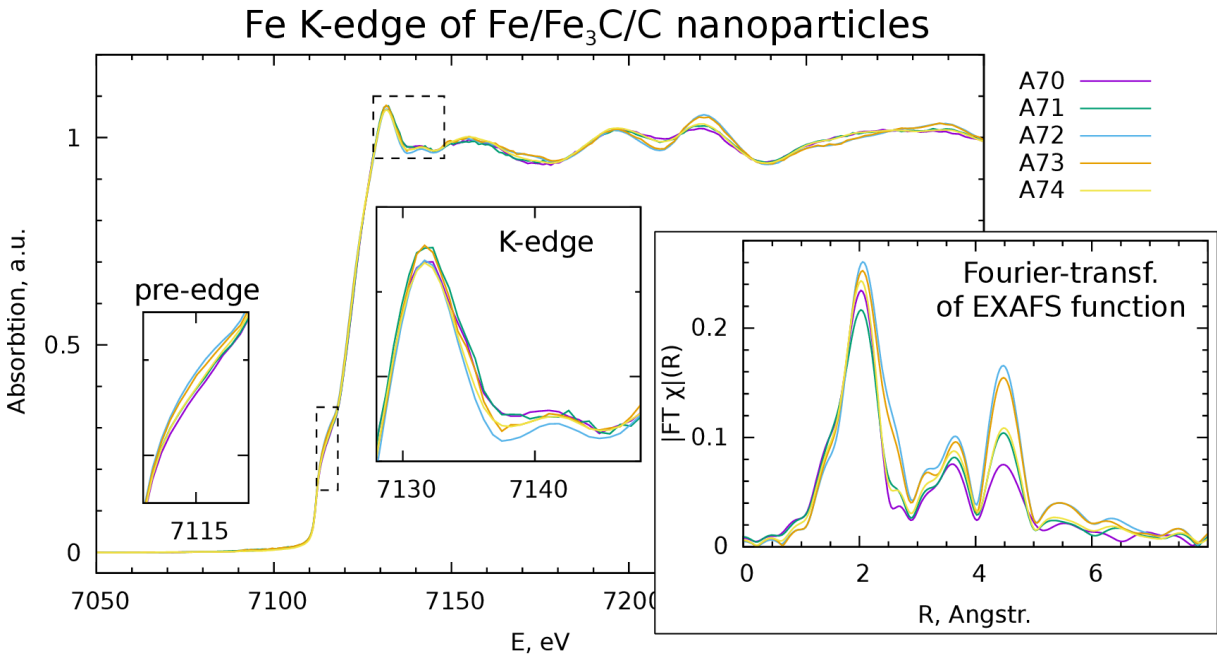

Figure 2. Measured experimental spectra of iron/iron carbide (A-samples) nanoparticles.

Table 1. Results of fitting of EXAFS of iron/iron oxide nanoparticles by two shell fit model

Sample	N(Fe-O)	R(Fe-O)	σ^2(Fe-O)	N(Fe-Fe)	R(Fe-Fe)	σ^2(Fe-Fe)	χ_υ^2
R1 (Fe/C)	1.0	1.84	0.0040	7.4	2.45	0.0078	600
R3	1.3	1.81	0.0040	4.3	2.42	0.0047	100
R4	2.3	2.01	0.0084	5.1	2.58	0.0040	700
R5	3.7	3.2	0.0040	3.7	2.62	0.0063	60
R2* (Fe_2O_3/C)	5.1	1.92	0.0130	5.1	2.49	0.0040	200

Table 2. Results of fitting of EXAFS of iron/iron carbide nanoparticles by two shell fit model

Sample	N(Fe-C)	R(Fe-C)	σ^2(Fe-C)	N(Fe-Fe)	R(Fe-Fe)	σ^2(Fe-Fe)	χ_υ^2
A70 (Fe/C)	-	-	-	5.6	2.42	0.0048	400
A71	1.9	1.89	0.0040	4.8	2.44	0.0040	300
A72	4.4	2.00	0.0088	5.7	2.51	0.0040	>999
A73 (Fe_3C/C)	4.7	1.97	0.0105	5.5	2.49	0.0040	>999
A74	1.6	1.88	0.0040	5.3	2.44	0.0040	900

The poor overall fitting quality indicates on the insufficience of used two shell fit model. This model should be improvaed by addition of more distant coordination shells of structures of bulk iron, iron oxide and iron carbides. Nevertheless, the results for the samples R1, R3, A70 and A71 shows their similarity to pure iron nanoparticle, as it was supposed by synthesis. Other samples, obtained as a result of more intensive pyrolisis, demonstrate more complex local atomic structure, which can be understood using more detailed model and using application of complimentary theoretical and experimental techniques.