XAS Investigation of Controllable core@shell Structure in Multicomponent Nanoparticles

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Our goal is to develop a versatile synthesis for the elaboration of metallic multifunctional nanosystems. To control the chemical order inside the intermetallic objects, the proposed approach is to take advantage of the different kinetics of decomposition of the metal precursors. The FeRh witness system was previously studied by EXAFS and XANES. The synthesis using H\textsubscript{2} as the reducing agent induced a core-shell distribution of metals with a well-ordered Rh core surrounded by disordered Fe [1]. On the contrary, adequate precursors reduced by a highly active amine-borane complex induced a much different structure, as evidenced by studies at Fe and Rh edge [2].

The next step is to develop the generality of the synthesis method and cover a magnetic iron core with different protective layers, e.g. gold or bismuth. The obtained materials are difficult to study using conventional methods: large, aggregated objects including the highly absorbing element Bi cannot be accurately studied by TEM. It is also challenging for single-wavelength XRD, or even its WAXS variant better adapted to nanomaterials, to evidence the exact structure involving both elements. EXAFS studies at the Fe K edge and Bi L\textsubscript{III} are thus essential to access both structure and chemical environment of the two species.

Figure 1: Two different preparations of BiFe nanoparticules at Fe K-edge and Bi L\textsubscript{III}-edge with reference foils (divided by two for comparison at Fe K-edge).

Systematic investigation of many samples evidenced several important points: only a limited size or disorder effect could be observed at the Bi edge (left figure), indicating that Bi environment is mostly unchanged for all samples so far. Quite differently, drastic differences could be observed at the Fe edge, most samples falling in one of only two different patterns: a strong EXAFS signal very close to the one produced by bulk iron (right figure, red curve) and a much weaker signal leading to distances displaced to smaller values (green curve), consistent with a different atomic organization. Chemical disorder involving light elements should also be considered for these samples, however free from any oxidation. This unexpected result brings new insights to the chemical route, and new measurements are planned for confirmation.

References