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_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_{III} are thus essential to access both structure and chemical environment of the two species.

Current results give evidence for the successful synthesis of 2 nm large ferromagnetic iron particles, with an organization strongly dependant on the stabilizing ligand (HDA or HMDS). These particles are either isolated (in *HDA-FeBi*) or aggregated in larger clusters (*HMDS-FeBi*). The bimetallic Fe-Bi nature of these large clusters has been clearly established through the chemical analyses of individual clusters performed by both EDS and STEM-EELS. Interestingly these bimetallic nanocomposites proved very resistant to oxidation, specially compared to pure iron particles synthesized under identical conditions [3].

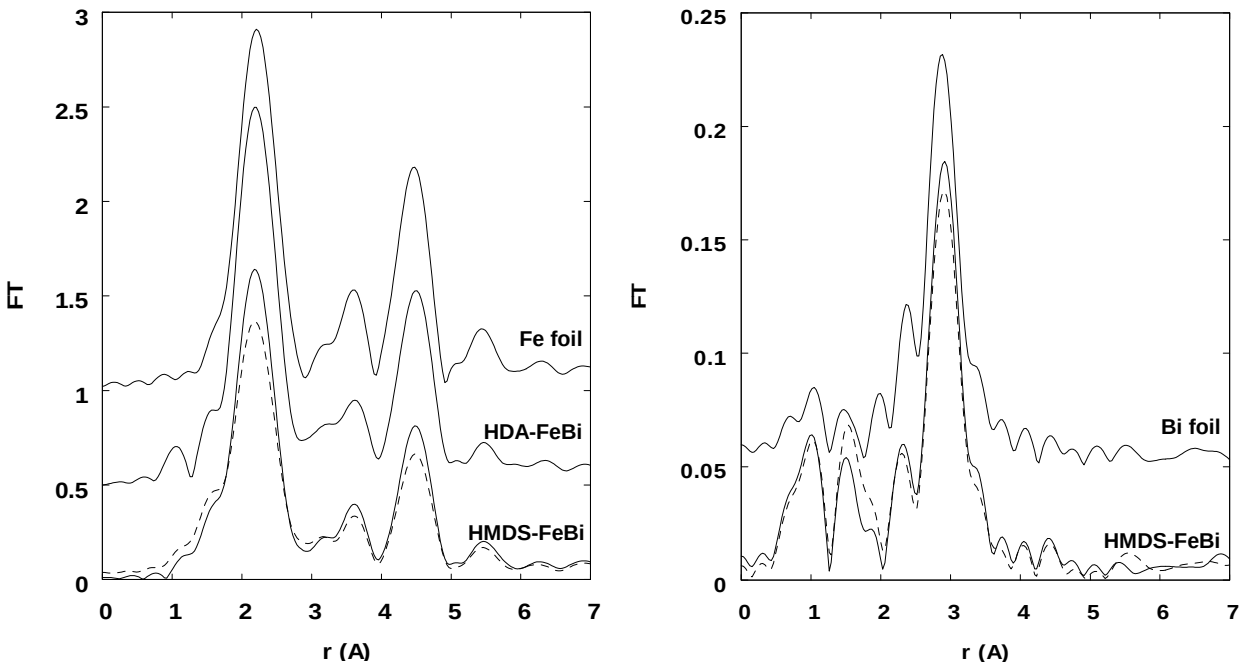

Figure 1: Uncorrected Fourier transforms of the EXAFS functions at Fe K absorption edge (left) and Bi L3 absorption edge (right). From bottom to top: *HMDS-FeBi* before and after exposure to air (solid line / dotted line respectively), *HDA-FeBi*, Fe foil (left), Bi foil (right).

Further measurements on materials synthesized in different conditions of composition and temperature evidence that FeB is first generated, and that its transformation to metallic iron is largely controlled by the quantity of bismuth available. These last results are still under investigation.

References

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