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Determination of the microstructure-property relationship of a magnetic nanomaterial thanks to a coupled laboratory-synchrotron approach

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In the search of new magnetic nanomaterials, organizing magnetic nanoparticles within a solid matrix is of great interest. For that purpose, our group developed an original bottom-up approach, based on coordination and sol-gel chemistry. It consists in the confined growth of Prussian Blue Analogs $A_4[B(CN)_6]_{2.7}$ (A,B=Co,Fe) nanoparticles (Np) within the organized mesoporosity of a silica monolith, which is then calcinated in a controlled atmosphere to transform the PBA in an oxide or an alloy [1,2]. On the one hand, the use of PBA Np as a precursor of the oxide/alloy Np enables a full control of the stoichiometry of the final phase. On the other hand, the use of a mesoporous silica monolith allows for a combined control of the Np size, shape and organization within the matrix and for an organization at the macroscale of the matrix, since surfactant-templated sol-gel chemistry enables to play with the internal organization [3] and external shape of the silica template.

In order to get a complete picture of the relationship between our elaboration process and the final nanocomposite (i.e. the oxide/alloy Np within the silica matrix) properties, we characterized its magnetic properties by SQUID magnetometry, and we systematically compared our synthesis pathway with another one widely used in literature, namely the use of metallic salts as precursors of the oxide/alloy. Despite the combination of powder and Small Angle X-ray Diffraction, IR and UV-Vis spectroscopies, and transmission electron microscopy, the macroscopic information obtained does not always enable to fully understand the properties of the nanocomposites. So we coupled this laboratory investigation with X-ray Absorption Spectroscopy to get fine information at the local scale.

We will present through the example of the Co oxide and metal nanocomposites how this coupled laboratory-synchrotron approach enabled to finely determine the microstructure of the nanocomposite (Figure 1), and hence better understand its microstructure-property relationship [1].

[1] L. Altenschmidt, *Assemblies of magnetic nanoparticles for the development of materials with new properties* , Thèse de l'Université Paris-Saclay (2021)
[2] V. Trannoy, *Vers l'élaboration de pistes magnétiques enregistrables : de la molécule au matériau*, Thèse de l'Université Paris-Saclay (2015)
[3] E. Delahaye et al., Chem. Eur. J. 21, 16906 (2015)

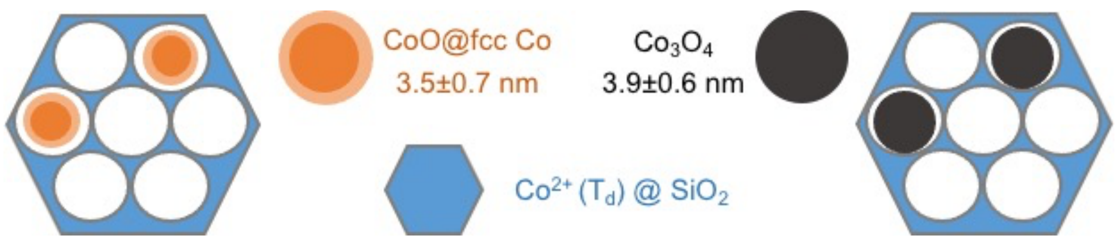

Figure 1 : Microstructure of the *(left)* Co metal and *(right)* Co_3O_4 nanocomposites determined by our coupled laboratory-XAS approach.