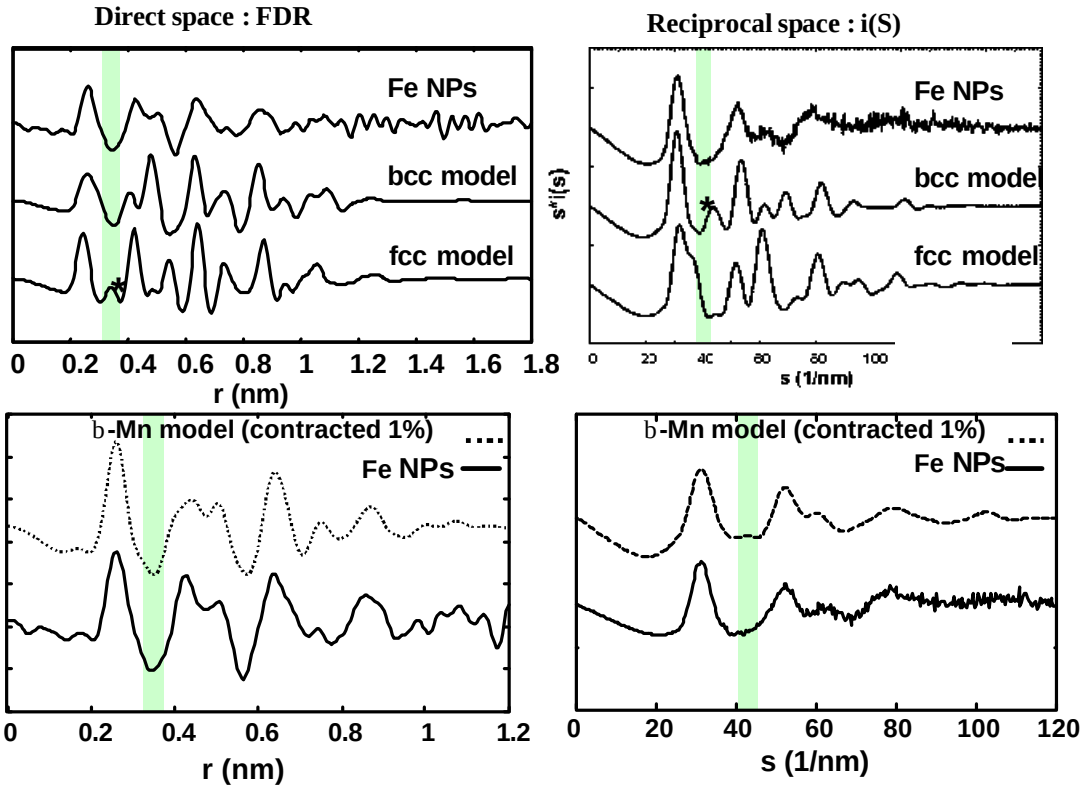
Wide Angle X-Ray Scattering (WAXS)

Nanoparticles (NPs) of very small diameter (<6nm) can be regarded as amorphous materials as the long range crystalline order disappears for such sizes : at small sizes X-Ray diffraction diagrams can't be directly interpreted anymore (peaks are too much enlarged and barely visible above the background signal) but the scattered radiation still contains all structural data. Indeed, irradiation of a sample with an X-Ray of sufficiently small wavelength (e.g. Mo, λ = 0,071069 nm) induces a secondary emission in every direction of space, the intensity of which, I(S), directly depends on the structural signature of the sample, for a given scattering vector S. $S = 4\pi\sin\theta / \lambda$.

To extract interatomic distances from the measurement of I(S), corrections are mandatory: subtraction of air and glass capillary contributions, subtraction of the contribution of the matrix, ligand or polymer, in which the NPs are embedded, correction for absorption and polarisation of incident radiation; a normalisation step then leads to the corrected intensity, I_{corr}. It contains all structural contributions due to interparticle (I_{inter}(S)) and intraparticle (I_{intra}(S)) scattering (and the intrinsic scattering signal corresponding to the chemical composition of the sample (I_{indept} (S)), which can be calculated. The simplest case is that of isolated NPs for which I_{inter}(S) can be neglected. In this case, I_{intra} (S) is called the reduced intensity, i(S), and corresponds to $i(S) = I(S) - I_{indept}(S)$. Conversion into direct space and thus access to the Radial Distribution Function (RDF), i.e. the occurrence diagram of interatomic distances inside the NPs, is achieved by means of a simple Fourier transformation of the i(S) function.

Comparison to RDF and i(S) diagrams generated from model atomic packings enables the identification of the structure of the NPs as shown in the case of iron NPs with diameters 1,8 (+/- 0,5) nm, dispersed in polydimethyl-3,5-phenylenoxide.



Such structural studies bring statistical data which are complementary to TEM and HRTEM studies. They are conducted in collaboration with P. Lecante, CR- CNRS, CEMES, Toulouse.

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