Advanced X-ray Total Scattering Methods based on the Debye Scattering Equation for Characterizing Nanomaterials

Federica Bertolotti[ab*], Daniele Moscheni[b], Antonio Cervellino[c], Norberto Masciocchi[b] Antonietta Guagliardi[d]

[a]*Aarhus Institute of Advanced Studies (AIAS), Aarhus University, Denmark;* [b]*Dipartimento di Scienza e Alta Tecnologia, Università dell'Insubria and To.Sca.Lab, Como, Italy;* [c]*Swiss Light Source, Paul Scherrer Institut, Villigen, Switzerland;* [d]*Istituto di Cristallografia, CNR, and To.Sca.Lab, Como, Italy;* *fbertolotti@aias.au.dk

Nanocrystals (NCs) represent an innovative class of materials showing highly designable and tunable properties. Their size/shape dependent physico-chemical properties provide the opportunity to develop innovative functional materials for an extremely vast range of applications.[1] In spite of this exciting scenario, an exhaustive characterization of NCs is still a challenging task.

In contrast to microscopies, where individual NCs are analysed, scattering techniques allow nanocrystals to be characterized at the atomic and nanometer length scales with high statistical significance.[2]

The very small size of particles in NCs can be considered as a *defective* representation of the corresponding bulk.[3] The extreme downsizing, together with the intrinsic defectiveness, non-stoichiometric composition and dynamic surface result in very broad Bragg peaks in the reciprocal space, a large diffuse scattering and unpredictable peaks shift/splitting. These features make the conventional X-ray diffraction techniques rather inadequate for the structural characterization of nanocrystalline materials.[4] The main advantage of the Total Scattering approaches (both in real[5] and reciprocal space[6]), compared with the standard powder diffraction methods, is determined by their ability to simultaneously model both Bragg and diffuse scattering, extracting all the information hidden in a powder diffraction pattern.

In this talk I will present frontier techniques based on X-ray total scattering and the Debye Scattering Equation approach. This method has been optimized in order to quantitatively provide distribution properties at the atomic and nanometer length scales, such as atomic arrangement, defectiveness of various kinds (vacancies, doping, stacking faults, nanotwins, surface relaxations and ligand-induced distortions), size and shape, morphology, all within a unique homogeneous framework.[7–10]

Experimental and modeling aspects will be presented along with an overview of applications.

[1] G.H. Carey, A.L. Abdelhady, Z. Ning, S.M. Thon, O.M. Bakr, E.H. Sargent, Chem. Rev. 115 (2015) 12732–63.
[2] J. Liu, D. Olds, R. Peng, L. Yu, G.S. Foo, S. Qian, J. Keum, B.S. Guiton, Z. Wu, K. Page, Chem. Mater. 29 (2017) 5591–5604.
[3] Simon J. L. Billinge, Igor Levin, Science 316 (2007) 561–565.
[4] B. Palosz, E. Grzanka, S. Gierlotka, S. Stelmakh, Z Kristallogr. 225 (2010) 588–598.
[5] Takeshi Egami, Simon J. L. Billinge, Underneath the Bragg Peaks. Structural Analysis of Complex Materials, Kidlington, Oxford OX5 1GB, UK, 2003.
[6] F. Bertolotti, D. Moscheni, A. Guagliardi, N. Masciocchi, Eur. J. Inorg. Chem. (2018) DOI: 10.1002/ejic.201800534.
[7] F. Bertolotti, D. Moscheni, A. Migliori, S. Zacchini, A. Cervellino, A. Guagliardi, N. Masciocchi, Acta Cryst. A 72 (2016) 632–644.
[8] F. Bertolotti, D.N. Dirin, M. Ibanez, F. Krumeich, A. Cervellino, R. Frison, O. Voznyy, E.H. Sargent, M.V. Kovalenko, A. Guagliardi, N. Masciocchi, Nat. Mater. 15 (2016) 987–94.
[9] F. Bertolotti, L. Protesescu, M.V. Kovalenko, S. Yakunin, A. Cervellino, S.J.L. Billinge, M.W. Terban, J.S. Pedersen, N. Masciocchi, A. Guagliardi, ACS Nano 11 (2017) 3819–3831.
[10] K.F. Ulbrich, F. Bertolotti, N. Masciocchi, A. Cervellino, A. Guagliardi, C.E.M. Campos, J. Mater. Chem. C 6 (2018) 3047–3057.