UNIVERSITÀ DEGLI STUDI DI TORINO
ALMA UNIVERSITAS TAURINENSIS

CrisDi
Interdepartmental Centre for Crystallography

Giornate del CrisDi

X-ray Powder Diffraction: a useful tool for Chemistry and Material Science

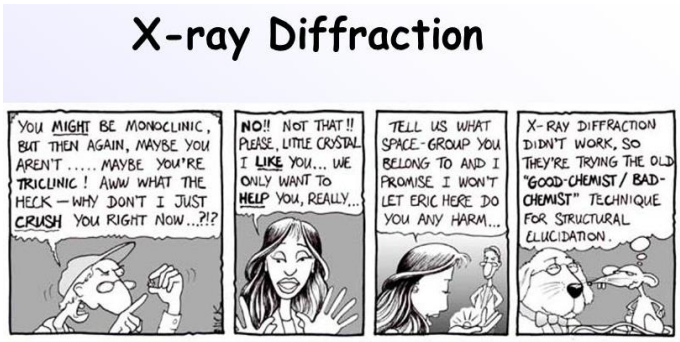
28 February, 2019 h 8:50

Dipartimento di Chimica, Università di Torino, Via Giuria 7, Torino

Room: Aula Magna

Abstract

Within the activities of the Interdepartmental Centre for Crystallography (CrisDi) of the University of Turin, promotion and dissemination of the crystallography remain a top priority objective.
To this end, CrisDi has planned several workshop which are intended to be an informal discussion meeting, where the crystallography is the common ground, in order to encourage the collaboration between partners of the same or different scientific area.
This second meeting is addressed to the Chemistry Department staff. The contributions would display how some crystallographic methodologies, such as traditional X-ray diffraction or non ambient X-ray diffraction techniques, chemometrics analysis of X-ray data, X-ray total scattering or Rietveld method, can be helpful from the daily laboratory activities to scientific problem solving.



Organizers: G. Fiore, D. Marabello, P. Benna, M. Milanesio

Lingua ufficiale Italiano

Registrazione gratuita, ma obbligatoria

Inviare una e-mail a: gianluca.fiore@unito.it entro il 10/02/2019

Programme	
9:00 - 9:05	*Welcome* D. Marabello – Direttore del centro interdipartimentale CrisDi, Dipartimento di Chimica – Università di Torino
9:05 - 9:15	X-ray powder diffraction facilities in Turin G. Fiore – Dipartimento di Chimica – Università di Torino
9:15 - 9:45	*Microstructure of materials by X-ray diffraction* M. Baricco – Dipartimento di Chimica – Università di Torino
9:45 - 10:15	*Analysis of diffraction data by means of chemometrics methods: examples and applications* M. Milanesio – Dipartimento di Scienze ed Innovazione Tecnologica – Università del Piemonte Orientale
10:15 - 10:45	*Coffe Break*
10:45 - 11:15	*Quantitative analysis of XRPD data: traditional methods* L. Palin – Dipartimento di Scienze ed Innovazione Tecnologica – Università del Piemonte Orientale, Nova Res s.r.l.
11:15 - 11:45	*Thermal evolution of Mn_xO_y nanostructures starting from two different templating agents* A. Chiodoni – Istituto Italiano di Tecnologia – Torino
11:45 - 12:15	*Advanced X-ray Total Scattering Methods based on the Debye Scattering Equation for Characterizing Nanomaterials* F. Bertolotti – Dipartimento di Scienza e Alta Tecnologia – Università degli Studi dell'Insubria
12:15 - 12:25	*Concluding Remarks*

Microstructure of materials by X-ray diffraction

M. Baricco – Dipartimento di Chimica – Università Torino

X-ray diffraction is an experimental technique suitable to obtain information on the microstructure of materials. From a careful analysis of the broadening of the X-ray diffraction peaks, it is possible to obtain the value of the size of the coherent scattering domains and of the microstrain. Different numerical techniques available to separate the two contributions from the line broadening of the diffraction peaks will be described (e.g. Scherrer, Williamson-Hall, Warren-Averbach). In addition, the effect of defects (e.g. stacking faults, twins, dislocations) on the line broadening will be considered. The role of preferred orientations in the investigated material (i.e. texture) on the diffraction pattern will be described, providing information on available experimental techniques to quantify them. Finally, measurements for the determination of residual stresses by X-ray diffraction will be briefly illustrated.

Analysis of diffraction data by means of chemometrics methods: examples and applications

M. Milanesio – Dipartimento di Scienze ed Innovazione Tecnologica – Università Piemonte Orientale

Principal component analysis (PCA) and regression (multifit) methods for XRPD data analysis methods, based on a "structure-free" approach will be introduced. Hints on their fundamentals and implementation and examples of application will be given. Strengths and weaknesses, compared to traditional methods, will be highlighted through examples. In particular, the theme of preferential orientation will be addressed. Finally, recent results on the application of quantitative analysis for the study of the kinetics of solid state reactions will be described.

Quantitative analysis of XRPD data: traditional methods

L. Palin – Dipartimento di Scienze ed Innovazione Tecnologica – Università Piemonte Orientale, Nova Res s.r.l.

Qualitative and quantitative analysis are the most widespread applications of powder X-ray diffraction in both academia and industry. The traditional methods of quantitative analysis will be described, starting from sample preparation and measurement execution, then passing to Rietveld refinements and then introducing methods that do not needs the use of the crystalline structure. After a brief description of the fundamentals of these methods, the parameters that influence the goodness of the result and examples of applications to mixtures of different complexity will be discusssed.

Thermal evolution of MnxOy nanostructures starting from two different templating agents

A. Chiodoni – Istituto Italiano di Tecnologia – Torino

Manganese oxides are known to be good catalysts towards the Oxygen Reduction Reaction (ORR). Two low-cost and green synthetic approaches have been considered, with the aim to obtain nanostructured manganese oxides with tailored morphological, compositional and catalytic properties; they are here presented. The crystalline phase evolution as a function of temperature was in particular investigated through in-situ XRD measurements, and combined with electron microscopy measurements. With this approach it was possible to associate a specific morphology to a specific crystalline phase, and to propose a crystallization mechanism for the different manganese oxides obtained.

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

Federica Bertolotti,[a] Daniele Moscheni,[a] Antonio Cervellino,[b] Norberto Masciocchi,[a] Antonietta Guagliardi[c]

[a]*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*.

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.[7–11] Experimental and modeling aspects will be presented along with an overview of applications, which range from quantum dots (as dry powders and colloidal suspensions), to mechanochemically processed alloys up to innovative engineered nanoapatites.

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