

Highlights from the Latin American conference on applications of powder diffraction

This special issue of *Powder Diffraction* contains papers presented at the *Latin American Workshop on Applications of Powder Diffraction and Mini-Course on Methods of Powder Diffraction*, held in Campinas, Brazil from April 16th to 20th, 2007. During the past few years, there has been a significant rise in interest in powder diffraction by the Latin American scientific community, not only in routine phase identification and quantification, but also in structure solution and refinement aiming at applications in materials science, physics, chemistry, archaeology, pharmacy, and biology. The recent construction and commissioning of a dedicated beamline for X-ray powder diffraction at the *Laboratório Nacional de Luz Síncrotron* (LNLS), a regional open facility, increased the number of opportunities for Latin American researchers to systematically carry out competitive research using powder diffraction as the main characterization tool. It thus seemed appropriate to bring together researchers to discuss the most recent advances in this area. The conference had 150 registered participants, with a total of 128 accepted abstracts.

The main conference was preceded by a two-day mini-course on the methods of powder diffraction (April 16th and 17th), taking advantage of the expertise of the long list of distinguished invited speakers. In this mini-course, each presentation lasted 45 to 60 minutes. **Peter Stephens** delivered an inspiring, opening lecture "Powder to the People," providing the historical aspects and present status of powder diffraction, followed by an excellent account by **Andy Fitch** of some experimental procedures employed for neutron and X-ray powder diffraction (using both conventional and synchrotron sources for the latter). **Jim Kaduk** then provided an overview of the principles of phase identification with powder diffraction, with interesting and practical demonstrations, followed again by **Peter Stephens** with a clear tutorial on the physical and mathematical descriptions of powder diffraction lineshapes. In the following lectures, **Armel Le Bail** and **Carmelo Giacovazzo** nicely addressed issues related to *ab initio* structure solution, namely, getting cell parameters (indexing), space group determination, and *ab initio* phasing. The program then shifted to aspects of structure refinement, with **Brian Toby** providing an illuminating introduction to the theory of least-squares fitting, and **Bob Von Dreele** yielding an authoritative account of refinement strategies in the Rietveld method. The second day of the mini-course started with **Juan Rodríguez-Carvajal** delivering a very complete introduction to magnetic structure determination and refinement by neutron powder diffraction, followed by **Simon Billinge** with an exciting account of the atomic pair distribution function method. **Brian Toby**, continuing his eloquent exposition of the statistical theory, introduced the *R*-factors and graphical analysis of fits as important tools to judge

quality in the Rietveld method. A nice description of the recent advances in transmission powder X-ray diffraction, with emphasis on conventional X-ray sources, was presented by **Brian Litteer**. The program of the mini-course was finally completed by enjoyable presentations of the potentialities of key software used in the analysis of powder diffraction data: EXPO2007 by **Carmelo Giacovazzo**, GSAS and EXPGUI by **Bob Von Dreele**, TOPAS by **Arnt Kern**, and FullProf by **Juan Rodríguez-Carvajal**. At this point, even before the opening of the main conference, the audience was already astonished by the quality of the presentations and degree of expertise of the speakers. Indeed, this can be verified by anyone, since most of the lectures described above were recorded and may be freely accessed at <http://www.lnls.br/lawpd/recorded.htm>.

The opening section of the main conference was chaired by **Iris Torriani**. After a short prelude, in which the current capabilities for X-ray powder diffraction at LNLS were described by myself, **Armel Le Bail** discussed the frontiers between crystal structure prediction and determination by powder diffractometry, and demonstrated for the first time the Predicted Crystallography Open Database (PCOD) (see his paper in this issue of *Powder Diffraction*). Afterwards, **Miguel Delgado** reviewed the recent research carried out by his group on the structure of multinary semiconducting compounds. In the following section, chaired by **Stefan Kycia**, **Bob Von Dreele** discussed the recent advances in protein powder diffraction, showing how the sharpness of typical protein powder Bragg peaks and their position sensitivity to external parameters compensate partially the loss of information caused by peak overlap, leading to successful structural analyses of small proteins. **Nivaldo Speziali** presented a study of phase transitions on $K_2Mo_xW_{(1-x)}O_4$ mixed compounds, including an analysis of modulated phases characterized by the presence of satellite Bragg peaks in the powder patterns. **Andy Fitch** showed a number of studies on complex systems by high-resolution powder diffraction patterns taken at the European Synchrotron Radiation Facility (ESRF). Finally, **Ernesto Estevez-Rams** reviewed how stacking disorder can be characterized through X-ray powder diffraction, also describing the recent relevant contributions of his group to this field. The program of the day was completed by a poster section, a visit to LNLS, and a welcome reception featuring a historical soccer match amongst the participants.

The first section of the second conference day was chaired by **Yvonne Mascarenhas**. **Carmelo Giacovazzo** shed light onto the use of direct-Patterson methods and Monte Carlo techniques for powder diffraction data, followed by **Fernando Rizzo**, who presented a study of nega-

tive thermal expansion in oxides. **Juan Rodríguez-Carvajal** showed the complex and intriguing low-temperature magnetic phase diagram of multiferroic TbMnO_3 , and **Leopoldo Suescun** presented his work on a new series of oxygen vacancy-ordered perovskite manganites. The following section, chaired by **Graciela Delgado**, was opened by **Bill David**, who showed how powder diffraction has become a key method in studying polymorphism in pharmaceutical materials. **Luís Gallego Martínez** then presented a structural study on the high- T_c superconductor $(\text{Hg,Re})\text{-1223}$ under different oxygen contents, and **Ana Elisa Bianchi** showed her work on microstructure characterization of copper oxide nanostructures; these last two studies are reported in this issue of *Powder Diffraction*. After lunch, **José Antonio Henao** chaired a session that started with **Jim Kaduk** reporting the crystal structures of several new trimellitates (derived from trimellitic acid, $\text{H}_3\text{C}_9\text{H}_3\text{O}_6$), and examining the bonding in these materials using quantum chemical calculations. **Carlos Parente** introduced the community to the new position-sensitive detector neutron diffractometer located at *Instituto de Pesquisas Energéticas e Nucleares* (IPEN), São Paulo, Brazil, and **Silvia Cuffini** described her work on crystal structure characterization of pharmaceutical drugs by powder diffraction. Finally, **Ariel Gómez** introduced the concept of pseudo-intensities for *ab initio* structure determination from X-ray powder diffraction data. The program of the day also included a poster session and social dinner.

The closing session, in the morning of the third conference day, was chaired by **Diego Lamas**. The methods for solving the structure of complex nanostructured materials (the so-called “nanostructure problem”) were described by **Simon Billinge**, followed by **Paulo Suzuki** with an analysis of the crystal structure of $\text{Nb}_5(\text{Si},\text{B})_3$ solid solution by high-resolution X-ray powder diffraction. **Peter Stephens** then illustrated some real cases from his own experience where some work beyond the routine was necessary to obtain the necessary structural information. **Adriana Serquis** presented a study on powder and film samples of cobaltites, demonstrating correlations between the microstructure and electrochemical properties. A final talk was delivered by **Brian Toby**, who presented his view on how Rietveld analysis should be advanced in the future. His talk motivated a vivid discussion with the audience, with many of the previous speakers also sharing their perspectives on the powder diffraction technique and related software.

The success of this conference may have been judged not only by the outstanding quality of the invited and contributed talks, but also by the interest of all participants on the poster sessions, leading to insightful interactions between beginners and experts. Almost the totality of the 104 accepted posters were actually presented by highly motivated researchers and students. Of course it would be impossible to mention all of the interesting contributions, so instead I highlight some of these works. **Márcio Góes** and co-authors presented a structural and morphological analysis of BaTiO_3 phase formation in powders prepared through the mechanical activation chemistry. **Antônio Adorno** and collaborators presented a study of the pearlitic reaction in copper-silver alloys with silver additions. **Hamilton Corrêa et al.** showed crystal

structure refinements of Co-doped lanthanum chromites prepared via combustion synthesis, and **Gisele Castro** and co-authors reported on the characterization of a lipid nanoparticle formulation for topical treatment of acne. **Daniela Leite** and collaborators studied the effect of calcination temperature during the formation of $\text{Sn}_{0.9}\text{Ti}_{0.1}\text{O}_2$ doped with CoO and Nb_2O_5 , aiming at potential applications as ceramic sensor devices. **Marcos Tadeu Orlando et al.** presented a study of the phase composition of human renal calculi. Zinc oxide was the subject of quite a number of contributions. For example, zinc oxide was studied in the form of thin films by **Ana Cláudia Bernardes-Silva**, **Milena Sabino**, and co-authors and also by **Gabriel Juárez et al.**, aiming at structural characterizations of samples designed for photovoltaic applications. Nanocrystals of transition-metal-doped zinc oxide, grown by a process involving dissolution of salts in coconut water, were studied by **Daniel Santos** and co-workers. An alternative synthesis method, now using gelatin as a precursor, was also employed by **Cláudio Remédios** and **José Sasaki** to grow nanoparticles of nickel oxide, which were characterized by X-ray powder diffraction. **Rodolfo Fuentes**, **Diego Lamas**, **Ismael Fábregas**, and co-authors presented a set of contributions with systematical investigations of the tetragonal-cubic phase transitions in nanocrystalline $\text{ZrO}_2\text{-CeO}_2$, $\text{ZrO}_2\text{-Sc}_2\text{O}_3$, $\text{ZrO}_2\text{-CaO}$, and $\text{ZrO}_2\text{-Y}_2\text{O}_3$ solid solutions. These systems are interesting because of a wealth of possible applications, such as redox or oxygen storage promoters in three-way catalysts, anodes in fuel cell technologies, and others. Finally, **Andréa Ferreira et al.** employed the Rietveld method to quantify crystalline phases of Portland cement clinker doped with transition metals. All of these studies produced peer-reviewed articles published in this issue of *Powder Diffraction*. I believe these selected contributions represent very well the quality and level of multidisciplinary of today’s Latin American powder diffraction community.

These special events would not have occurred without the help and support of many people and organizations. The members of the organizing committee, **Cristiane Rodela**, **Daniela Zanchet**, **Fábio Furlan Ferreira**, **Guinther Kellerman**, and **Roosevelt Droppa, Jr.**, are acknowledged for their dedication in the preparation and execution of this conference. Special thanks are also given to the LNLS secretariat, in particular **Ana Lúcia Ferreira**, who carried much of the load of the organization with dedication and professionalism. The members of the International Advisory Board, **Armando García Perez**, **Daniel Vega**, **Iris Torriani**, **Jorge Garín**, **José Antonio Henao**, **Lauro Bucio**, **Miguel Delgado**, **Silvia Cuffini**, and **Yvonne Mascarenhas**, played an important role in the suggestion of invited speakers, in the divulgation of the conference in their home countries, and for general advice on the organization of the scientific program. **Miguel Delgado** and **Iris Torriani** are acknowledged for conceiving the conference itself, during the XX Congress of the IUCr in Florence, Italy, 2005. A number of organizations, foundations, and companies supported the conference and mini-course, contributing to their success, namely LNLS, which hosted the event, **IUCr**, **ICDD**, **Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP)**,

Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Centro Latino-Americano de Física (CLAF), Banco do Brasil, Bruker AXS, PANalytical, and Rigaku. I consider that such a significant support by these entities revealed their commitment and interest in the development of an even more robust powder diffraction community in Latin America. Finally, we acknowledge the editorial staff of *Powder Diffraction*, especially **Ting C. Huang** and **Miguel Delgado**, for supporting the publication of the con-

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