

Accuracy in Powder Diffraction IV Program / Topics

April 22-25, 2013 – NIST, Gaithersburg, MD

Plenary I – Accuracy In Powder Diffraction - Are We There Yet?, *Bob von Dreele*

Microsymposium 1 – Standards, Chair: *Jim Cline*

Mission Statement: In this session we will examine the technical requirements, and manner of executing SI – traceable measurements of lattice parameters with powder diffraction at NIST. Measurement issues that are not specific to line position, such as quantitative analysis, require SRMs that are certified with an alternative measurement method. We address the means by which these certification experiments are designed to impart a meaningful understanding of the sources of both random and systematic error.

Microsymposium 2 – Instrumentation, Chair: *Pam Whitfield*

Mission Statement: Laboratory, synchrotron and neutron powder diffractometers have all seen new sources, optics and detectors introduced over the past decade; in many instances revolutionizing what experiments are possible. Both speed and accuracy have benefitted but not always at the same time....

Microsymposium 3 – Data Collection and Analysis, Chair: *Arnt Kern*

Mission Statement: In the past two decades a multitude of instrument and data analysis software developments have exponentially broadened the capabilities, and thus the application range, of X-ray diffraction analyses. Most significant hardware advances were made in the areas of X-ray detection and X-ray beam conditioning, providing data quality and amounts of data not thought to be possible only a few years ago. At the same time computing methods have radically improved the quality of information obtained from X-ray diffraction data, including refinement of non-crystallographic parameters.

Microsymposium 4 – Structure Determination and Refinement, Chair: *Jim Kaduk*

Mission Statement: This session will cover both current and prospective methods of structure validation; how do we determine that we've gotten the right (or at least a chemically-reasonable) answer. The tools include both comparison to existing structures, and quantum chemical techniques. Such computational methods are increasingly useful both in structure validation and structure prediction/solution. New algorithms for structure solution will also be included.

Microsymposium 5 – *In situ* Studies, Chair: *Robert Dinnebier*

Mission Statement: Soon after the first powder patterns appeared in literature, the power of the method for following processes in-situ was recognized. Since then, the time scale and the precision of the method improved by several orders of magnitude. With the general availability of high intensity

Agenda

laboratory and synchrotron sources and the use of 1D- and 2D- position sensitive detectors the field of in-situ X-ray powder diffraction has experienced a renaissance. The experimentalist is faced with the challenge that a vast amount of data needs to be reproducibly processed and analyzed. This session will present up-to-date tools for measurement and evaluation of in-situ powder diffraction data with a focus on accuracy.

Manufacturer's Session, 9 minute shorts, Chair: *Ian Madsen*

Mission Statement: Commercial manufacturers will be given an opportunity to highlight recent developments in their diffraction instrumentation and data analysis. Speakers will be given a strictly enforced 9 (nine) minutes to portray their latest offerings. Awards will be presented to those that can come closest to the 9 minute mark. Those exceeding 9 minutes, however, are disqualified.

Poster Session – While all oral presentations at APD-IV are invited, other meeting attendees have the opportunity to contribute posters which will be displayed for of the duration of the meeting. Poster content should be in keeping with the general theme of the meeting, namely, accuracy and on precision in powder diffraction.

Microsymposium 6 – Quantitative Phase Analysis, Chair: *Ian Madsen*

Mission Statement: Quantitative phase analysis (QPA) is being applied to an ever increasing range of mixed phase systems of interest including mineral exploration and processing, material science and pharmaceuticals to name a few. This expansion is driven in part by (i) access to instruments with high-intensity sources and (ii) the proliferation of whole pattern analysis methods coupled to interfaces which make the methods more accessible to the novice user. However, the fundamental limitations embodied in the observed diffraction data still determine the level of accuracy which can be attained. This session will present the current state of the art in QPA and highlight some of the approaches being taken to improve accuracy and precision.

Microsymposium 7 – Proteins & Pharmaceuticals, Chair: *Bob von Dreele*

Mission Statement: These materials comprise the most formidable problems addressed for structure determination and refinement by powder diffraction. Moreover, there is a heavy reliance on data enhancement (restraints) and parameter reduction (constraints) techniques which can compromise the quality of the results. Thus, these issues will be the main focus of this session.

Microsymposium 8 – Mineralogical Applications, Chair: *Johan deVilliers*

Mission Statement: Accurate powder diffraction data and their interpretation can make the difference in the identification of minerals, in describing their distribution in ore bodies and in their beneficiation during processing. The importance of adequate sampling, accurate QPD, and also the correlation with sample chemistry will be discussed as well as the use of cluster analysis in data interpretation

Open Forum – The Session Chairs moderate audience driven questions and points of discussion.

Microsymposium 9 – Pair Distribution Studies and Total Pattern Analysis, Chair: *Simon Billinge*

Agenda

Mission Statement: Total scattering (TS) and atomic pair distribution function (PDF) analysis of powder diffraction data is emerging as a powerful way to study structure at the nanoscale. As it gains wider acceptance it is becoming crucial to establish protocols for validating the reliability of results coming from this kind of study. In many cases errors are not even propagated to the TS/PDF functions. Even when they are, error correlations, which may be significant, are not considered. Systematic errors are rarely considered in any detail. Finally, complicated modelling is often carried out with little rigorous understanding of the validity of the resulting models. This session will investigate both these problems and efforts under way to address them.

Microsymposium 10 – Stress / Strain, Chair: *Andreas Leinweber*

Mission Statement: Diffraction patterns from polycrystals are always affected by the microstructure of the material under consideration, leading to line-broadening, line-shifts and further effects. Such effects on the one hand complicate phase identification and structure determination but on the other hand allow extracting valuable microstructural information. The speakers will present new approaches how to extract reliable microstructure information from diffraction data.

Microsymposium 11 – Where to from here? Chair: *Jim Cline, Ian Madsen*

Plenary II – Powder Diffraction: The Best Is Yet To Come, *Bill David*

Plenary III – The First X-ray Diffraction Results from the Mars Science Laboratory, *Dave Bish*