Preface

Powder diffraction is a versatile, widely used method with a manifold of applications. Even after almost a full century of steady progress and development, further advances occur and new applications emerge. It has turned out that, as methodological and instrumental progress occurs, new applications materialise, which, in turn, provide a driving force for further methodological and instrumental developments. At the time being, powder diffraction profits enormously from a combination of instrumental advances and massive computing power (see what follows). This opens the door to the solution of more complex crystal structures, the simultaneous use of multiple diffraction lines or even multiple diffraction patterns in diffraction investigations of the microstructure of materials and the like.

Modern laboratory diffractometers with improved X-ray optics (as multilayer mirrors, double monochromators, polycapillary collimators and the like), more efficient detection systems (e.g. linear multiple solid-sate detectors, two-dimensional gas-filled detectors, image plates, CCD's etc.) and brighter and more brilliant X-ray sources (as microfocus tubes, rotating anode sources etc.) are nowadays available. These developments provide a gain in resolution and permit investigating smaller sample volumes (micro-diffraction) and faster acquisition of diffraction patterns (time-resolved and in-situ studies). Experiments which would have been possible only at a synchrotron beam line in the past may nowadays be performed in-house using state-of-the art diffraction instrumentation.

On the other hand, **third generation synchrotron facilities** like ESRF, SLS and other installations equipped with insertion devices provide high flux densities at high energies, pushing the limits of ultra fast in-situ and high resolution studies even farther. The availability of two-dimensional detectors allows the collection of full Debye-Scherrer rings which increase considerably the amount of detected photons, increases the accuracy of measured data, and reduces problems associated with coarse-grained and textured samples. This is especially useful in protein powder diffraction due to the reduction of collection time in comparison to detection systems based on crystal analysers, and provides, when combined with focussing optics, the angular resolution indispensable for the successful indexation of large unit cells, precise phase analysis and line broadening studies.

Neutron powder diffraction continues to have a high impact in many fields of solid state chemistry and physics: magnetic structures and precise crystallographic structures of oxides, metal hydrides, battery materials, etc. Recent **neutron powder diffraction advances** include the development of diffractometers combining high resolution and high flux, thereby allowing the solution of a large class of structural problems. Very small or hydrogenated samples as well as high pressure work at different temperatures can now be tackled in machines like GEM (ISIS) or D20 (ILL) in a routine way. If enough sample material is available, full diffraction patterns of very high quality, and free from systematic errors (texture), can be obtained in a few minutes. This opens the large field of real-time high-precision crystallography with data treatment based on sequential Rietveld refinements of a large amount of diffraction patterns taken as a function of temperature. If higher resolution is needed improved machines like HRPD (ISIS) and super-D2B (ILL) are available. Traditional applications of neutron powder diffraction as the determination of magnetic structures will be

improved with projected new machines having higher intensity and resolution at low Q (for instance WISH at ISIS). Initiatives like XPRESS (ISIS) and EASY (ILL) are providing a simpler access to the neutron facilities for powder diffraction.

Pair Distribution Function powder diffractometry is another field that largely benefits from the increasing availability of synchrotron radiation up to high energies and ToF neutron sources. In this technique access to high Q values is of paramount importance to diminish truncation errors in the Fourier transformation. This technique allows analysis of local structural features which do not follow the periodicity of a crystal (see what follows on Workshops).

Due to a combination of modern robotics and control software it is possible nowadays to process almost instantly a huge amount of collected powder diffraction data. In this way combinatorial screening studies permit sampling of a huge compositional domain for optimising a specific property of a material (synthetic chemistry), and phase transitions can be traced with high time resolution. But not only radiation sources, optics and detection systems have improved. Also noteworthy advances have been achieved in sample environment, extending from more sophisticated reaction chambers (in-situ studies) to the combination of diffraction with other measurement techniques like Raman spectroscopy. A final aspect that one should not forget and to which considerable attention has been paid is sample preparation, as, for example, the preparation of texture-controlled samples or the preparation of cryo-cooled protein samples.

Software development is also increasingly important. The evolution of the structure solution algorithms in reciprocal or direct spaces progressed so far that the limiting factor is not the structure solution algorithm any more but rather the synthesis of good quality samples allowing unambiguous indexing of Bragg peaks. One technologically important field profiting enormously from continuous software development consists of the investigation of lattice defects, residual stresses and texture in thin films and surfaces layers of materials.

One of the characteristics and strengths of powder diffraction is the ease of sample preparation: Virtually any form of material as powder, flat plate etc. can be investigated, vacuum is not required, as is the case for electron diffraction etc. Moreover, powder diffraction patterns can be collected very fast. Therefore, there is now a growing trend to exploit this data collection simplicity by measuring multiple patterns in order to tackle more complex problems. One example is the treatment of anisotropic changes in the metric induced by physical processes (pH, T, P, radiation damage...) to increase the number of extracted integrated intensities. Other examples are data processing in techniques like isomorphous replacement in which treatment of multiple patterns is necessary, or also in high-throughput screening.

A simple way of following the powder diffraction evolution is by looking at the topics of recent relevant international powder diffraction workshops. Obviously, these topics reflect not only consolidated interests of the powder diffraction community but also emerging fields. Of special relevance were the **three satellite workshops** of the EPDIC 10 Conference held in Geneva (Switzerland). One of the workshops was devoted to **pharmaceutical applications**, more specifically to new methods and trends in X-ray powder diffraction which are relevant

to the pharmaceutical industry with special emphasis on regulatory aspects of daily laboratory work. In the second workshop a significant part of the public domain **powder diffraction software** was described in detail by the authors. The third workshop dealt with the interaction of **nano-materials and powder diffraction**. Since, due to the complex structure of nano-materials, the standard methods of structural analysis are of limited use, the development of new approaches and tools for structural analysis is an impetus. New X-ray diffraction techniques and instrumentation specifically designed for nano-materials characterisation were described in this third workshop (nanoparticles, nanoporous materials and nanostructured bulk material). It was shown how one can limit the range over which the structural model is to be refined on atom-to-atom distances when using the Pair Distribution Function approach. Focusing on small distances up to a few Angstroms will illuminate the local structure whereas refinements over a wide range will yield the medium- and long-range structure. Particularly interesting in this workshop were also additional examples that combined, with powder diffraction data, information either from computer simulations based on molecular dynamics, or from low-angle scattering measurements, or from electron precession measurements on a single nanocrystal. Due to its importance, an ECM-23 satellite school dedicated to the complementarily nature of powder and electron precession diffraction was organised in Antwerp (Belgium). In the summer of 2007, two small workshops on powder diffraction take place at the ERSF. One will concentrate on the developments and directions of powder diffraction as applied to the investigation of proteins, to better determine the real possibilities of the technique. In principle, this technique should be regarded as complementary to recognised single-crystal techniques. However, for those protein applications more oriented to materials, powder diffraction in combination with improved molecular replacement methods should have a brilliant future. The second one deals with Pair Distribution Function analysis using Total Scattering of X-rays and neutrons. The purpose is to generalise the different Total Scattering data analysis strategies available, so that in the future this approach can be done routinely. In both cases, however, these methods are in a virginal state and it will take some years until these methods obtain maturity.

We are glad to have the opportunity to introduce the proceedings book of EPDIC 10. Its contents give a clear and representative overview of the state-of-the-art in powder diffractometry. A statistical analysis of the contributions and a few editorial remarks can be found in the Editorial Notes. We hope you will find the reading of this new edition of EPDIC proceedings at least as interesting, stimulating and suggestive as we have.

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