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Report on the Workshop Group Meeting on: Next Generation Powder Diffractometers

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I. OUTLINE OF THE WORKSHOP

The workshop was held on Thursday, June 18 1998, and was attended by a small group of people (~8), representing US, European, Russian and Japanese facilities. The attendees were asked to work on the following list of questions:

- What are the most promising future scientific opportunities for neutron powder diffraction (NPD)?
- How can we extend the present, most successful NPD techniques, into the next century? Are there new techniques, or methods that were not very successful in the past and should be revisited?
- How can we link instrument types with scientific requirements? How have the present instruments designs come about?
- Can we come up with a short list of truly "novel" instruments?
- What technology do we need to make our ideas into real instruments?

II. SCIENTIFIC OPPORTUNITIES

A short discussion was held on the future scientific opportunities for NPD. It was agreed that a lengthy debate was not appropriate, since plenty of publications already exist on the subject. However, most of the participant felt that "physical" diffraction, "chemical" crystallography (including catalysis and in-situ reactions), materials science and engineering science will still represent the core applications of NPD. The issue of whether more "routine" industrial measurement capabilities (like drug structure determination and testing of real engineering components) should be offered was debated, but the reaction of the participants on this subject was rather negative. As far as the interaction between neutrons and other radiation sources (namely X-ray synchrotron) and techniques is concerned, the general opinion was that complementarity should be stressed over competition.

III. TECHNIQUES

In our opinion, structural refinement is by far the most successful NPD technique. There was general consensus on the fact that the primary direction to extend refinement techniques should be in the direction of greater speed. This will allow complex phase diagrams to be mapped in detail. Also, kinetic studies, including both stroboscopic studies of reversible phenomena and "single-pulse" measurements of irreversible processes, should become routine. Another extension of the technique should be in the direction of studying smaller samples, both for novel materials and for demanding special environments (e.g., diamond anvil cells). The Q-domain of the data should be extended: low-Q data are needed for magnetic diffraction and host-guest problems, areas in which pulsed sources should progressively become competitive with reactors. High-Q data are needed to study subtle defects, lattice anharmonicity and to study disorder. In this respect, it was remarked that reciprocal space and real-space data (Fourier-transformed) should become more complementary, not only for liquid and amorphous materials, but also for studying disorder and local effects in crystalline materials. Some of the "traditional" strengths of NPD, like isotopic substitutions, should be carried into the next century. Also, combined x-ray and neutron refinements should become more widespread.

The possibility of solving unknown small-molecule crystal structures on a routine basis was debated at length. Since pharmaceutical companies are among the potential users of this technique, this issue has a potentially large financial impact on neutron scattering in general. The majority of the participant felt that, as things stand, the likelihood of successfully competing with or offering a useful complement to synchrotron-based techniques in this field is small. However, the scenario may change as new instruments are developed. On the contrary, there was general consensus on the fact that the majority of new instruments should incorporate some structural solution capabilities, especially to study new phases which can only be created "in-situ" and, in particular, metastable phases. The connection between this and the levitating sample techniques was underlined.

Among the other desirable extensions of NPD, the following were mentioned: i. Diffraction imaging; ii. Establishing a continuous link between powder and single-crystal diffraction (for textured samples, fibres, twinned crystals etc.); iii. Tackle inelasticity correction by measuring $S(Q_{elastic})$ as a function of wavelength; iv. Exploiting polarised neutrons and polarisation analysis; v. Develop extended Real-Space representations of the data (spherical harmonics, etc.).

The general consensus was that neutrons should not be used to test real industrial components before they are marketed, but should rather the efforts should be concentrated on engineering science (strain modelling, etc.). Also, ultra-high-resolution applications were not believed to be of particular interest.

IV. INSTRUMENT DESIGNS

The participants were asked to take a fresh look at the present instrument designs and their historical development. Most of the present designs were developed in the eighties, under a strict set of technological constraints. In particular, the lack of computing powder and disk storage space required a significant amount of electronic pre-processing (focusing) to be performed. Also, linear position-sensitive detectors were not as widely available as in the present days. Alternative instrument concepts were already envisaged in the eighties, but were not implemented. Clearly, the technological limitations have now changed, and this could have an impact of future instrument designs. Also, it was clearly indicated that the data produced by the new instruments should be easier to analyse even by the casual user. Therefore, the pulse-shape and geometrical aberrations should be minimised, even at the expense of some flux or of an increased detector cost.

The wisdom of maintaining a segmented detector structure, divided in several banks, was questioned by various speakers. A continuous detector design, with fine pixillation also along the polar coordinate, would allow the full three-dimensional structure of the data to be exploited. In this scenario, the user would be free to use all the data (e.g., for single-crystal or texture applications) or to chose the one-dimensional projection that best suits his/her needs (e.g., angle-dispersive data for magnetic structure determination and large structure refinements). The benefit of this approach to determine the most appropriate inelasticity corrections for disordered systems was also discussed. The difficulties connected with building such instruments were also debated. Having an appropriate collimation to reduce backgrounds was seen as a potential problem. Also, such concept would require the development of "expert" software codes that would help the user to visualise the data and make informed and timely choices.

The panellists were asked to propose one or more truly "novel" instrument concepts. It was agreed that many of the present-day instruments will still be valid in the future, but no need was felt to discuss them. The following "innovative" designs were suggested:

- Small-Sample Powder & Single crystal diffractometer. This instrument will have a 40-80m flight-path and could benefit from a high repetition rate from a cool or cold moderator. It would have a compact design and will be highly pixillated in all three dimensions. Its software should have very good visualisation, slicing and projection capabilities. Such and instrument would be suitable for powder and single crystals, crystallographic and magnetic structural solution, host-guest problems, texture and microstructural analysis.

- "Fast Neutron" diffractometer. This instrument will have a 20-30m flight-path, and should be designed for combination Real/Reciprocal space techniques applied to disordered materials, very high-Q Rietveld, Pair Distribution Functions, inelasticity corrections and anharmonicity studies.

- Polarised-neutron TOF diffractometer. It will benefit from a ^3He polarising filter for single-crystal studies. The development of fast spin nutators, in conjunction with a narrow wavelength bandwidth and polarisation analysis, will allow it to separate structural from magnetic scattering in powders.

- Time-Resolved Diffractometer. A dedicated instrument should be developed for time-resolved studies, with the aim of reaching single-pulse diffraction. This instrument will have a rather short flight-path, or, alternatively, focusing optics, and will work at high repetition rates.

A particular emphasis was placed on dedicated special-environment diffractometers, built around a single piece of equipment (e.g., high-pressure cells, pulsed magnets etc.). Also, it was agreed that the flexibility of all instruments should be enhanced by means of add-on focusing "snouts" or capillary devices. The possibility of building "combination" instruments (elastic + inelastic) was also discussed.

V. TECHNOLOGICAL REQUIREMENTS

The workshop was concluded with an extended discussion on the technological requirements of future NPD instruments.

- Targets. The distribution of NPD instruments between low- and high-repetition rate targets should be revised, since many of the proposed instruments would benefit from a high repetition rate.

- Moderators. There was unanimous consensus on the fact that a "good" pulse shape should be pursued, even at the expense of some flux. For example, this makes cold coupled moderators rather unattractive for NPD applications.

- Optics. The issue of vertical focusing versus polar angle detector coverage was briefly touched. A vertically-focused beam effectively saves on detector cost, and is more efficient for small samples where extreme resolution is not required, and at the other extreme, for very tall samples. However, it was felt that the possibility of having a "clean" unfocused beam should be preserved, and focusing should be an "add-on" option. Also, it was agreed that future guides for long-flight-path instruments should be straight, in order to preserve at least a fraction of the epithermal neutrons. The past concerns about fast and delayed neutron background have been overcome by the development of efficient T_0 (nimonic) choppers.

- Choppers. High-speed choppers should be developed, in order to allow efficient bandwidth tailoring at high-repetition-rates. Flexible and efficient chopper control systems should be developed, to allow ramping and "sliding" of the chopper phases.

- Electronics. It should be possible to de-couple the chopper time frame from the data time frame, for extended-Q data collection on a single data set.

- Detectors. The emphasis is on pixillation, mechanical flexibility, efficiency and stability.

- Polarisers. The present ^3He filters are perceived as not yet adequate for an efficient polarised-beam diffractometer, especially if the correlation technique is employed (see paper by G. Williams in these proceedings). In particular, the relaxation times should be extended beyond the present ~80 hrs. Also, fast spin flippers and nutators should be developed.

- General Software. Highly-pixillated instruments require powerful visualisation and data reduction codes, capable of helping the user to make timely decisions. This is even more crucial if the instrument is used to explore complex, multi-dimensional phase diagrams. The development of these codes should be given high priority and adequate funding. It was proposed that some of the expertise in the field on Monte Carlo instrument simulation should be transferred to this domain. The new codes should be interfaced with existing databases to allow, for example, fast phase identification.

- Rietveld Software. This obviously represents a crucial component in the NPD data analysis. It should be possible to routinely perform on-line Rietveld fits, as it is already the case for constant-wavelength data. The Rietveld method is being and should be further extended to more than one dimensions, for instance to analyse texture data, single-crystals and phase diagrams. The possibility of an international collaboration on the matter was suggested. Also, it was proposed that complex data could become "public" after a certain period of time, following the model of astronomical data.

- Measuring Strategies. The presently implemented single-user, multi-environment approach is still to be the main operation mode for powder instruments. The benefits of this approach, especially in terms of educational value for young researchers, were perceived to outweigh the potential gains in instrument performance obtainable form a multi-user, single environment mode, which could nevertheless be employed for some routine measurements. Also, the possibility of remote instrument operation ("Virtual Lab.") was briefly discussed, but it was felt that this technology is, at present, still ahead of its time.