Assessment and Recommendations of the Powder Crystallography Advisory Group (PCBAG), March 2009.

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Introduction

The Powder Crystallography beamline advisory group (PCBAG) (Wilkinson, Mitchell and Kaduk) are charged with providing guidance on the operation and future direction of the powder crystallography beamline, 11-BM. On March 17th, the scientific and technical staff associated with 11-BM (Toby, Suchomel, Doebbler, Von Dreele and Ribaud) met with advisory group (PCBAG) members A.P. Wilkinson and J. Kaduk to provide a beam line status report and an outline of their future plans. J. Mitchell met with beam line staff the week before, as he could not attend the March 17th meeting.

11-BM was originally conceived as a world class powder diffraction beam line capable of operating in the 4.8 – 40 keV energy range using either, 1) a 2D detector for parametric studies at modest resolution ($\Delta d/d \sim 1x10^{-3}$), with a time resolution of seconds to minutes, or 2) a multi-analyzer diffractometer for very high resolution powder diffraction ($\Delta d/d \sim 2x10^{-4}$) with very good sample throughput (~1 hour per data set). The second of these capabilities has been pursued and brought to fruition first, as it is unique within the USA, and it has the potential for great scientific impact.

Beam line status

The multi-analyzer diffractometer at 11-BM is fully operational and can routinely produce spectacular quality powder diffraction patterns in a short period of time. This diffractometer, and the data from it, is truly worthy of the label "world class". To support the high throughput of this diffractometer, a sample mounting robot is in use and a "mail in" process has been developed and implemented, that is both user friendly and provides a quick turn around. The "mail in" process is carefully thought out and provides a very high level of accountability, as all user requests, measurements, and outcomes (publications, abandonment of data analysis etc.) are tracked with the aid of custom software tied to an underlying database. However, the "mail in" process, as it stands, only offers room temperature and 100 K data collection using a Cryostream cooler. Current usage of the mail in process is modest, when compared to the instrument capacity, but plans are in place to ramp it up (see later).

11-BM can be used over a wide energy range. However, operation at low energies is problematic and energy changes take longer than the beam line staff, and this advisory group, would like. Ideally, energy changes of several keV should take less than 1 hour to accomplish. This would greatly aid resonant scattering studies where data is needed at multiple absorption edges and far away from any edge. Beam line characterization studies are in progress that will provide a knowledge base to guide rapid energy changes.

In the past, 11-BM suffered from a monochromator crystal breakage. This unfortunate incident made the beamline almost unusable for a significant period of time. Also the replacement crystal may have some performance issues when compared to the original crystal. Operating without a spare monochromator crystal on hand is a major vulnerability for 11-BM. It is very important that the efforts of the beam line staff to

build a strong user program are not negated by a monochromator failure that could take the beam line out of use for an extended period of time. The availability of a spare crystal would not only remove this vulnerability, it would also allow the current crystal to be removed and reworked, so that it performs well, without an interruption in operations.

The current mirror mounts are not well suited to frequent adjustments, as changes are not fully reproducible. Improved mirror mounts would aid energy changes and beam steering.

While a very nice mail in process for 11-BM access has been developed, general users (experimenters submitting GUPs) can not currently request beam time for on-site experimentation. At present, there is no user friendly interface that can be used for on-site experimentation and there are concerns that inexperienced users could do significant physical damage to the beam line while using the current interface. The development of a robust easy to use interface for on-site users, that is flexible enough to facilitate a wide range of measurements and integrated with the beamline's measurement/sample/usage tracking database, will require a significant time commitment on the part of a programmer.

Presently, the only sample environment available for routine use at 11BM is a Cryostream capable of operating between ~80 and 500 K. It can be used with samples that are mounted by hand or by the beamline's sample handling robot.

The originally proposed 2D detector capability of 11BM has not yet been implemented, as the development and operation of the "world class" high resolution diffractometer has, rightly, taken priority.

Plans for the future

The beamline staff plan to dramatically grow the user community associated with the instrument while simultaneously enhancing the capabilities of 11-BM. A campaign promoting the "mail in" service to a broad range users (materials chemists, condensed mater physicists, earth scientists, small molecular crystallographers etc) is about to be launched. New sample environment, suitable for use < 80 K, is likely to be available at 11-BM in the near future, and various possible arrangements for high temperature experiments are being investigated. In parallel with these efforts, further characterization of the beam line will be undertaken along with an exploration of possible optical upgrades, so that operations can be made more efficient.

Recommendations of the BAG

1) We strongly encourage the beam line staff to aggressively grow the user community for 11-BM. Their existing plans to promote the "mail in" service are an excellent first step. Care should be taken to market only what can be delivered. We would like to see a description of how the users (especially inexperienced ones) will be supported once they have received their data.

2) The "mail in" process and its user interface should be further enhanced to facilitate a wider range of temperatures and scans types using the existing Cryostream cooler and sample robot.

3) We strongly recommend that funds be made available for a spare monochromator crystal, so that a) in the event of a crystal failure there is not a protracted "down period" and b) the existing crystal can be reworked so that its performance is enhanced. Long down periods are intolerable when you have a rapid turn around high throughput component to your operating model, as they would undermine the growth and acceptance of the "mail in" service.

4) A wide range of sample environment needs to be made available to users ASAP. Many very important types of experiment are precluded by the current inability to operate < 80 K, > 500 K or in a controlled atmosphere. < 80 K operation should be the highest priority with operations above 500 K using, perhaps, a hot air blower as the next priority. Strategies for obtaining adequate sampling statistics (rock, spin, translate etc.) while in constrained sample environments need to be carefully explored, as the extremely high resolution of the diffractometer makes sampling statistics a critical issue.

5) We strongly encourage beam line staff to rapidly develop and implement a workable process to accommodate general user requests for on-site experiments. We understand staff concerns that inexperienced on-site users could break beam line components while using the current "unfriendly" on-site user interface. However, other beamlines, for example 1-ID and 1-BM, have operated successfully for years without developing a fool proof user interface. The PCBAG believes that it is appropriate to follow a suggestion made by Robert Von Dreele during the March 17th meeting. Initially, a user friendly interface, for on-site use, that offers basic capabilities should be implemented. After some on-site access experience has been gained, perhaps involving "friendly" general users, an upgrade to the interface should be implemented to better accommodate the activities that were found to be needed during this trial period. The user friendly on-site interface, even in its basic form, should be integrated at some level with the existing database for usage, measurement and outcome tracking. On-site use is likely to be very important for experiments requiring anything other than the Cryostream cooler. Additionally, the existing "mail in" process works well for measurements that you know you need, but it is not well suited to experiments were the outcome of one measurement will determine what the next measurement should be.

6) The BAG recommends that priority continue to be placed on the development of a user program based around the high-resolution diffractometer. We believe that a very good scientific and operating case can be made for another beam line being dedicated to parametric studies (kinetics, temperature ramps, pressure ramps, varying chemical environment) using a 2D detector. For example, 1-BM, after some optical upgrades to allow usage at higher energies than are currently possible, could quite nicely fulfill this role. Alternatively, a currently unused beam line could be optimally configured for this. The APS materials science user community also needs a flexible beamline, which can be configured to carry out unusual experiments. A bending magnet, or better still an insertion device, with operational capability in the 20-60 keV range would facilitate a wide variety of parametric powder diffraction experiments and potentially support a number of

materials science applications. However, the idea of a 2D detector at 11-BM should not be completely abandoned. Rapidly looking at a sample as a function some parameter using a 2D detector and then immediately following up with carefully selected high resolution measurements on the diffractometer could be a very useful capability.

7) We encourage the beam line staff to continue their efforts to make energy changes time efficient, as this would support resonant scattering measurements. These efforts would be aided by funding for mirror mount upgrades. However, a wide range of experiments can be done well at a single relatively short wavelength. In the immediate future, the development of the beam line's user community and overall program should take priority over full optimization of the variable energy capability.

8) The development of new powder crystallographic capabilities at the APS should be carried out in the context of the resources available at other national user facilities. Priority should be given to capabilities that have potential for great scientific impact and complement those of other facilities, such as NSLS (I and II) and SNS.