# DND-CAT Medium-Term Upgrade Proposal

The DND members have built sector 5 at the APS to pursue a program of scientific research and technological development using the brilliant x-ray beams provided. The core scientific and technical areas of interest to the DND members are: polymer science, surface and interface science, nano-materials, catalysis and environmental science. DND has chosen to focus on several key techniques in designing and building sector 5: simultaneous SAXS/WAXS measurements, x-ray standing wave and surface/interface scattering, powder diffraction and EXAFS/XANES. DND supports a strong research program for its members and for General Users interested in using the DND facilities, especially in polymer science, interfaces, and EXAFS.

Our long-term (7-10 year) vision of the development of the DND sector at APS consists of a canted-undulator ID beamline, with one line dedicated to SAXS/WAXS and specializing in polymer science and nano-materials. This line could be well served by a large-offset monochromator. The second beamline envisioned in the long-term would be dedicated to high-resolution x-ray scattering and spectroscopic techniques, utilizing both high-energy-resolution x-ray fluorescence and in-vacuum high-resolution electron detection, with the goal being the identification of chemical-state specific locations of atoms and molecules at surfaces and in buried interfaces. The medium-term upgrades to the DND sector proposed in this plan will be compatible with this long-term vision.

## *Insertion Device Beamline 5ID*

### 5IDD SAXS/WAXS applications

The key x-ray optical upgrade we propose is to add a vertically focusing mirror 54 m from the ID source in our existing 5IDB station. This new vertical mirror, in combination with our existing pair of horizontally focusing mirrors, will allow us to increase the usable flux for SAXS/WAXS measurements in 5IDD between 5 and 20 times, depending on the beamsize desired for particular measurements. The second key item for upgrade is a new SAXS/WAXS detector system, potentially using the PILATUS pixel array detector modules from Dectris. Currently the modules have 172 μm x 172 μm pixels providing photon counting at 2e6 photons/pixel/sec with no dark or readout noise and 3.6 ms readout time. A PILATUS 2M (a 3x8 grid of modules) would be an excellent SAXS detector, and two identical detectors (MAXS and WAXS) with a custom tiling arrangement could potentially provide overlapping solid angles (on alternating sides of the scattering cone) from 0.002 A-1 to 2 A-1.

The combination of flux improvement and improved detector efficiency will open up several areas of application unavailable now with our current x-ray flux/detector capabilities:

- Study of higher shear rates for polymers and polymer solutions

- Ability to follow the evolution of damage in polymer films during stretching (utilizing the DND-CAT Instron servo-hydraulic system)
- Ability to image intermediate phases occurring only during temperature ramping
- Ability to image dynamic industry-related processes such as injection molding in real-life conditions
- Greatly improved detection limits for biomolecule solution scattering measurements

Key scientific users of this facility:
DuPont: David Londono
Northwestern: Wesley Burghardt, Joseph Hupp, Chad Mirkin, Alfonso Mondragon, Kenneth Shull, Samuel Stupp, Michael Wasielewski
Dow: Brian Landes, Lizhi Liu, Brandon Kern
General Users: Frank Bates, Minnesota; Yan Gao, GE; Ronald Jones, NIST; Timothy Lodge, Minnesota; Robert Moore, Virginia Tech; Sanjeeva Murthy, Rutgers;

Cost estimates: Vertical Focusing Mirror System: $350K; Upgraded SAXS/WAXS detection system: $2000K

## 5IDC Surface and Interface Science applications

Experiments in 5IDC utilize several techniques, predominantly x-ray standing waves (XSW) but also including reflectivity and surface diffraction. The focusing mirror described above could significantly enhance reflectivity and surface diffraction measurements, but may not work well with the dominant XSW measurements because of inherent slope errors in the mirror.

A key planned specific enhancement for 5IDC is a removable shielded pass-through beampipe infrastructure upgrade for the beam passing through to 5IDD (in the long-range upgrade this would become a permanent configuration). Michael Bedzyk of Northwestern and collaborators have submitted a proposal to DOE to upgrade the existing in-situ UHV chamber in 5IDC with a high-resolution electron detector. To properly upgrade and operate this potential new facility, continuous access to the 5IDC station is essential. The shielded beampipe upgrade will enable this mode.

Key scientific users of this facility:
DuPont: David Londono
Northwestern: Scott Barnett, Michael Bedzyk, Mark Hersam, Tobin Marks, Chad Mirkin
Dow: Xue Han, Brian Pate
GUs: Orlando Auciello, Jeffrey Catalano, Paul Fenter

Cost estimate: Shielded Removable Passthrough Beampipe: $100K

## 5IDB High Resolution Powder Diffraction

The APS provided 11-detector high resolution powder diffractometer has been installed and commissioned in 5IDB. Unfortunately, several previously known deficiencies in the

system continue to hamper its full utilization as designed. Key upgrades which would allow the system to perform well in the full 11-detector high throughput mode would include new multi-axis analyzer crystal mounts (theta and chi adjustments), along with the replacement of all 11 scintillators with high count rate compatible YAP scintillators and high-rate counting modules. This will allow the system to fully utilize the high brilliance of the Undulator A source at 5IDB.

Key scientific users of this facility:
DuPont: William (Mike) Guise, David Rosenfeld
Northwestern: David Seidman
Dow: Richard Chafin, Xue Han, Brian Pate

Cost Estimate: Internal detection system upgrade: $150K

## *Bending Magnet Beamline 5BM*

The sector 5 bending magnet beamline was originally envisioned as consisting of four stations: 5BMA - optics station, 5BMB - central 1.5 mrad of bending magnet beam which had been diffracted up 1 m above the white beam, 5BMC - inboard 2 mrad of beam, and 5BMD – endstation, outboard 2 mrad of beam. Our long term vision for 5BM, as recommended by several SAC reviews, no longer includes operations in 5BMB, which opens up opportunities for using this station as a secondary optics enclosure to optimize beam characteristics for the two downstream stations, 5BMC and 5BMD.

### 5BMD EXAFS/XANES and General Diffraction

The DND-CAT 5BMD station optics have been optimized primarily for high-quality EXAFS measurements. The pair of vertically focusing Rh-coated mirrors allow for a parallel beam to be monochromatized by a double crystal Si(111) monochromator and then focused back in a 1:1 configuration downstream in the 5BMD station. This optimal optics configuration continues to work very well for both CAT members and GUs.

The general diffraction measurements in 5BMD utilize a sagittal focusing crystal which works extremely well to increase flux for diffraction by accepting 1.5 mrad of beam and delivering it to a general purpose Huber diffractometer. Unfortunately this bent crystal is unsuitable for spectroscopy and must be time-shared inside the single 5BMD monochromator in 5BMA. The energy cut-offs from the Rh-coated mirrors also hamper full utilization of the desired energy range.

The proposed optics upgrade to the 5BMD optics would involve first adding an additional Pt stripe to both 5BMD mirrors, and reconfiguring 5BMA so that the sagittal bender is in a new, completely separate monochromator. This will allow for rapid switchover between spectroscopy and scattering, allowing DND to offer these techniques unrestricted scheduling and avoiding beamtime downtime for switchover operations. The ability to utilize the Pt coating stripe on the mirrors for harmonic rejection will extend the range of energies which can be focused in 5BMD to 30 keV at 3 mrad, enhancing the

usability of the station for anomalous scattering/reflectivity and resonant reflectivity measurements.

Key scientific users:
DuPont: William (Mike) Guise, David Rosenfeld
Northwestern: Michael Bedzyk, Robert Chang, Jean-Francois Gaillard, Mark Hersam, Harold Kung, Thomas Mason, Kenneth Poeppelmeier, Bruce Wessels
Dow: David Barton, Joo Kang

Cost Estimate: Mirror refurbishment with new horizontal translation stages: $150K; New monochromator internals: $100K

### 5BMC Powder Diffraction/X-ray Microtomography

The implementation of a new second monochromator for 5BMD would be most easily implemented if the optics for 5BMC were moved into the 5BMB station. A new monochromator and new internal design for 5BMC would allow for a dual purpose optics chain for 5BMC. The current double crystal Si(111) system is suitable for the high-resolution powder diffractometer and for edge-subtraction x-ray microtomography (XMT), but not well suited for standard XMT. The new monochromator would allow for exchange (possibly in-situ) of Si(111) crystals for multilayers which would greatly increase the flux available for XMT measurements, similar to the optics implemented for the 2BM XMT capability. This increased flux will greatly improve throughput especially for the high resolution XMT which relies on very inefficient but high spatial resolution 5 micron or 1 micon implanted YAG scintillators. This should greatly improve the utilization of the 5BMC station both for internal CAT users and for GUs.

Key scientific users:
DuPont: William (Mike) Guise, David Rosenfeld
Northwestern: David Dunand, Jean-Francois Gaillard, Aaron Packman, Peter Voorhees
Dow: Xue Han, Brian Pate

Cost Estimate: New 5BMC monochromator including new multilayers: $600K

## *Prioritization*

Based on total scientific impact and especially GU impact, the prioritization of these proposed upgrades is as follows:

1. Vertical Focussing Mirror for 5IDD/5IDC
2. Removable shielded beampipe passthrough for 5IDC
3. Upgraded SAXS/WAXS detection system for 5IDD
4. Internal analyzer and detector upgrade for 5IDB powder diffraction system
5. 5BMD mirror Pt stripe enhancement and horizontal translation stages
6. 5BMD second monochromator for horizontal focusing
7. 5BMC upgraded monochromator w/ multilayer crystals