The Future of Spatially-Resolved Polychromatic Neutron and X-Ray Microdiffraction

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Polychromatic microdiffraction is an emerging materials-characterization tool made practical by powerful X-ray and neutron sources, and by advanced optics and software. With polychromatic techniques, local crystalline properties including phase, texture (orientation), elastic strain, and defect density can be mapped with submicron spatial resolution in three dimensions. Here, we describe the evolving ability to nondestructively map local crystal structure in three dimensions and discuss how future advances will help address long-standing issues of inhomogeneous grain growth, deformation, fracture, and elastic strain. Current and future applications impact virtually all materials including electronic, solar, and light-emitting-diode (LED) materials, nanomaterials, structural materials, and joining materials. In addition, the ability to focus small beams on small samples dramatically increases signal-to-noise and greatly reduces the cost for extreme environmental chambers required for high-pressure, high-temperature, high-magnetic field or corrosive environments. Polychromatic techniques efficiently use source brilliance and minimize the required sample volume, which is essential for hard-to-make materials, irreplaceable materials, and for radioactive, toxic, or otherwise dangerous materials. New polychromatic neutron capabilities will significantly extend the range of samples that can be studied with neutrons and presents important new scientific opportunities for studies of magnetic materials, low Z elements, fragile crystal structures, and small samples in extreme environments.

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I. INTRODUCTION

SPATIALLY resolved polychromatic X-ray[1–3] and neutron[4,5] microdiffraction techniques are among the most important emerging tools for diffraction mapping. Related techniques such as electron backscattered diffraction (EBSD)[6] and four-dimensional X-ray microscopy[7] have advantages with respect to two-dimensional (2-D) spatial resolution or imaging speed, respectively; however, polychromatic X-ray microdiffraction techniques are unique in their ability to nondestructively resolve three-dimensional (3-D) structure with submicron resolution and with sensitivity to elastic and plastic strain tensor distributions.[8]

Because of its unique ability to nondestructively study evolving mesoscale structure and defects, polychromatic microdiffraction has made important contributions to both long-standing fundamental problems of materials physics and to critical specific materials issues. Most of the recent progress has occurred within the last 5 years and the impact of polychromatic methods is expected to further increase with technical advances. During the last decade, for example, Budai *et al.* have shown the ability to study thin-film grain growth on a grain-by-grain basis in high-temperature superconductor (RABiTS) films.[9] This study not only provided valuable guidance for the growth of high-performance RABiTS films, but demonstrated the ability to obtain detailed combinatorial information on film growth including elastic strains, defects, and local texture that would previously have been impossible or very time consuming. In a related series of experiments, Barabash *et al.* have characterized the defect distributions and local structure of functional materials (GaN) grown on structured substrates.[10] Here, the ability to study the local structure of the films provides new insights into strategies to reduce the defect density of these materials for light-emitting-diode (LED) applications.

In another important recent series of experiments, Patel, Padmore, and co-workers discovered extensive deformation in electronic interconnects during electromigration.[11–13] Deformation had not played an important role in theories of electromigration and the direct observation of deformation during electromigration helps to clarify the important factors that affect device reliability.

In research that addresses long-standing fundamental issues of materials physics, Larson *et al.* have mapped the 3-D local defect structure under nanoindents in originally perfect single crystals.[14] This research challenges state-of-the-art models of deformation and provides new insights into the self-organization of defects in a situation where the starting and boundary conditions

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are well understood and can be directly compared to state-of-the-art computer models. Maass *et al.* have also recently reported deformation modes in micron-sized pillars that defy deformation rules expected in larger structures.[15] This work provides additional insights into fundamental deformation physics.

Budai *et al.*[16] have also recently reported 3-D observations of grain growth in Al where grain boundary migration and grain coarsening can be directly compared to theoretical predictions. Here, the experimental measurements show an unexpectedly important role of defects in grain growth (grain refinement).

In addition, experiments underway impact virtually all of materials physics. In functional materials and energy materials, defects and local structure are critical to materials performance. For example, in solar cells, novel LED materials, and detector materials, structural defects can enhance—but usually decrease—performance. In many cases, spatially resolved measurements can unambiguously determine the impact of local structural changes on materials behavior when coupled with simultaneous local probes of efficiency or output. Similarly, in joined materials, residual stresses, local texture, and defects all play important roles in materials behavior. For example, nondestructive measurements of fusion welds, electron beam welds, friction stir welds, and Sn-based solders can provide detailed new information to test theories of materials behavior. In the case of Sn-based joining materials, an international collaboration is being developed to test theories of the important problem of Sn whisker growth. Applications to multiphase materials, nano/micropillar materials, and measurements of defect development in polycrystalline materials are all possible and underway.

Although the list of high-impact experiments could continue, the message of this article is that recent advances in polychromatic microdiffraction capabilities will continue and that new developments will further revolutionize the characterization of evolving mesostructure in materials. In particular, we discuss here near-term advances in X-ray polychromatic microdiffraction that will provide far more detailed 3-D maps of mesoscale structure in far less time. We also discuss new opportunities with spatially-resolved polychromatic neutron techniques that will allow for experiments that simply are not possible today. We begin by summarizing the basic principles that underlie polychromatic microdiffraction to provide sufficient understanding of the current state-of-the-art to appreciate emerging new methods.

II. BASIC THEORY AND CURRENT IMPLEMENTATION

Polychromatic microdiffraction techniques are fundamentally related to Laue diffraction,[17] but use an incident beam focused to micron-sized dimensions. The small incident beam coupled with good detector spatial-resolution allows for precision angular measurements of the diffracted beams and reduces the sample volume probed. As in standard Laue diffraction, X-rays penetrate the sample, attenuated primarily by photoabsorption. However, if a particular wavelength meets a crystalline lattice, where it satisfies the Bragg condition, then it is scattered specularly from the Bragg planes with high efficiency (Figure 1). Ignoring the tiny index of refraction corrections, the Laue reflections can be used to determine the local orientation of the crystalline planes.

$$\hat{n}_{hkl} = \frac{\hat{\mathbf{r}}_{hkl} + j}{|\hat{\mathbf{r}}_{hkl} + j|} \qquad [1]$$

Here, $\hat{n}_{hkl}$ is the Bragg plane normal for the *hkl* Bragg planes, j is the incident beam direction, and $\hat{\mathbf{r}}_{hkl}$ is the unit vector for the Laue reflection.

Spatial resolution in two dimensions is provided by the beam size. Resolution along the beam direction is determined using a technique called differential aperture microscopy.[18] As illustrated in Figure 2, the pixel-by-pixel intensity of the millions of pixels in an X-ray sensitive area detector can be measured as a function of the wire position as the wire is translated in front of the

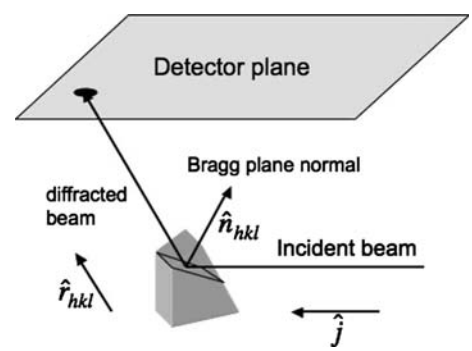

Fig. 1—The incident and diffracted beam directions determine the Bragg plane normals.

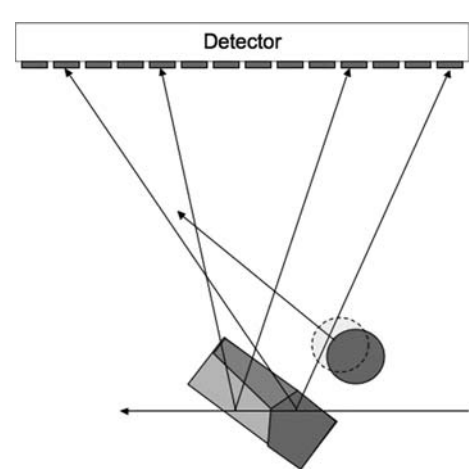

Fig. 2—Schematic for differential aperture microscopy. Note that at a given pixel, the difference between the intensity before and after the wire is moved is equal to the difference between the intensity at the leading edge of the wire subtracted from the intensity at the trailing edge of the wire.

sample. This is similar to a knife-edge scan, and the derivative of the knife-edge scan depends on the intensities near the leading and trailing edges of the wire as a function of wire position. By ray tracing past the wire edges to the incident beam path, the intensity distribution along the incident beam can be reconstructed.

Once the Laue spot distribution is determined for a particular volume element (Voxel) the spots can be analyzed to determine the phase, texture (orientation), elastic strain tensor, and ultimately the Nye or dislocation tensor. If the phase of the sample is already known, four reflections formally determine the deviatoric elastic strain tensor and the local crystal orientation.[19] If the hydrostatic strain is also needed, a single measurement of the energy of one Laue spot can be used to determine the volume of the unit cell.[19] To determine the Nye (or dislocation) tensor, multiple voxel measurements are required so that elastic strain gradients can be separated from the unpaired (or geometrically necessary) dislocation density.[20]

Special instrumentation and software is essential to realize the true power of polychromatic microdiffraction. Figure 3 shows the essential elements needed for general purpose 3-D polychromatic microdiffraction. Station 34-ID-E at the Advanced Photon Source is the only 3-D X-ray crystal microscope currently in operation. This beamline uses a type A undulator to produce a tunable spectrum with intense peaks on even and odd harmonics. For polychromatic measurements, the undulator axis is pointed off the beamline axis so the spectrum has less intense variations. Figure 4 illustrates the intensity spectrum from the undulator A in either on-axis or off-axis configurations and compares it to other sources. As illustrated, the undulator provides a very brilliant X-ray source for monochromatic experiments and is still approximately two orders of magnitude more brilliant over a wide bandpass than second generation sources.

The polychromatic X-ray beam from the undulator is focused onto the sample with a pair of differentially deposited Kirkpatrick–Baez mirrors.[21,22] These mirrors typically focus the beam to about a 500-nm spot size at the sample. The overlapping Laue patterns from the crystalline voxels along the beam path are recorded with a Roper 2 K × 2 K X-ray sensitive CCD located at 90 deg to the incident beam (Figures 2 and 3). The

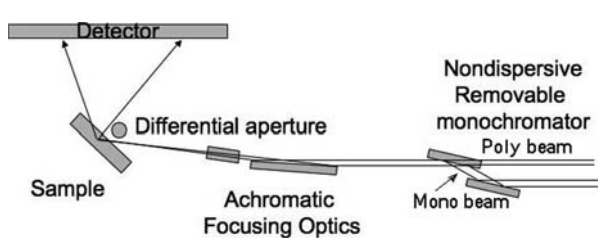

Fig. 3—Schematic of the essential elements for polychromatic microdiffraction. These include (*a*) a nondispersive monochromator that can be inserted into the beamline, (*b*) achromatic focusing optics, (*c*) a precision stage for positioning the sample, (*d*) a differential aperture, and (*e*) an X-ray sensitive area detector.

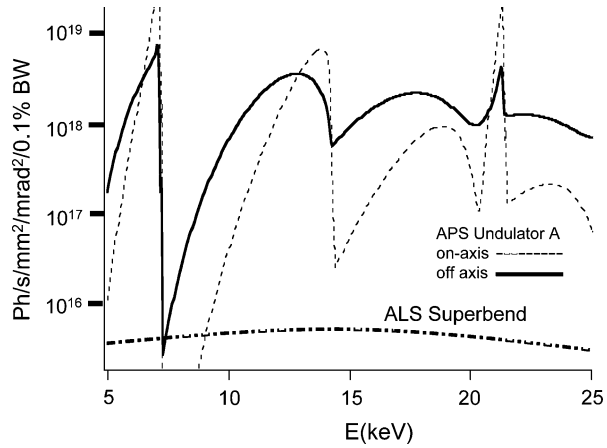

Fig. 4—Comparison of the brilliance for an APS type A undulator to the brilliance of an ALS superbend. On axis, the peak brilliance is about three orders of magnitude higher. Off axis, the average brilliance is about two orders of magnitude higher.

detector has about a 4.5-second readout for each image in full frame mode. With binning, the readout speed can be improved to about 2 seconds, but further improvement is not possible due to inherent delays in the CCD software of the current detector. Exposure times for samples can vary from 0.01 to a few seconds depending on the strength of the sample Laue reflections, and at least an order-of-magnitude greater flux can be focused on the sample with more perfect mirrors for 10 times shorter exposure times. Differential aperture microscopy is currently performed using a single wire (Figure 2) that scans parallel to the sample surface.[18]

With these tools, there are three kinds of experiments that are commonly used. In the simplest experiments, the Laue patterns form thin films and their substrates are measured. These measurements can cover fairly large areas, because there is usually no need to disentangle the Laue patterns of the film and substrate. The time required for data acquisition is usually about 10 to 100 ms depending on the sample, so data collection is dominated by the CCD readout time (2 to 4.5 seconds). With thin film samples, measurements with a VGA quality picture (640 × 480 pixels) can be collected in about a week.

With 3-D measurements, the data collection is even slower. Typically, about 400 images are required to reconstruct about 40 voxels along a beam path. Because the differential-aperture wire is moved while everything else remains stationary, each measurement of a line through the sample is called a wire scan. Somewhat fewer than 400 images can be used in each wire scan, but then the reconstructed Laue spots do not cover as large an angular range and the deviatoric strain tensor information is compromised. As a result, a 3-D map with 10 × 10 × 40 voxels of local crystal information takes about 24 hours.

The most difficult scans are combined monochromatic/wire scans that measure the diffraction *vector* and origin of Laue spots.[23,24] Here, the actual counting time can begin to contribute to the overall data collection time, and even simple line scans and area scans within a

sample are tedious. On the other hand, monochromatic/wire scans provide unambiguous information about the local crystal orientation and *d* spacing and have been used to demonstrate 3-D elastic/plastic strain in a single crystal[23] and to study the accommodation of strain in dislocation cells.[24] As we describe in Section III–B, the limitations of polychromatic microdiffraction should improve dramatically in the near future.

III. ADVANCES IN MEASUREMENT SPEED AND RESOLUTION

A. *X-ray Spatial Resolution*

Although 500-nm spatial resolution is sufficient for many experiments, the intrinsic limit of the approach is set by the diffraction limit, which for total-external-reflection mirrors is near 20 nm. Yamauchi *et al.*[25] have demonstrated focused monochromatic beams of ~24 nm, and Hignette *et al.*[26] have demonstrated wide bandpass beams below 100 nm. Recently, Liu *et al.*[27] have demonstrated polychromatic beams suitable for polychromatic nanodiffraction with dimensions below 80 nm. To exploit the achievable spatial resolution of high-performance reflective optics, a second optical platform has been installed in station 34-ID-E with the goal of mounting a polychromatic nanodiffraction capability. The plan is to use special prefigured mirrors from Tinsley[27] in a nested geometry. The nested or confocal geometry (Figure 5) is used by Osmic[28] to focus X-rays with multilayers. This geometry has major strengths and weaknesses compared to a standard KB mirror system. Advantages are the ability to collect a larger beam divergence (lower diffraction limit), a better geometrical demagnification for one axis, and the ability to use mirrors with nearly identical figures. The main drawback is that all the X-rays strike near the corner between the two mirrors (Figure 5). Thus, the mirror perfection needs to be best at the edge of at least one mirror where polishing usually produces edge errors.

We intend to cut superpolished monolithic mirrors along their length so the reflective surface comes from the center of the original polished mirror surface. The mirrors will then be assembled, as shown in Figure 5(b) or (c). Assuming that the cut mirror surfaces do not deform, metrology predicts that the existing prefigured mirrors are capable of producing ~40-nm beams. Of course, serious issues with beam stability must also be addressed to realize the promise of the existing mirrors.[29] Ultimately, however, this is only a first step toward far better spatial resolution. More accurate mirrors and more complicated geometries can push the diffraction limit to 11 nm or even in some cases to a few nanometers.[8]

In addition to improved diffraction-limited spatial resolution, there are some experiments where the tails of the intensity distribution are important. For example, in diffuse scattering experiments from polycrystalline samples, it is important to minimize the signal from adjacent grains, because the Bragg reflections of adjacent grains are many orders of magnitude more intense than the diffuse scattering signals.[30] The tails of the focal spot can ultimately be traced back to the abrupt termination of the beam profile focused into the focal spot. Abrupt terminations almost always lead to tails in diffraction scattering, as recognized in crystal truncation rods. A possible solution is the use of "apodizing," where the beam intensity profile is tailored prior to focusing. This offers the chance to significantly improve the ratio of the intensity at the focus to the intensity in the tails of the focus (Figure 6).

B. *X-Ray Data Acquisition Speed*

As described in Section II, a major limitation of the current hardware is the readout rate of the CCD detector. We have recently begun procurement of an X-ray sensitive area detector system with ~60 times faster data acquisition. This system consists of three amorphous Si detectors with dedicated electronics and

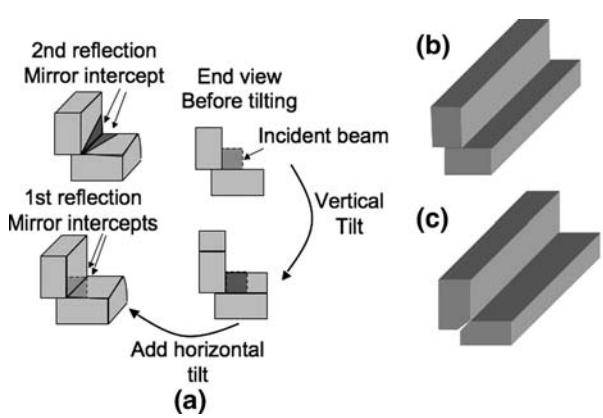

Fig. 5—(*a*) through (*c*) Nested (confocal) Kirkpatrick–Baez mirrors reflect some rays from the horizontal mirror and some rays from the vertical mirror first. The mirrors have the same geometrical demagnification and can collect a larger divergence in both directions than sequential elliptical mirrors. Two ways to assemble mirrors are shown in (b) and (c).

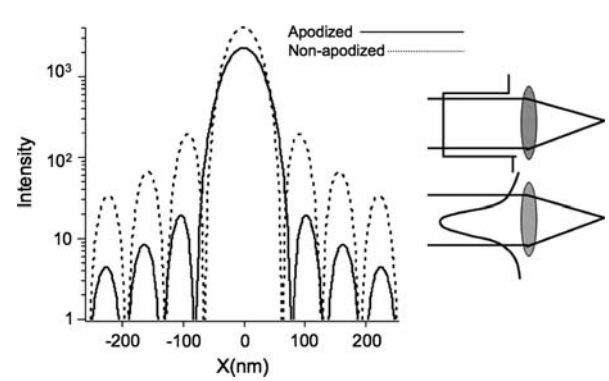

Fig. 6—The near-focal-spot intensity from an ideal focusing lens shows large tails near the focus due to the abrupt termination of the beam divergence onto the focal spot (dotted line and the top right figure illustrating the focused beam intensity profile). Even a simple apodization reduces the diffraction tails of a focused beam by a factor of 5 compared to the peak intensity (solid line and bottom right figure). More efficient apodization profiles are possible.

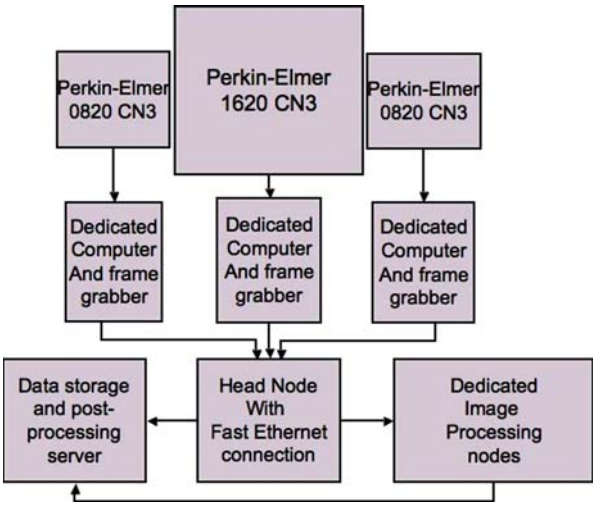

Fig. 7—Fast image collection system includes three detectors with dedicated frame grabbers linked to a dedicated cluster for real-time data analysis.

data analysis hardware (Figure 7). The detectors are capable of reading out at a maximum of 15 frames per second (fps), and the detectors will cover a larger solid angle than our current system with 1.5 times the pixels. Because of the huge amount of data that will be generated, a dedicated computer cluster will be used to reconstruct the voxel-by-voxel images and determine the local crystallographic information.

Although the detector system currently in procurement will have a tremendous impact on polychromatic microdiffraction research, even more powerful systems are on the horizon. For example, efforts at BNL lead by Peter Siddons[31] and at LLNL lead by Howard Padmore[32] are projecting 2 K × 2 K X-ray sensitive area detectors with 100 to 200 fps readout rates. Such systems offer other advantages including lower noise, greater X-ray sensitivity, and the potential for energy discrimination. If parallax and pixel position accuracy can be preserved through an X-ray converter, than this next generation of detectors can push the data acquisition speed of polychromatic X-ray microdiffraction by an additional order of magnitude.

In addition to faster detectors, there are other ways to accelerate 3-D polychromatic microdiffraction measurements. For example, studies of multiple wire differential aperture geometries suggest that a system with virtually identical performance to our current single wire system can operate with ~2.5 times fewer images.[33] In such systems, the sample angle relative to the X-ray beam is lowered to about 30 deg, which has about a 10 pct impact on maximum depth, but allows for the use of multiple wires and reduces the number of steps required to decode the overlapping Laue patterns. Additional gains can be made by using both the leading and trailing edges of the wire.

Even more ambitious differential-aperture approaches, with data-collection-time reductions of an order of a magnitude, are possible using coded aperture techniques or successive approximation approaches. Here, the main complication is the energy dependence of absorption in patterned films. If signal-to-noise is sufficient *and* if the software can accommodate the energy-dependent absorption, than data collection can at least in principle be accelerated by almost four orders of magnitude with a 200 fps detector with 10 times fewer scan steps. This will allow for VGA quality depth resolved images in around 17 hours. This would allow for a new class of experiments where unusual features in the sample are discovered *after* an initial sample survey. Of course, such revolutionary methods will require on-the-fly scans and improved mirror performance to reduce optimum image exposure times to ~5 ms, but both steps are reasonable and are under development. In addition, completely new approaches will be required to visualize and understand the massive amount of information represented by the 3-D distributions of tenor fields collected through polychromatic microdiffraction. This visualization and analysis effort will be driven in part by a growing polychromatic-microdiffraction community. For example, 2-D polychromatic microdiffraction facilities are operational at the Advanced Light Source, the National Synchrotron Light Source, the Canadian Light Source, and the Swiss Light Source. Other facilities are planned for Soleil, the European Synchrotron Radiation Facility, and the Australian Light Source.

IV. NEUTRON POLYCHROMATIC MICRODIFFRACTION OPPORTUNITIES

Neutrons offer important additional opportunities for experiments based on polychromatic microdiffraction. In particular, neutrons are sensitive to magnetism, can scatter strongly from low-Z materials, are gentle on delicate crystal structures, and can penetrate deeply into samples or through environmental chambers. These valuable properties align with a number of critical materials questions. However, even the most powerful neutron sources are inherently weak compared to third-generation synchrotron sources with ~12 orders of magnitude lower brilliance and a cost of about 13 orders of magnitude more per neutron; ~$\$10^{-3}$ per neutron *vs* $\$10^{-16}$ per X-ray. By fully using the available neutron bandpass and by using large divergences, the cost per neutron can be reduced to a more practical ~$\$10^{-9}$ per neutron, but clearly neutron sources must be efficiently used to exploit their inherent potential.

Achromatic neutron supermirror focusing offers a practical way to focus wide-bandpass neutron beams to micron dimensions. Small neutron beams are essential for heterogeneous samples or for small samples in environmental chambers. Recent experiments have demonstrated efficient neutron focusing to ~90 μm with simple KB mirror optics, and mirror optics with a goal of 25 μm are now under development. Again, nested Kirkpatrick–Baez mirrors offer the ability to collect larger divergences, which is important to maximize the signal. A prototype nested KB mirror system for the spallation neutrons at pressure (SNAP) beamline is shown in Figure 8.

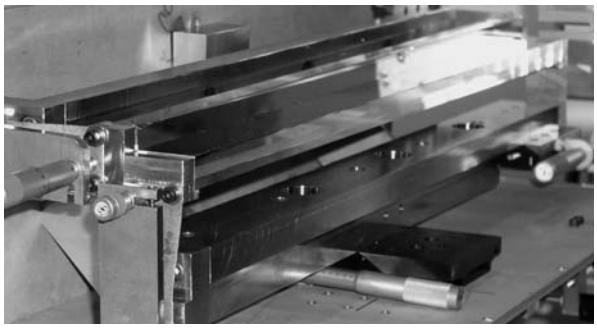

Fig. 8—Nested neutron supermirrors for the SNAP beamline.

Even without the nesting geometry, small-beam KB mirrors have been used for demonstration experiments on small single crystals and high-pressure samples. For example, the polychromatic thermal neutron flux from the Chalk River reactor was used to study small single crystals with a sub-100-μm beam.[34] These experiments illustrate the ability to study crystal structure with vastly improved signal-to-noise for small samples imbedded in an environmental chamber. The further improvement of signal-to-noise anticipated with energy-sensitive area detectors will allow for the study of even smaller samples, which is driving the development of even better neutron focusing optics.

In addition to samples with small-scale heterogeneities and samples that are inside environmental chambers, polychromatic neutron microdiffraction is very important for fragile crystal structures, particularly structures that are difficult to produce in large sizes or which may be dangerous to handle when in large volumes. For example, recent X-ray experiments have been able to study defects in grains of polycrystalline samples where large beams would have introduced an unacceptable overlap of diffuse scattering patterns from neighbor grains. These measurements were made to illustrate how activated samples could be studied in small volumes that would be difficult or dangerous to study as large single crystals. In related experiments, small explosive microcrystals were studied. However, these degraded in the X-ray beam—even with cryocooling—before the defects could be measured. With neutrons, the radiation damage problem will be vastly lower, which will allow for explosive, reactive, or otherwise X-ray sensitive materials to be probed.

A first application of polychromatic microdiffraction for neutrons is anticipated for the SNAP beamline at the Spallation Neutron Source. This beam will use focusing optics to improve the signal-to-noise for very small high-pressure samples imbedded in diamond anvil and other load cells. Polychromatic methods[35] are ideally suited for neutron studies of high-pressure samples, and beams of 25 to 100 μm will allow for measurements on samples at pressures similar to those used at synchrotron radiation facilities. Compared with X-ray measurements, the *spallation* neutron experiments will have the advantage that no energy scans are required.[34]

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