3.4 X-ray Optics Fabrication and Metrology

The APS users have continued to avail themselves extensively of our capabilities in the areas of metrology, thin film deposition, crystal fabrication, and mirror design. New capabilities have been acquired and improvements have been made to further enhance the range of x-ray optics that can be made and characterized for users. An atomic force microscope (AFM) has been commissioned, and its operation has been steadily improved to allow characterization of surface roughness at the Angstrom rms level. A vestibule was constructed in the metrology clean room that served to improve the environmental isolation of this microscope (and the entire metrology lab). This was needed because the AFM was found to be extremely sensitive to environmental conditions. A facility improvement was also made in the main polishing room. Cleanliness in this room was much improved by simple changes in the floor and ceiling materials and with the implementation of dust isolation. These improvements were effective in significantly reducing the occurrence of fine scratches in the polished crystal surfaces. A CCD camera has recently been procured for use in x-ray topography. Heretofore the optics fabrication and metrology (OFM) group has used film, and the CCD detector was obtained with the intent both to speed up data taking and to facilitate quantitative analyses of strain in fabricated crystal optics. Also a specialized dicing saw has been procured, which will be used for separating wafers into chips after x-ray lithography and subsequent wafer-level device processing has been performed. This saw will be useful for separating micro- and nanomachines made on large wafers. The saw will also be useful for the sawing of fine grooves for strain release of bent x-ray optics. Improvements in the thin-film deposition facility were made. Software to permit long deposition runs to be automated has been implemented. Multilayers with more than 200 individual layers were made in this way. Reactive sputtering for the deposition of TiO_2 has recently been accomplished by the addition of a simple gas-handling apparatus. Furthermore, a vacuum heater was added to permit deposition by evaporation, as well as by sputtering. A stand-alone x-ray reflecto-meter has been fully commissioned and is being used to calibrate layer thicknesses using the software package known as IMD (Windt, 1998). A mirror for SRI-CAT sector 4 has been designed. This mirror required special design considerations because the beamline employs two undulators that are not in line and are intended for separate beamline paths. The beamline layout is such that beams from both undulators impinge upon the main heat-load mirror simultaneously; this fact complicated the mirror design considerably. A water-cooled x-ray monochromator to reflect and render useful off-axis radiation from an undulator beamline has also been designed. Finally, an international workshop to chart out the future of metrology for x-ray and neutron optics was held at the APS.

3.4.1 X-ray Optics Metrology Laboratory

The x-ray optics metrology laboratory is located in a 10,000 Class clean room in the APS experiment hall and houses four different instruments that are fully operational:

1) a long trace profiler (LTP) for measuring slope error and curvature of mirrors up to 2 m in length (Takacs et al.,1999; Assoufid and Her, 1999),

2) a TOPO3D/2D surface roughness microscope that has a height resolution on the order of 1 Å resolution rms,

3) a WYKO-6000 surface profiler for measuring figure error with a maximum aperture of 150 mm and accuracy and repeatability of 1/100 λ and 1/200 λ, respectively,

4) an Explorer atomic force microscope.

During the last two years, we handled 161 metrology measurements. This grand total is broken down as follows: 27 for SRI-CAT, 3 for MU-CAT, 3 for MHATT-CAT, 2 for IMM-CAT, 3 for CMC-CAT, 4 for MR-CAT, 6 for BESSERC-CAT, 5 for CARS-CAT, 3 for SBC-CAT, 2 for COM-CAT, and 16 for UNI-CAT. Also included are 59 measurements performed for the OFM/UPD group that were indirectly for the benefit of the APS users, and 28 measurements performed for non-APS users including BNL, ESRF (Optics Group), Beamline Technology Corp., and the ANL Chemistry Division.

3.4.1.1 Recent enhancements in the metrology cleanroom

Several features were recently implemented to enhance the metrology laboratory environment and measurement accuracy:

1) A vestibule was constructed at the entrance of the metrology cleanroom.

2) A control box was added to allow one to adjust airflow speed, as well as temperature, from the inside of the cleanroom. The airflow velocity can now be lowered to minimize airflow-induced vibration during mirror measurements while still maintaining adequate cleanness of the room. The vestibule provides an additional barrier against dust particles when the airflow velocity is reduced or when the airflow system is completely shutdown.

3) All of the instrument computers are now connected to the APS network, and a single printer located outside of the main cleanroom is used, thus eliminating printer paper, which was one the major source of dust particles.

A mapping of particle measurement performed within the cleanroom, before and after modification, showed a substantial reduction in particle count.

3.4.1.2 Mirror metrology

Over 200 optical measurement requests have been handled since the operation of the metrology laboratory began. The measurement requests included beamline mirrors and mirror bender assemblies, as well as a variety of small components and substrates. The chart in Fig. 3.30 shows the evolution of optical measurement handled at the metrology laboratory since 1996, with a constant increase in the total volume of measurement. Requests related to beamline mirrors and mirror-bender assemblies make up about 29% of the total. Mirrors are typically 1 m long, and the most common substrate materials are silicon and ULE, and their optical quality has constantly improved during the last seven or eight years.

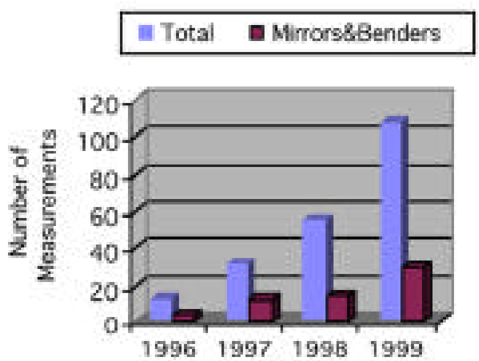

Fig. 3.30. Volume of metrology measurement requests handled over years. The total number of measurements is indicated in blue, and the volume of the mirror- and mirror-bender-assembly related measurements is shown in red.

Nowadays mirrors with surface roughness and slope error on the order of 1.5 Å rms, and <2 µrad rms, respectively, can be easily obtained. The most common substrate (39% of the ones we characterized) is silicon. The pie charts in Fig. 3.31 show the distribution of suppliers of mirrors used at the APS.

The large majority of the evaluated components were from APS CATs and users, but requests have also been handled for other non-APS users, as well as for

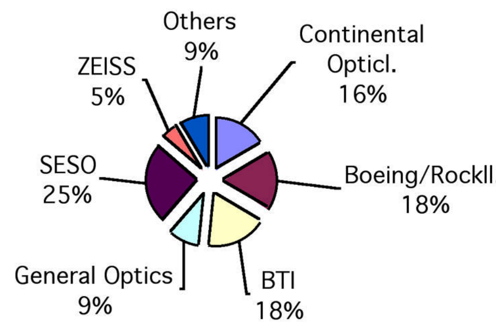

Fig. 3.31. Suppliers of APS mirrors evaluated at the metrology laboratory.

private businesses, such as BTI and SSG corporations. In particular, the APS metrology laboratory has supplied crucial measurement data for developing a Kirkpatrick-Baez (KB) mirror in collaboration with Oak Ridge National Laboratory (ORNL) and BTI (G. Ice et al., 2000).

Figure 3.32 shows a GlidCop mirror from UNI-CAT being carried to the TOPO3D/2D instrument for surface roughness evaluation. It is the largest (1.4 m long) and heaviest (150 kg) mirror to be evaluated. The mirror width (180 mm) allows the user to intercept a 6-mrad x-ray beam fan from the 33-BM beamline. Because of its size and weight, special care was necessary during metrology measurement.

3.4.1.3 Atomic force microscope

The AFM is a very useful metrology tool because it can probe features with sizes and a range of spatial wavelength that are relevant to x-rays. It is particularly useful in studying thin films and in the development of multilayer optics.

Fig. 3.32. Photograph of the UNI-CAT 1.4-m-long water-cooled GlidCop mirror being moved under the TOPO3D/2D system for surface roughness evaluation. The mirror is 180 mm wide and weighs 100 kg and so requires careful handling. It will be used for focusing a 6-mrad beam from the 33-BM beamline

At the APS metrology laboratory, an AFM Explorer system is used, and we have seen a growing demand in measurement requests with this instrument. It is a stand-alone unit that can be used to evaluate surface topography of samples up to 2 cm x 2 cm area, and it has two different scanners that can probe areas of 130 µm x 130 µm, and 3µm x 3 µm, with a height range of 12 µm and 0.8 µm, respectively. The system works either in contact or noncontact modes. The noncontact mode is very important when evaluating optical surfaces.

Figure 3.33 shows AFM images of a C/W/Si test sample at the OFM deposition facility. The sample is to be used to study the growth of C/W mutilayers for hard x-ray use. The AFM image revealed a structure that cannot be seen by an optical interference microscope.

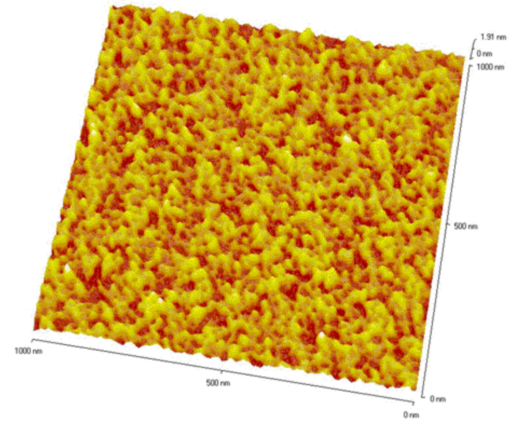

Fig. 3.33. An AFM image of a C/W/Si test sample. The image was taken using a Topometrix Explorer system equipped with a 12-µm Z scanner. The sample has a root mean square surface roughness of 0.182 nm. The AFM revealed details that cannot be resolved with an optical interference microscope.

3.4.2 International Workshop on Metrology for X-ray and Neutron Optics

A two-day International Workshop on Metrology for X-ray and Neutron Optics, the first of its kind, was held at the APS on March 16-17, 2000, and was attended by 64 world-class scientists and engineers, from government laboratories around the world. Several corporate representatives were also among the attendees. The workshop announcement, schedule, and corporate sponsor information are posted on the web at www.aps.anl.gov/conferences/xnom/

Workshop topics and discussions centered around state-of-the art and future needs in metrology instrumentation and techniques used to characterize x-ray and neutron optical substrates, particularly long grazing-incidence mirrors, supermirrors, and multilayers for use in x-ray synchrotron radiation, x-ray FEL and neutron beamlines.

The workshop included formal presentations, as well as informal presentations and group discussions, which allowed tackling a broad area of topics and issues. Among the main topics were the long trace profiler, stitching interferometry, metrology and mirror characterization with x-ray synchrotron radiation, standard issues, and metrology environment requirements.

3.4.3 Deposition Laboratory

Since September 1998, we have made over 370 regular depositions, and more than 500 mirrors and experimental samples have been made in this period. Among them over 160 Au/Cr and Au/Ti on Si samples were fabricated for SRI-CAT x-ray lithography experiments. Other coatings include: Ti, Cr, Al, B_4C, etc., on Si_3N_4 membranes and Be windows for SRI and UNI CATs; Cr on Mylar high-pressure ion chamber windows for ANL-Physics; Al, B_4C/Al, MgF_2/Al on Si visible light reflective mirrors for the undulators in the APS low-energy undulator test line (LEUTL) project; Au and W on Si_3N_4 membranes and Ti on Be x-ray beam detectors for SRI CAT; Al/Cr, Pd/Ti, and Pt/Cr on Si, ZERODUR or glass harmonic rejection mirrors for SRI, IMCA, Bio, and BESSRC CATs; and Rh/Cr, Pt/Cr mirrors for PNC and GSE-CARS CATs.

Additionally, multilayers for sagittal focusing and complicated multilayer structures, such as laterally graded and multistrip multilayers, have been developed. Reactive sputtering has been implemented to make compound thin films such as TiO_2. ^{57}Fe waveguides using multilayer structures have been fabricated in the large sputter deposition system by incorporating a house-invented precision-temperature-controlled evaporator into the system.

Both the large and small deposition systems have been fully automated. An electronic circuit coordinates the sputter-gun power supplies and the substrate movement. It turns the gun on and off at a desired moment for each layer deposition. In the old process, the guns were on all the time. This automation allows us to make more uniform multilayers, save target materials, and fabricate multilayers with larger number of bilayers. An example of the control that we have achieved is shown in Fig. 3.34, which is a transmission electron micrograph of a multilayer structure consisting of 100 layers of W and 100 layers of C (Courtesy of M. Kirk, R. Csencsits, and R. Cook at ANL/MSD). The period thickness is 25.3 Å, and the peak reflectivity is 78% (Macrander et al., 2000). The regularity of the layer thickness and the low roughness are seen to be very good. This deposition control is only possible after repeated calibration from preparatory growths. The thicknesses are then measured rapidly with a stand-alone x-ray reflectometer. Example data for a specular reflection scan are shown in Fig. 3.35. The accompanying simulation was obtained using the IMD software package (Windt, 1998). This figure shows data for a specular scan (in red) and a simulation (green line).

This example is chosen because i) the data exemplifies data obtained with the reflectometer, ii) the thickness of the W layer invoked for the simulation, 0.53 nm, is near the limit of our technological ability, iii) the incomplete agreement between the simulation and the data and the fact that the invoked rms roughness, 0.5 nm, is roughly the same as the W layer thickness is taken to mean that the modeling software needs to be improved/updated.

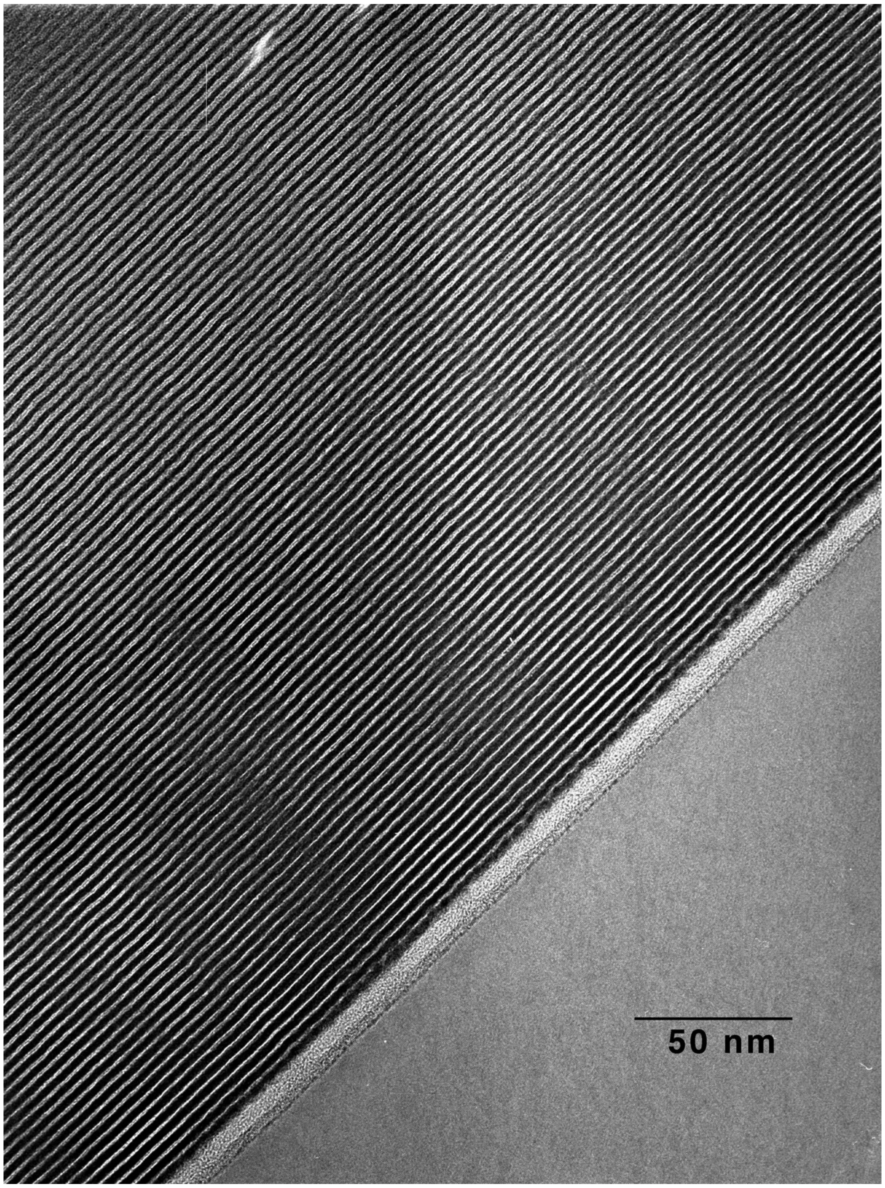

Fig. 3.34. A transmission electron micrograph of a multilayer structure consisting of 100 layers of W and 100 layers of C (courtesy of M. Kirk, R. Csencsits, and R. Cook at ANL/MSD).

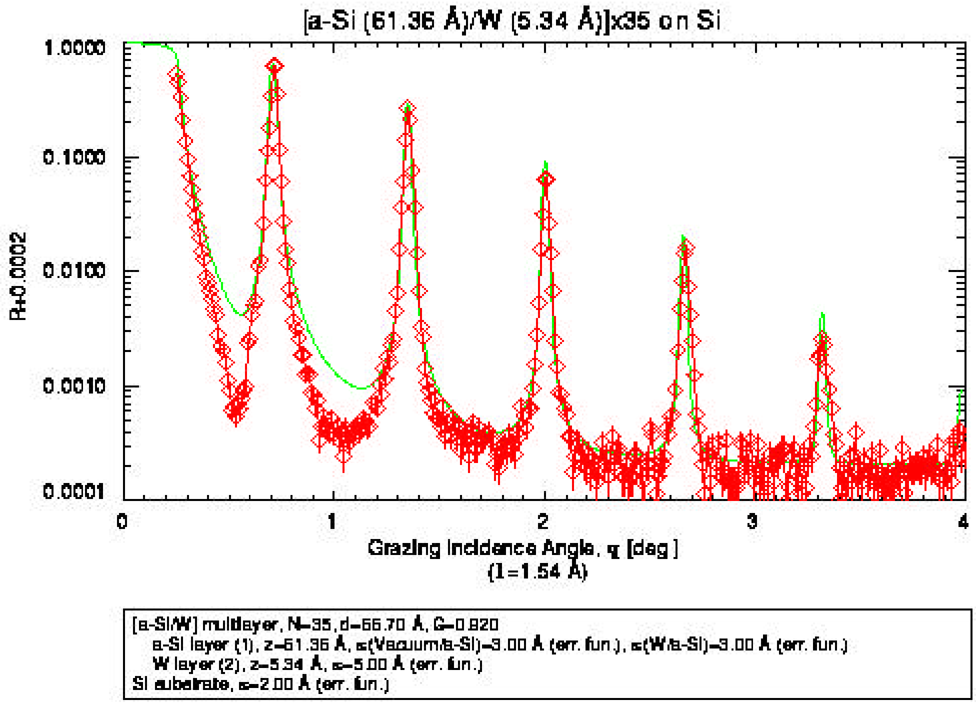

Fig. 3.35. Example data for a specular scan (in red). The accompanying simulation (green line) was obtained using the IMD software package (Windt, 1998).

Recently, we have designed and installed a vacuum heater in the large deposition system. We made novel use of permanent magnets to overcome the challenge of mounting vacuum heaters in an existing vacuum system. Vacuum heaters are important for sample annealing and vacuum baking.

3.4.3.1 Microfocusing mirrors

Kirkpatrick-Baez grazing-incidence mirrors are widely used at the APS as micro-focusing optics. We have coated ~100 of these mirrors for our users. Most mirrors consist of 5 nm Cr and 40 nm Rh (or Pt, or Pd) on Si single crystals or float glasses. We have studied the film growth and roughness of these systems using *in situ* spectroscopic ellipsometry measurements. We found that too thick a Cr underlayer will increase the film roughness, while a 5 nm Cr underlayer will provide both a good adhesion and a smooth surface (Liu et al., 1999). Good results have been obtained for these mirrors. CARS-CAT demonstrated that a system of two 100-mm-long, actively bent mirrors in a KB arrangement could achieve a double-focused beam of 0.8 μm x 0.85 μm and flux density gain greater than 10^5 from 10 keV undulator x-rays. Figure 3.36 shows their focusing results using a fluorescence knife-edge coated at the APS deposition lab. Other CATs, such as PNC-CAT and UNI-CAT, are also using KB mirrors coated at the deposition lab.

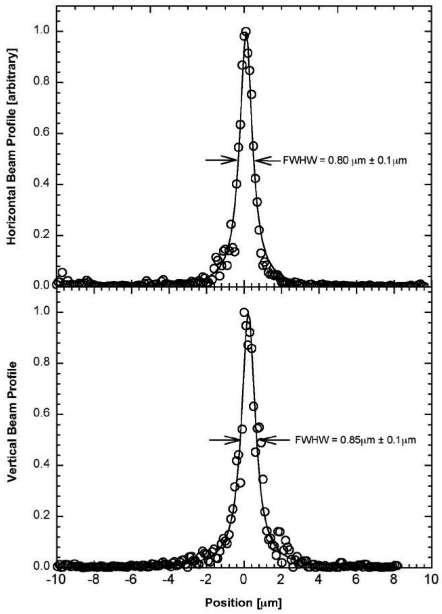

Fig. 3.36. A doubly focused 10 keV undulator x-ray beam obtained from actively bent KB mirrors by CARS-CAT.

3.4.3.2 Graded multilayer deposition

Laterally graded multilayers consisting of uniform W layers and wedge-shaped C layers have been made at the deposition lab for tunable x-ray double-monochromator applications in collaboration with HASY-LAB. The double monochromator has two identical graded multilayers in series, as in the conventional double-crystal mono-chromator arrangement. By letting the x-ray beam hit slightly different (bilayer) d–spacings on each multilayer, one can adjust the bandpass and peak energy of the transmitted beam. Also, since the Bragg angles of the two multilayers are not constrained to be the same, the angle of the transmitted beam can be varied in the vertical plane. This option may be an attractive alternative to the conventional way for studying liquid surfaces in reflectivity and grazing-incidence diffraction measurements.

The films were made by DC magnetron sputtering with the sputtered atoms passing a contoured mask while the substrate was moving. Two different masks were designed to produce either a uniform or graded thickness profile. The mask was fixed on the top of the shielding can of the sputter gun. To determine the shape of these two masks, a thickness profile was measured for a film deposited without any masks installed. The results were compared with that of theoretical calculations of thickness distribution. The mask was then designed to appropriately increase or reduce the flux of the sputtered atoms along the axis perpendicular to the moving direction of the substrate.

Two identical graded multilayer samples were made in the small deposition system for testing at HASYLAB in a double-crystal monochromator arrangement. The multi-layer comprised 60 bilayers of W and C on 100 x 25 x 3 mm float glass with a d-spacing varying from 35 to 60 Å and an average gradient of 0.27 Å/mm along the long direction. The W layer was uniform, while the C layer thickness was graded. Figure 3.37 shows the bilayer spacing as a function of lateral distance as measured from x-ray diffraction data (Macrander et al., 1999).

In addition to W/C, Mo/Si and W/Si graded multilayers have also been fabricated. Graded multilayers have many other novel applications. For example, test samples have been provided to Bio-CAT for x-ray fluorescence detection experiments, and experiments with x-ray standing waves have also been planned.

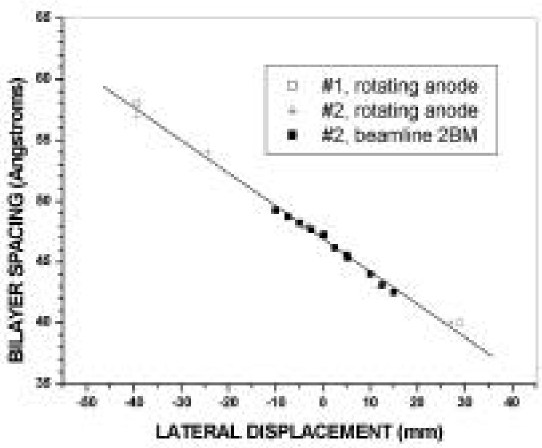

Fig. 3.37. Combined data for the bilayer spacing as a function of lateral distance as measured from the x-ray diffraction data for a W/C graded multilayer.

3.4.3.3 Double-multilayer mono-chromator for beamline 2-BM

Four stripes of W/C and W/Si multilayers have been coated on two identical Si multilayer (ML) mirrors (100 x 145 x 50 mm) in the large deposition system. Each of the stripes on one of MLs pairs up with a matched stripe on the second ML in a (+,−) double-multilayer monochromator (DMM) configuration. The four stripes on each ML provide four different d-spacings. The stripes are 22 mm wide, coated on two mirrors in the same runs. The thick W/C strip (1.55 nm W / 2.15 nm C x 100 bilayers) has to be coated in two separated runs, with the C-gun cleaned in between, to prevent the C target from arcing. This strip needed a total of 17 hours of deposition. To coat every C layer in the W/C multilayer, the mirrors were moved over the C target 18 times during the coating to provide needed uniformity. Our fully automated system enabled us to accomplish all these smoothly. Test results showed a nonuniformity of ~5%.

3.4.3.4 Reactive sputter deposition

We have succeeded in reactive sputtering for the first time at the APS. TiO_2 films have been made in the large deposition system by sputtering a Ti target in a mixture of Ar and oxygen (85 Ar / 15 oxygen). Two MKS (2159) gas flow controllers together with a MKS (247) power supply (set point source) were used to control the ratio of Ar and O_2 flows. A MKS (250) pressure controller was used to control the total pressure. The gas pressure and composition were stable during the deposition. The TiO_2 films could also be annealed in an O_2 environment after the growth using our house-made vacuum heater.

Spectroscopic ellipsometry was used to measure these films. Our collaborators have shown that these thin TiO_2 films could be used to adsorb/desorb Sr ions as previously done using TiO_2 single crystals. This is important for future x-ray standing wave studies of electrical double-layer structure at the TiO_2-water interface using W/C multilayers.

We are now able to make complicated thin-film structures involving multilayers, graded multilayers, MBE-type isotope thin films, and compound thin films in the same vacuum chamber without the need to break the vacuum.

3.4.4 Fabrication Laboratories

The OFM fabrication laboratory continued to serve user communities of the APS. It has also proved possible to help other synchrotron facilities. The lab prepared new crystal elements (such as monochromators, in particular, cryomonochromators, interferometers, and diced analyzers) for x-ray beamlines and/or improved (i.e., reshaped, re-etched and repolished) crystals that had been already used on the beamlines. All together, in the past two years, the lab delivered to users 394 optical elements. Of these, 158 crystals were for SRI-CAT (including the old and new XFD, and UPD), 8 crystals were for Bio-CAT, 29 crystals for BESSRC-CAT, 2 crystals for IMM-CAT, 40 crystals for PNC-CAT, 14 crystals for MU-CAT, 4 crystals for MHATT-CAT, 12 crystals for UNI-CAT, 17 crystals for CARS-CAT, 38 crystals for DND-CAT, 17 crystals for IMCA-CAT, 2 crystals for CMC-CAT, 4 crystals for SBC-CAT, 3 crystals for HP-CAT, 1 crystal for MR-CAT, 3 crystals for MSD-ANL, 6 crystals for IPNS-ANL, 2 crystals for Physics Dept.-ANL, 2 manifolds for SSRL-Stanford, 6 crystals for Rockefeller University, 4 crystals for Albert Einstein College of Medicine, 16 crystals for Northwestern University, 2 crystals for University of Washington, and 4 crystals for LBL-Berkeley.

Parallel to production activities, some R&D projects were carried out.

1) The polishing room underwent essential reconstruction. A new entrance with a vestibule was added, walls were sealed along the floor, the floor was covered with special linoleum, and the ventilation system was enhanced and air quality improved. With all these modifications, the room was converted into a semi-clean room. As a result, we no longer have problems with scratches that previously often occurred on polished surfaces due to particles entering the room from the nearby loading dock.

2) The lab was equipped with a new polisher (Fig. 3.38) that was built by the Strasbaugh company according to our specifications. The machine is equipped with: a 36” polishing table that can be totally submerged into the polishing slurry, three motorized polishing rings of 12” diameter, and a motorized cutter for making groves in the polishing pads. It is possible, using this machine, to polish crystals (up to 12” dia.) in the so-called floating mode (crystals fully submerged in the slurry, no additional load imposed on the polished object). This type of polishing should result in surfaces of very low roughness and very high flatness. So far, using Rodel pads IC-1000 with a square pattern 0.25” x 0.25”, we were able to polish 4” dia. silicon slabs to a roughness of 3.25 Å rms and a flatness of $\lambda/3$.

Fig. 3.38. New polisher from Strasbaugh.

3) In order to enhance our capabilities of crystal cutting, a new saw (Kulicke&Soffa dicer Model 984-10) was purchased. After pre-installation preparations (bringing electrical power, water, sewer and compressed air lines), the machine was installed by the manufacturer. The machine will allow dicing of different materials (e.g., manufacturing of silicon-focusing analyzers and separation of micromechanical objects) and precise cutting of small, mainly silicon, crystals.

3.4.5 X-ray Characterization Laboratory

The x-ray laboratory continued to serve the APS users by offering them opportunities to test their crystals or thin layers deposited on different substrates. Available to any authorized user, a single-axis diffractometer, a Laue camera, and a double-axis diffractometer (all of which are installed at conventional x-ray generators—Rigaku and Spellman) were used to obtain the crystallographic orientation and to do further testing of numerous crystals. The instruments were primarily used for the needs of the fabrication laboratory. The main experimental activities in the x-ray lab were concentrated at the Topo Test Unit (TTU) and the triple-axis diffractometer installed at the rotating anode generator.

In the past two years, the TTU was used for 162 topographic test runs. Predominantly silicon crystals (monochromators and analyzers already manufactured, and sometimes ingots to be used for fabrication) but also 31 diamond crystals, were investigated. A majority of the runs were performed on samples for XFD and/or UPD. However, many crystals for UNI-CAT, IMCA-CAT, MU-CAT, IMM-CAT, CMC-CAT, COM-CAT, HP-CAT, SBC-CAT, CARS-CAT, Bio-CAT, and MHATT-CAT were also tested.

The triple-axis diffractometer was predominantly used for reflectivity measurements (all together 50 single- or multilayers were investigated) and for testing samples that were later used at the beamlines (all together about 18 different samples were checked). Most single-and multilayer samples measured in the lab were produced in the OFM deposition laboratory. Measurements supplied data needed for correction of deposition procedures or constituted testing of final products (e.g., four big 100 mm x 120 mm tungsten/carbon graded multilayers were tested that were used later in Denmark as monochromators). In addition, four beryllium single crystals were investigated under compressive stress. Stress was applied either in the c crystallographic direction (2 samples) or in the a crystallographic direction (another 2 samples). The measurements were carried out in the stress range 0 to 700 MPa using specially designed load frames. A typical triple-axis arrangement with a Ge (111) monochromator and a Ge (111) analyzer was utilized. Additionally, a CCD camera was used for image data acquisition. As the compressive stress on a sample increases, the single crystal fractures into smaller crystal grains of a very complex behavior. The stress-strain response exhibits strong anisotropy. Results are currently under evaluation.

Although there were no major changes to the equipment used in the x-ray laboratory in the past two years, some small developments are worth noting.

1) Operation of the Topo Test Unit was improved by adding new monochromators and new software that significantly accelerated sample orientation (finding the correct sample tilt position with respect to the monochromator crystallographic planes). Another major improvement of the TTU is to come soon. A new CCD camera (built to our specifications by Roper Scientific) was purchased. It will allow the collection and storage of digitized images. The camera was put into operation temporarily at the triple-axis diffractometer and used for Be experiments. Mounting of the camera on the TTU requires essential mechanical reconstruction of the diffractometer.

2) Upgrading of the triple-axis diffractometer continued.

- A new sample holder was constructed and built. It allows load frames (e.g., hydraulic press) or big slabs with deposited multilayers to be mounted in the center of the four-circle goniometer and translated in two directions normal to each other.
- A second detector that monitors the intensity of the monochromatic beam was added.
- The main detector subassembly was reconstructed. The analyzer table was improved and a platform for mounting a CCD camera was added.
- In addition to EPICS control, the diffractometer can now be run under SPEC control.