[illegible] CONF-870610--18

[illegible] 1 1987

LBL-24000
Preprint

Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

Accelerator & Fusion Research Division

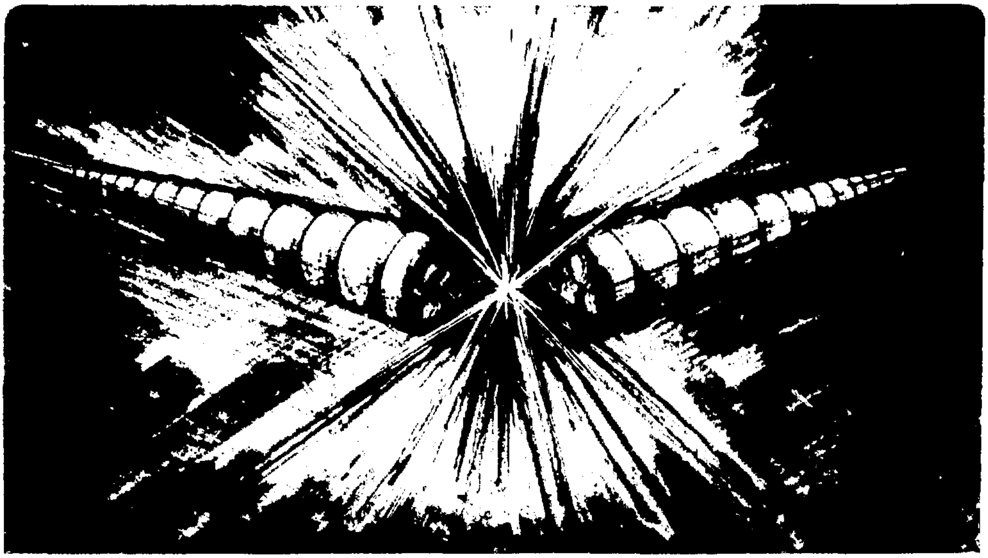
Center for X-Ray Optics

Presented at the 5th National Conference on
Synchrotron Radiation Instrumentation,
Madison, WI, June 21–25, 1987, and to be
published in the Proceedings

Multilayer-Coated Mirrors as Power Filters in Synchrotron Radiation Beamlines

J.B. Kortright, P. Plag, R.C.C. Perera,
P.L. Cowan, D.W. Lindle, and B. Karlin

July 1987



Prepared for the U.S. Department of Energy under Contract DE-AC03-76SF00098

LBL--24000

DE88 003872

MULTILAYER-COATED MIRRORS AS POWER FILTERS IN SYNCHROTRON RADIATION BEAMLINES

J.B. Kortright, P. Plag, and R.C.C. Perera

Center for X-Ray Optics

Lawrence Berkeley Laboratory

University of California

Berkeley, California 94720

P.L. Cowan and D.W. Lindle

Quantum Metrology Group

National Bureau of Standards

Gaithersburg, Maryland 20899

B. Karlin

National Synchrotron Light Source

Brookhaven National Laboratory

Upton, New York 11973

ABSTRACT

Multilayer-coated mirrors, rather than conventional total-reflection mirrors, have been proposed as a means to reduce power incident on the first optical element of high resolution monochromators. We have designed, fabricated, installed and characterized a multilayer pre-mirror specifically for the 800-4000 eV range for the X24-A bending magnet beamline at the National Synchrotron Light Source. Various aspects of this application are discussed, including power and thermal considerations, beamline layout considerations and constraints, choice of multilayer materials and substrate, and techniques to ensure lateral uniformity of the multilayer. Results of a preliminary characterization of a mirror coated with a SiC/V multilayer and installed in the beamline are also discussed.

1. Introduction

The fraction of the power radiated from a synchrotron radiation source incident upon downstream optical elements can be reduced by using a multilayer Bragg reflector with moderate resolution as the first beamline optical element, instead of a conventional total reflection mirror. This reduction in power can be achieved without significant loss in efficiency of the optical system over the limited bandpass of the multilayer, provided the multilayer Bragg peak is reflective enough. While this multilayer power filter concept has been mentioned in the literature [1,2,3,4], this work represents the first step towards implementing this concept in a synchrotron radiation beamline. Multilayer pre-mirrors may find utility in situations where thermal loading on the resolution-determining optical elements will otherwise impair performance. Such situations may develop at the next generation of high-brightness synchrotron radiation sources, and already prevail in certain cases at existing sources, even on bending magnet beamlines. One such case is in the 800-2000 eV photon energy range, where crystals of interest for high resolution monochromators (e.g. beryl, quartz, InSb) are damaged by excessive radiation [5,6].

We have investigated this multilayer pre-mirror concept for the X24-A beamline at the National Synchrotron Light Source (NSLS), with the goal of protecting radiation sensitive monochromator crystals in this difficult 800-2000 eV energy range. Even at photon energies above 2 keV, and using Si crystals (more thermally conductive than those mentioned above), time-dependent heating of the first monochromator crystal results in significant beam position motion as well as energy calibration shifts during the course of a fill. This experience, together with the known problems with crystals of interest for lower energies, has forestalled high resolution spectroscopy experiments using the more heat-sensitive crystals at this beamline. Here we report design considerations for this effort and preliminary results from a multilayer pre-mirror installed in the beamline.

2. Design of multilayer pre-mirror

The National Bureau of Standards beamline X24-A at the NSLS was designed to allow the installation of a multilayer coated pre-mirror [3], by making the entire downstream portion of the beamline pivot about the pre-mirror reflecting surface (see Figure 1). Many factors affecting the choice of the constituent multilayer materials and operating angular and energy ranges were considered in the design stages, and led us to investigate relatively low-Z material combinations of relatively large period for the multilayer pre-mirror coating.

The Bragg equation relates the multilayer scattering angle, the multilayer period and the wavelength. The desire to operate the multilayer in the 800-2000 eV energy region restricts the usable combinations of Bragg angles and multilayer periods, with limiting trends of small-angles/large-periods and large-angles/small-periods. The small-angle/large-period combination is favored, primarily because it provides lower dispersion, resulting in smaller angular displacements of the beamline downstream of the pre-mirror. However, for a multilayer of given constituent materials, operating in the small-angle regime provides less power filtering than does operating in the large-angle regime, where the multilayer Bragg peak and the critical angle for total reflection, θ_C, are better separated. A pre-filter with two, parallel multilayers would resolve this conflict between the desire for small angular beamline deflections and for maximized power filtering, but would suffer from reduced throughput due to the two reflections. Other advantages of operating in the small-angle/large-period regime include less sensitivity to interface roughness, lower absorbed power density at the pre-mirror, and the fact that the Bragg reflectivity of multilayers generally deteriorates as periods decrease beyond some lower limit.

The choice of constituent materials for the multilayer was driven largely by the desire to work at small angles and the optical properties of materials in the 800-2000 eV energy range. Relatively low-Z materials are appropriate for at least two reasons. First, low-Z materials have smaller θ_C compared to high-Z materials, since, neglecting anomalous dispersion, $\theta_C \propto Z^{1/2}$, and thus yield better separation of multilayer Bragg peaks from critical angles. Second, the use of low-Z materials for each of the two different layers results in a relatively small effective structure factor for the multilayer unit cell, which in turn yields relatively narrow multilayer Bragg peaks compared to the more traditional high-Z/low-Z material combinations often chosen for x-ray optical multilayers. Both of these factors result in better power filtering capability of the multilayer pre-mirror and are consistent with the desire to work at small angles.

Multilayers of several relatively low-Z materials combinations were investigated. These included: Ti/C, SiC/C, SiC/Si, SiC/V, SiC/Ti and Si/Mo. Calculations of expected performance, fabrication of multilayer test samples by magnetron sputtering, measurement of reflectivity at a variety of wavelengths, and comparison of measured performance with calculations were a part of this investigation. The SiC/V combination [7] gave the best performance in the energy range of interest, when considering both peak reflectivity and separation of the specular from the Bragg-diffracted radiations. The SiC/V multilayer fabricated for this application was designed to have 17 layer pairs of SiC and V with a period of 147 Å. The relative thickness of SiC to V was roughly 4 to 1, which yields the strongest first order Bragg peak in the energy region of interest. The multilayer was intended to have V as the topmost layer. However, during deposition of the final mirror, one of the sputtering targets arced and deposition stopped at a point such that 16 layer pairs of SiC and V, with SiC as the topmost layer, is a more accurate description of the multilayer deposited on the pre-mirror substrate.

Figure 2 shows the measured angular reflectivity at 1487 eV (8.34 Å) of a multilayer deposited as a witness sample during the same sputtering run when the pre-mirror was coated. The witness sample substrate was a super-polished quartz optical flat. Also shown is the calculated performance for the multilayer which assumes (as do all calculations in this paper) an ideal structure, with pure layers, described by bulk values of the optical constants, and with compositionally sharp and smooth interfaces. The measured multilayer features are in relatively good agreement with the calculation. The measured Bragg peak falls at the same position and has roughly the same width compared to the calculation (instrumental broadening is very small). The measured peak intensity is roughly 57% of that calculated. This is typical of relatively good multilayer performance in this energy region. Both the calculation and the measurement would show slightly increased reflectivity if the multilayer were fabricated with V as the topmost layer.

Thermal or power considerations for this application of a multilayer power filter are of two types. One concerns the effectiveness of the multilayer as a power filter compared to a grazing incidence mirror, and the other concerns the power absorbed at the pre-mirror and its implications. Of primary interest is the amount of power not passed by the multilayer pre-mirror that would be passed by a total reflection pre-mirror. Figure 3 shows calculated reflectivities $R(\hbar\nu)$ for the SiC/V multilayer and for the Ni-coated total reflection mirror which the multilayer was designed to replace. In these calculations the angles of each mirror are chosen for comparable reflectivity at 1500 eV and are typical of operating conditions at the beamline. The area above the multilayer reflectivity curve and below the Ni curve represents additional power filtered out by the multilayer. Using the calculated spectrum of an NSLS bending magnet with a horizontal acceptance of 9.5 mrad, 77 watts of radiated power is incident upon the mirror. Multiplying the calculated power spectrum by the two reflectivity curves in Fig. 3 predicts that the Ni mirror will reflect 11 watts while the SiC/V multilayer will reflect 3 watts. In general this power reduction depends somewhat on the energy and angular ranges of operation and on the coatings of the various mirrors. Thus, an order of magnitude reduction in the power passed by a multilayer pre-mirror, compared to a total reflection mirror, is a reasonable general estimate of the power filtering capabilities of a multilayer pre-mirror.

The second type of thermal consideration is the power absorbed by the multilayer pre-mirror and its possible detrimental effects on the beamline's optical performance. Considering that roughly 85% of the incident power is absorbed by the grazing incidence mirror in the situation described above, while roughly 95% of the incident power is absorbed by the multilayer mirror, we see that the increase in absorbed power in the multilayer case is not great. Even so, the question of heating in the multilayer pre-mirror deserves consideration. A real concern is the stability of the multilayer coating under prolonged exposure to absorbed radiation. Another is thermal distortion of the mirror surface, which can degrade performance of the optical system [8]. Both of these concerns argue for maximum mirror temperatures as low as possible during operation, and require decisions regarding the mirror substrate material and the method of drawing heat away from the mirror substrate. Another concern is the desire to bake-out the mirror at roughly 100°C upon installation. Calculations showed that for the case of the X24-A beamline, the mirror surface temperature during operation should not exceed this bake-out temperature for the mirror substrate system described below. Annealing studies of the SiC/V multilayer combination showed that 24 hours at 100°C does not degrade the optical performance of the multilayer itself. The multilayer Bragg reflectivity of the SiC/V combination remained unchanged after 8 months at room temperature. Higher power future synchrotron radiation beamlines may present significant multilayer stability problems.

The mirror substrate is shown in Figure 4. This OFHC copper substrate was coated with electroless nickel, and polished to have rms roughness of less than 10 Å for spatial wavelengths less than 5 mm, and to be flat to less than 1/20 of an optical wavelength for spatial wavelengths greater than 5 mm. This substrate was mounted onto a copper mating piece, which in turn was clamped onto a third copper piece through which cooling water can flow. Assuming conductive cooling through this copper pathway with several copper-copper interfaces, the maximum temperature of the multilayer coating was estimated to be no greater than roughly 70°C above that of the heat sink at the copper feed-through. The shape of the substrate was chosen with two considerations in mind. Mechanical deformation of the polished, coated optical surface due to mounting was considered. The design shown in Fig. 4 is an effort to isolate mechanical stresses of mounting from the mirror's clear aperture. This design also provides for side cooling rather than bottom cooling. This helps ensure a minimum temperature drop across the mirror substrate from top to bottom, which in turn minimizes thermal distortion along the length of the mirror which could impair optical performance.

The mirror's clear aperture of 5 inches by 2.5 inches required the development of the capability to deposit a multilayer with period uniform to 1-2% over this area. The sputtering system used for multilayer deposition contained 4 inch diameter sputtering targets which did not initially provide the required uniformity. This uniformity was achieved by introducing apertures between the stationary sputtering targets and the rotating substrates close to the substrates. By iteratively adjusting the shape of the aperture to compensate for the varying integrated deposition as a function of position on the substrate, and monitoring the actual multilayer period as a function of position by its many orders of Bragg reflection, a multilayer uniform to better than 1% in period was deposited onto the polished mirror surface. The mirror coated in this fashion was flat, though a collimating mirror with relatively large curvature could in principle be coated with similar uniformity.

3. Preliminary beamline characterization

The multilayer pre-mirror was installed in the X24-A beamline with the NSLS x-ray ring currents as high as 230 mA for roughly one month of operation.

The short time available for <u>in situ</u> studies did not enable a full performance evaluation of the multilayer coated pre-mirror. In particular, absolute reflectivities were not obtained, and we were unable to determine the ultimate utility of the multilayer pre-mirror for limiting the heating of radiation sensitive crystals.

Characterization of reflectivity as a function of energy at fixed angle was more easily accomplished than reflectivity as a function of angle at fixed energy. Figure 5 shows $R(h\nu)$ data in a higher energy range than that intended for the multilayer pre-mirror operation. Superimposed in Fig. 5 is the calculated $R(h\nu)$ for the second order multilayer Bragg peak. The measured $R(h\nu)$ has not been corrected for background or for varying detector efficiency with energy and is plotted on an arbitrary vertical scale. Since the second order peak is significantly narrower than the first order peak, this comparison strongly suggests that the multilayer on the pre-mirror was quite uniform, as the measured Bragg peak is not substantially broader than the calculated peak.

The temperature of the multilayer pre-mirror was monitored while it was in the synchrotron radiation beam. Two thermocouples were used, one placed at a lateral edge of the optical surface and the other on the bottom of the copper mirror substrate. With no water cooling, the maximum pre-mirror temperature was about 70°C with a ring current in excess of 100 mA. The temperature difference between the two thermocouples was no more than 1-2°C, which means that low thermal distortion occured along the length of the mirror with the side cooling substrate design in Fig. 4. When water cooling was applied to the pre-mirror through two copper-copper interfaces, the maximum temperature of the pre-mirror dropped to roughly 30°C. Downstream of the pre-mirror, it was noted that the increase in temperature of the first crystal in the monochromator was never more than roughly half of that observed at the same ring current when the Ni total reflection pre-mirror was installed.

After the multilayer pre-mirror was removed from the beamline, its surface roughness was measured with a WYKO optical profilometer [9] to be no more than 1-2 Å greater than that measured for the polished substrate before it was coated with the multilayer. This is evidence that the multilayer was not structurally damaged during its exposure to the synchrotron beam.

4. Summary

Progress has been made in demonstrating the feasibility of multilayer pre-mirrors as power filters in synchrotron radiation beamlines. Calculations show that multilayers can reduce the power incident on a high resolution monochromator from a bending magnet source by as much as an order of magnitude compared to conventional total reflection mirrors. The increase in absorbed power at the multilayer pre-mirror compared to a total reflection mirror is small. A prototype multilayer pre-mirror has been designed, fabricated, installed and tested in the X24-A bending magnet beamline at the NSLS. Techniques were developed to coat the entire clear aperture of the mirror with a multilayer whose period was uniform to better than 1 percent. The multilayer reflectivity was reasonably close to that calculated using a model of an ideal multilayer. In the beamline the temperature at the multilayer never exceeded roughly 70° C with no cooling water flow, and was considerably less with cooling water flowing. The multilayer remained undamaged after one month of exposure with the ring operating with currents as high as 230 mA. The temperature at the first Si monochromator crystal was reduced with the multilayer pre-mirror compared to the temperature using the total reflection mirror. These preliminary characterizations encourage further development of this multilayer power filter concept to ascertain whether this device will permit the use of more radiation sensitive crystals in the monochromator.

Future high power beamlines which might benefit from a multilayer pre-mirror should be designed with several considerations in mind. Most existing synchrotron radiation beamlines are constrained geometrically so that use of a single multilayer pre-mirror is not feasible. The ability of the entire beamline to pivot about the multilayer is of primary importance, but will not be compatible with certain experiments. Well-controlled, synchronous motions of the pre-mirror and beamline facilitate characterization and utilization of the device. As radiated power densities on the multilayer increase, questions of long term multilayer stability will become more important, as will designs for the effective removal of heat from the mirror that minimize thermal distortion. It should be noted that a multilayer pre-mirror will filter out a smaller fraction of the power from an undulator-based source because of the coincidence in the peaked structures in the multilayer reflectivity and source spectrum.

Acknowledgements

Discussions with M. Howells, E. Hoyer, J. Underwood and R. DiGennaro and A. Thompson are gratefully acknowledged. Partial characterization of the multilayers at the Lawrence Berkeley Laboratory was contributed by J. Kerner and B.L. Henke. This work was supported by the U.S. Department of Energy, Office of Basic Energy Science, under contract No. DE-AC03-76SF00098. Work done at NSLS was supported by the U.S. Department of Energy under contract No.

DE-AC020-76CH00016.

References

1 W.K. Warburton, Z.U. Rek and T.W. Barbee, in: X-ray Microscopy, eds. G. Schmahl and D. Rudolph (Springer-Verlag, Berlin 1984) p. 163.

2 J.B. Hastings and W. Thomlinson, Nucl. Instr. and Meth. 222 (1984) 128.

3 P.L. Cowan, S. Brennan, R.D. Deslattes, A. Henins, T. Jach and E.G. Kessler, Nucl. Instr. and Meth., A246 (1986) 154.

4 E. Zeigler, Y. Lepetre, I.K. Schuller and E. Spiller, Appl. Phys. Lett. 48 (1986) 1354.

5 Z. Hussain, E. Umbach, D.A. Shirley and J. Feldhaus, Nucl. Instr. and Meth. 195 (1982) 115.

6 Z. Hussain, J.J. Barton, C.C. Bahr, E. Umbach and D.A. Shirley, Nucl. Inst. and Meth., 208 (1983) 333.

7 By SiC we mean thin layers sputter-deposited from a silicon-carbide target. These layers are amorphous and have composition close to stoichiometric SiC. For more information on these films see J.B. Kortright and D.L. Windt, to be publ. in Appl. Optics.

8 R. DiGennaro, W.R. Edwards and E. Hoyer, in: SPIE Proc. 582, eds. R. Tatchyn and I. Lindau, (1985) p. 273.

9 The WYKO optical profilometer was used with a 2.5X objective. Mention of the name WYKO in no way constitutes an endorsement for this product.

Figure Captions

Figure 1. The NSLS X24-A beamline optical components downstream of the pre-mirror pivot about the downward deflecting pre-mirror on a rigid sled. This soft x-ray beamline has a practical energy range from 800 to 5500 eV.

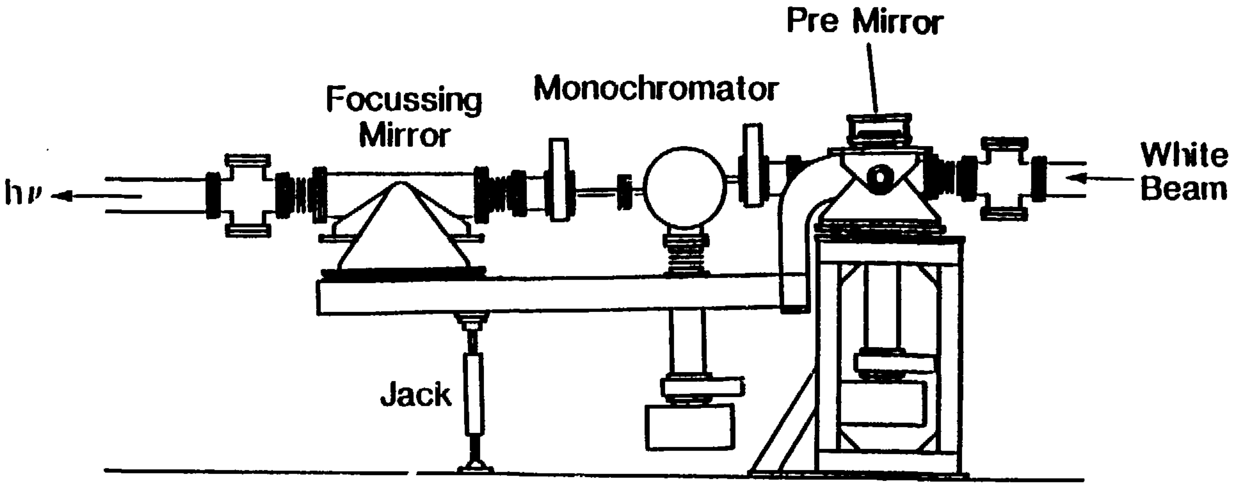
Figure 2. The measured and calculated reflectivities at 1487 eV (8.34 Å) of the SiC/V multilayer deposited onto the pre-mirror substrate plotted versus angle of incidence. The measured sample was a small witness sample coated during the same run as the pre-mirror, and the calculation is for an ideal structure.

Figure 3. Comparison of the calculated reflectivities of the SiC/V multilayer and of the Ni-coated mirror which it was designed to replace plotted as functions of energy. The area under the Ni reflectivity curve and above the multilayer curve represents the additional power filtered out by the multilayer.

Figure 4. The pre-mirror substrate shown here is made of OFHC copper, coated with electroless nickel and polished before applying the multilayer coating. The mirror was mounted with bolts through holes in the two legs.

Figure 5. Measured pre-mirror reflectivity as a function of energy is plotted on an arbitrary scale compared to the calculated reflectivity profile of the SiC/V multilayer second order Bragg peak. The similarity of width measurement and calculation is an indication of the uniformity of the multilayer coating

over the area of the mirror.



XBL 877-3248

Figure 1

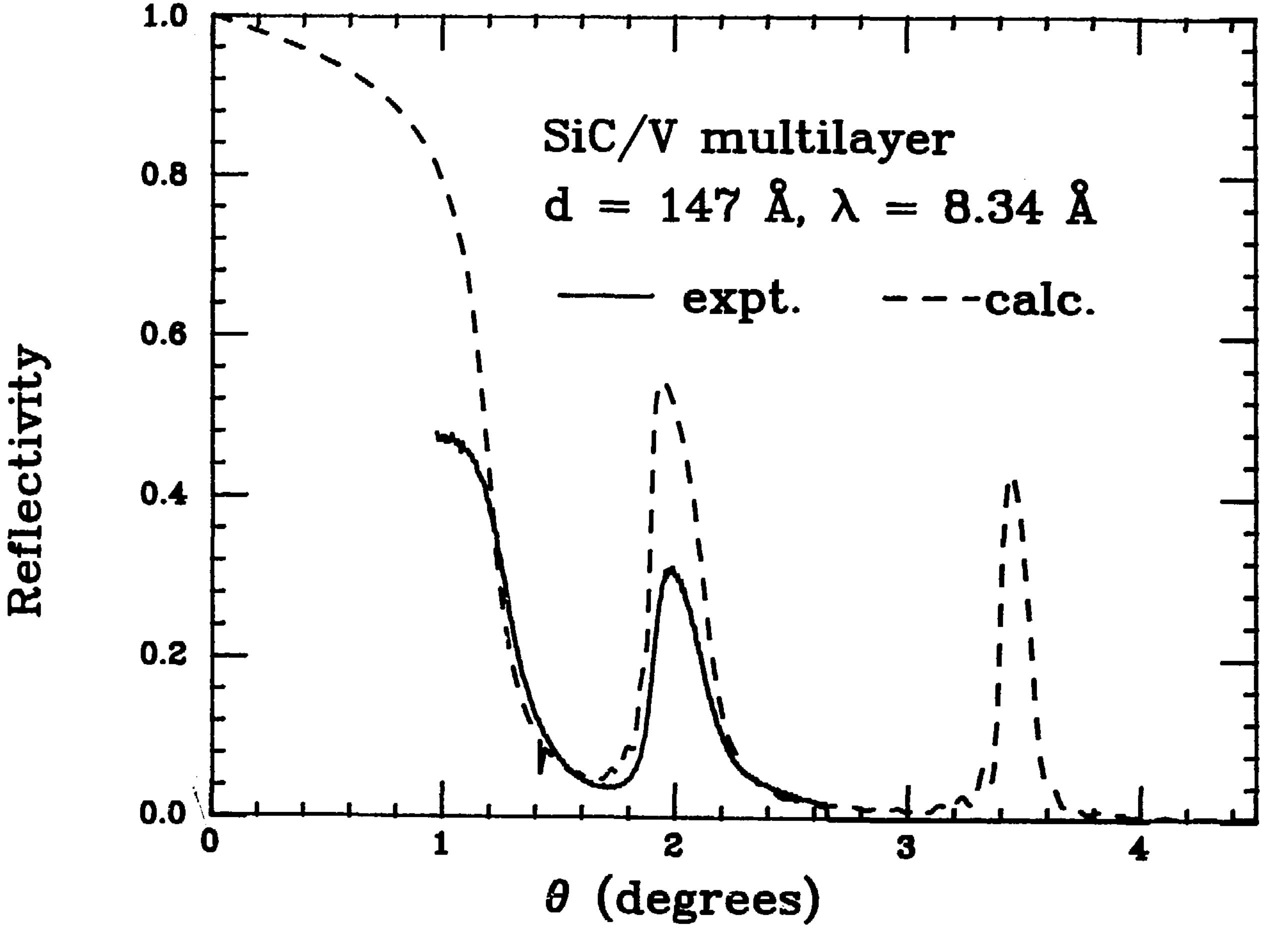

SiC/V multilayer
d = 147 Å, λ = 8.34 Å
—— expt.
- - -calc.
Reflectivity
1.0
0.8
0.6
0.4
0.2
0.0
0
1
2
3
4
θ (degrees)

Figure 2

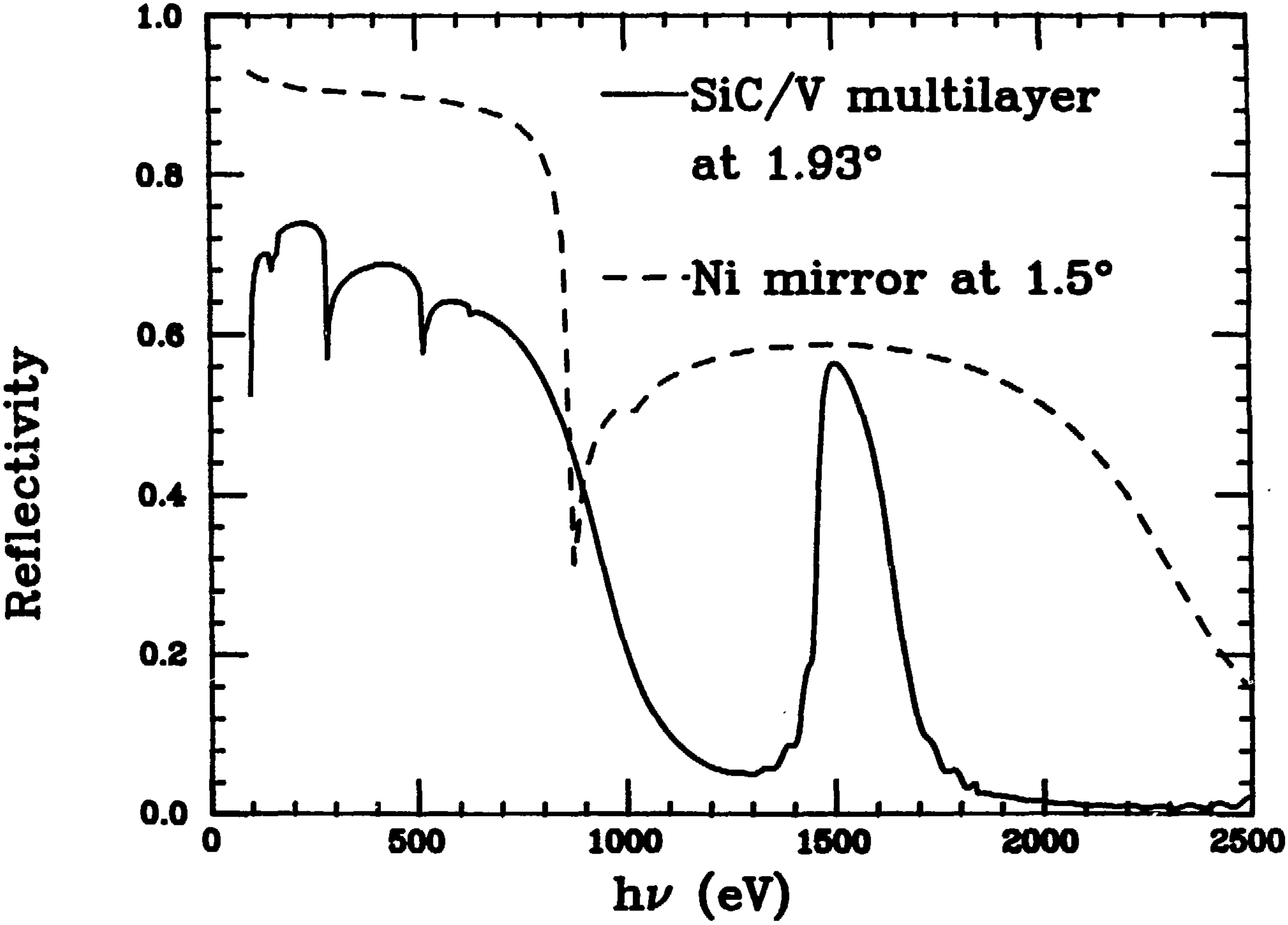

Reflectivity
1.0
0.8
0.6
0.4
0.2
0.0
0
500
1000
1500
2000
2500
hν (eV)
SiC/V multilayer at 1.93°
Ni mirror at 1.5°

Figure 3

CBB 871-623

Figure 4

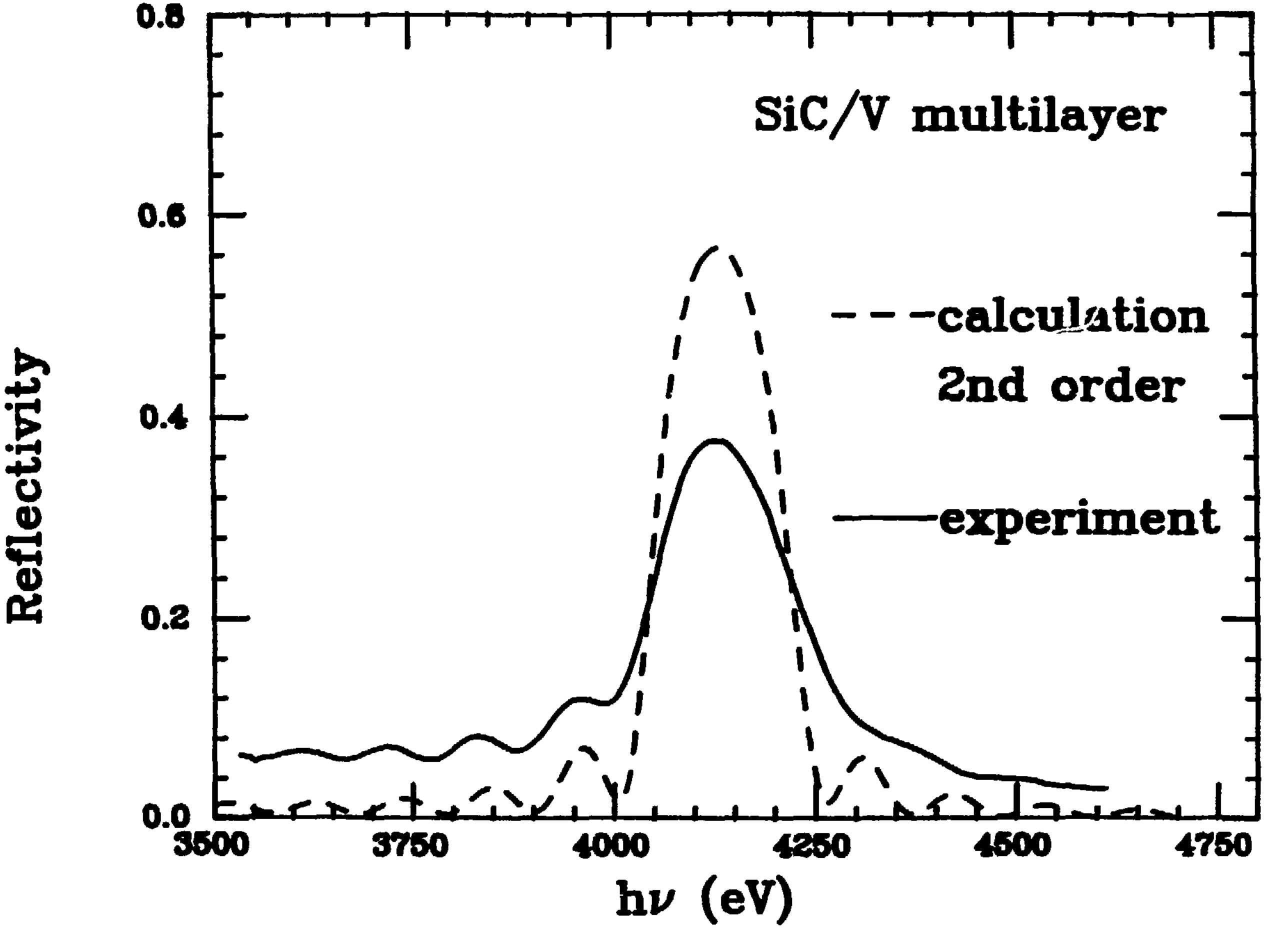

SiC/V multilayer
calculation
2nd order
experiment
Reflectivity
0.0
0.2
0.4
0.6
0.8
3500
3750
4000
4250
4500
4750
hν (eV)

Figure 5