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X-RAY REFLECTIVITY VS MIRROR SURFACE QUALITY

J. P. Stoering
M. Rice
G. A. Burginyon
R. Crabb
E. L. Augusta
C. L. Wang

January 20, 1979

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ABSTRACT

In an attempt to correlate x-ray reflectivity with carbon mirror surface quality, we compared the relative reflectivity of five mirrors of different smoothness and optical flatness. Measured and theoretical reflectivity values are in agreement for mirror smoothness $> 30 \text{ \AA}$ and optical flatness $< 1000 \text{ \AA}$. We could not establish any other correlation.

INTRODUCTION

To determine the mirror polishing specifications required to approach theoretical x-ray reflectivity values, we investigated the x-ray reflectivity of five flat, vitreous carbon mirrors of varying quality. We used a Henke x-ray tube to produce vanadium continuum x rays in the 0 to 8.5-keV range. To obtain mirror reflectivities as a function of energy with the mirror held at a particular angle, we divided the reflected by the incident bremsstrahlung continuum spectrum. These measurements give the desired cut-off energies and reflectivities directly as functions of energy, without requiring theoretical conversion from curves obtained by varying the mirror angle while looking at monoenergetic x rays. We compared the resulting reflectivity curves with theoretical curves for a perfectly flat and smooth mirror surface, and then attempted to correlate reflectivity and mirror quality. We obtained curves at three grazing mirror angles corresponding to three critical cutoff energies.

The three best mirrors (optical flatness $< 1000 \text{ \AA}$ and smoothness $> 30 \text{ \AA}$) reflected at theoretical values. Beyond that, as shown in Figs. 1 and 2, we could not establish a correlation between x-ray reflectivity and mirror surface properties.

MIRROR PROPERTIES

Vitreous Carbon Material

Vitreous carbon is an interesting substance. It was synthesized in 1967 from pyrolysis of an organic polymer (a phenolic resin) which produces random glass-like bonding between the carbon atoms.

The following qualities of vitreous carbon,¹ make it especially suited for x-ray reflectivity measurements: *high purity* (typically < 200 ppm total ash content), *a high degree of surface chemical inertness*, including low porosity and low gas permeability as well as a high specific heat

Fig. 1. Relative reflectivity vs flatness. The lines connect equal mirror area exposures as explained in text.

Fig. 2. Relative reflectivity vs average smoothness.

and melting point; *good machinability* with the ability to be ground and polished like optical glass with little pitting; *low x-ray absorption cross section* which sharpens the reflection cut-off and a K absorption edge below the energies commonly reflected.

Mirror Quality Criteria

To correlate vitreous carbon mirror quality with x-ray reflectivity, one must have criteria for "quality" mirrors in just the same way that 100% is the best reflectivity. There are essentially two criteria: flatness and smoothness. Flatness is a measure of the overall surface planarity, tilt, waviness, etc. It is affected by mirror fabrication temperature stability, subsurface damage, substrate bonding, and other factors. The central square inch of the mirror is the most important. Smoothness indicates the micro-finish scattering ability and peak-to-valley surface variations due to such effects as scratches, digs, pits, chemical corrosion, etc.

Optical Data Characterizing the Mirrors

For flatness characterization we used a Zygo Fizeau interferometer in the Tyman mode (single reflection) with a fringe width of 6328 Å (distance between adjacent maxima) and micro-interferometers at 9/16 magnification, 6 × (Zeiss Macro-Lens), 38 ×, 1000 × (Leitz Micro-Lens) with 2743-Å (10.8-p) fringe spacing. For smoothness characterization we used Metrology's Talystep surface tracing facility capable of measuring 5-to-10-Å variations. We observed qualitative features from photographs using a Reichert microscope with a Differential Interference Contrast (D.I.C.) Nomarski attachment at 177 × magnification. The results of these measurements and other general information about the mirrors are presented in Table 1.

We used five vitreous carbon mirrors labeled VC-0, VC-1, VC-2, VC-3, and VC-4. With the exception of VC-4, these mirrors were supplied to us by Polycarbon, Inc. VC-4 was manufactured in France and is believed to be higher in porosity.

X-RAY REFLECTIVITIES OF MIRRORS

Experimental Setup and Procedures

The experimental layout shown in Fig. 3 consists of a Henke-type x-ray tube², goniometer table, and a windowless Si(Li) detector. Thin Formvar films about 70 µgm/cm² vacuum isolate the x-ray tube and detector sections from the goniometer/mirror chamber. This permits operation of the Henke tube and solid-state detector at high vacuum (10^{-6} and 2×10^{-7} Torr, respectively), while the mirror chamber is maintained at about 5×10^{-4} Torr via liquid nitrogen sorption pumps.

The Henke tube generates x rays by electrostatically focusing electrons from a tungsten filament located behind the anode to a small area on the front surface of the anode. This geometry protects the anode target area from tungsten contamination by the filament. Interchangeable

Table 1. General features and Surface Characterization of Five Vitreous Carbon Mirrors.

Mirror	Material	Bonding	Substrate	Visual surface assessment	Area, in.	Optical Flatness, Å		Average smoothness, rms (Å)
						Parallel direction	Perpendicular direction	
VC-0	PC-Unknown 3 mm thick	Unknown	Fused silica	Aged or wrinkled appearance, small pits, several scratches or tracks	0.5 X 0.5	944	1406	1175
					0.75 X 0.5	1644	1406	1525
					1 X 0.5	2637	2182	2409
VC-1	PC-100 [†] 3 mm thick	Epoxy	Kovar	Fairly uniform polish with low scatter, 1 scratch	0.5 X 0.5	253	443	348
					0.75 X 0.5	570	570	570
					1 X 0.5	822	1376	1099
VC-2	PC-100 3 mm thick	HE-79 polyester	Fused silica	Fairly uniform polish with low scatter, 2 scratches	0.5 X 0.5	379	506	443
					0.75 X 0.5	506	506	506
					1 X 0.5	944	703	824
VC-3	PC-100 3 mm thick	Polyester cement	Fused silica	Very uniform polish with minimal scatter	0.5 X 0.5	190	190	190
					0.75 X 0.5	316	379	348
					1 X 0.5	443	506	475
VC-4	French manufacture	Unknown	Unknown	Rough, milky or stained areas, large pits, several scratches	0.5 X 0.5	2182	6328	4255
					0.75 X 0.5	3164	6328	4746
					1 X 0.5	4983	12656	8820

Fig. 3. Experimental set-up for low-energy x-ray reflectivity measurements of vitreous carbon mirrors.

anodes permit selection of a variety of targets. For mirror reflectivity comparisons we used a vanadium anode at a current of 0.58 mA and rms voltage at 7.20 kV (peak at 12.0 kV). We also used an iron anode at 7.39 kV rms and 0.71 mA to measure the reflectivity of one mirror (VC-2).

A tantalum slit near the Henke tube exit helps to eliminate fluorescent radiation from materials in the tube mounting structure. The x-ray collimation in these measurements restricted the illuminated area of the mirror to a central portion ranging from 1×0.5 in. to 0.5×0.5 in., depending on the angle setting. Each mirror was mounted on a goniometer table and data recorded at $\theta = 0, 0.443, 0.643$, and 0.972 deg, corresponding to critical energy cutoffs of no cutoff, 3.25, 2.25, and 1.50 keV, respectively. Because the relationship between mirror quality and reflectivity might depend on mirror angle, we studied several different mirror angles, and therefore energy cutoffs.

Initially, the mirrors were optically aligned with the chamber open and then final alignment corrections were made using x rays under vacuum. We estimate the overall absolute alignment uncertainty to be within ± 0.02 deg, which has a negligible effect on reflectivity values.

Before insertion into the vacuum tank, we cleaned the vitreous carbon mirrors by placing lens paper on each mirror, pouring on equal amounts of toluene and acetone, and then slowly dragging the lens paper off, ensuring a lint-, dust-, and grease-free surface.

The x-ray-beam-deposited energy density is much too low to cause mirror distortion.

A Kevex-Ray Micro-X windowless silicon solid-state detector was aligned for maximum received reflected signal. The characteristics of the diode are reported by Musker.³ It has an energy resolution of about 150 eV at 5.9 keV. We kept count rates below 750 counts/s to ensure against pileup.

Reflectivity Curves

Figure 4 shows a representative reflectivity curve for the VC-2 mirror obtained by dividing the reflected spectrum (curve B in Fig. 5) by the direct iron anode spectrum (curve A in Fig. 5). An open beam run was obtained before and after each reflection run to minimize beam instability effects.

The raw data spectra shown are predominantly bremsstrahlung continuum from the iron anode with the characteristic iron lines superimposed. Contaminant K α radiation from carbon, silicon, and, to a lesser extent, vanadium and aluminum, are also evident. Altogether, we took 28 such spectra.

Figure 4 compares measured and theoretical reflectivity curves. The theoretical curve is based on Honl's theory for the refractive index as presented in Compton and Allison.⁴ It assumes a perfectly flat and smooth carbon mirror surface with a material density of 1.54 gm/cm^3 as suggested by the manufacturer. We have used the actual measured angle in computing the theoretical curve

Fig. 4. Measured and theoretical reflectivity vs x-ray energy for $\theta=0.443$ deg. Theoretical curve which has detector resolution folded in, is shown by solid line. Measured curve is the result of dividing the reflected spectrum (curve B, Fig. 5) by the unreflected spectrum (curve A, Fig. 5).

Fig. 5. Iron anode x-ray spectrum: curve A is the direct unreflected spectrum ($\theta=0$ deg), curve B is the reflected spectrum from VC-2 mirror with the mirror angle set at 0.443 deg.

shown. The two curves do not agree perfectly. The measured reflectivity is 3 to 5% lower than calculated. However, the differences are comparable to experimental uncertainties and therefore are probably not significant. Errors from counting statistics alone amount to $\pm 4\%$. We believe that we are measuring reflectivities within $\pm 5\%$ of true values.

Comparative Mirror Performance Based on X-Ray Reflectivity

We used three somewhat equivalent criteria to make our comparison: (1) With the mirrors set at one of the three discrete reflection cutoff angles, each mirror's reflectivity at a particular energy value is plotted against the reflectivity of VC-3 at the same energy value. VC-3 was chosen as the standard because it has the highest reflectivity of any of the mirrors. The slope of the plot obtained by a linear fit leans toward the axis of the better reflecting mirror and is simply an average of the reflectivity ratios at each energy point. We obtained the relative reflectivity values shown in Table 2 by averaging these slopes for the three cutoff angles. By presenting ratios of mirror reflectivities rather than absolute values, we condense the reflectivity information for several cut-offs into a single number, which tends to cancel spurious instrumental effects in the data; (2) This criterion is simply the ratio of the total area under each reflectivity curve to that of the VC-3 mirror; (3) To eliminate an observed instrumental effect below 1 keV caused, in part, by detector icing, we integrated above 1 keV and then averaged the area ratios for the three cutoff angles for each mirror.

Table 2. Comparative Mirror Performance based on Reflectivity Measurements.

Mirror	Average of point-by-point reflectivity ratios ^a , %	Ratio of total areas ^b , %	Area ratios taken above 1 keV ^c , %
VC-0	72.1 ^d	71.6 ^d	70.7 ^d
VC-1	94.1	95.5	97.3
VC-2	93.8	96.0	98.3
VC-3	100.0	100.0	100.0
VC-4	88.4	91.4	91.0

^aSlope comparison, method 1 in text.

^bMethod 2 in text.

^cMethod 3 in text.

^dThese are average reflectivity ratios for the three different cut-off energies.

The results shown in Table 2 indicate that the method used for comparison does not make much difference; the ordering is clear by any of the methods. The VC-3 mirror is the best; next, VC-2 and VC-1 are equal in reflectivity performance; VC-4 is third best; and VC-0 is a distant last. It appears that just by comparing the ratios of the area under the reflectivity curves, the relative performances are accurately judged.

RESULTS

Figures 1 and 2 show the results of these studies. The data in Fig. 2 suggest that reflectivities within 5% of theoretical values can be expected for a vitreous carbon mirror having an average smoothness less than about 30 Å, with an added implication in Fig. 1 that optical flatness does not greatly exceed 1000 Å. We did not note any other general correlation between x-ray reflectivity and mirror surface quality. The three flatness values plotted for each mirror in Fig. 1 correspond to separate flatness measurements for each of the three illuminated mirror areas defined by the mirror angular setting at a particular cutoff energy. To eliminate confusion, the 1- X 0.5-in. mirror areas are connected by a dashed line, and a solid line ties the 0.75- X 0.5-in. data points. The 0.5- X 0.5-in. areas are left unconnected. Each point is the average of the surface flatness measured in the direction parallel and perpendicular to the incident x-ray beam direction as indicated in Table 1. A single point was sufficient to plot the average (rms) smoothness (Fig. 2), as the Talystep records show consistent smoothness over each exposed area for all five mirrors.

Because the optical flatness of the central 0.5- X 0.5-in. area of VC-0 is nearly identical to the flatness of the 1- X 0.5-in. area of VC-1, one may be tempted to attribute the low reflectance of VC-0 to lack of smoothness and thus relax the flatness criteria to achieve 95% reflectivity. The plausibility of this argument is questionable, however, as the reflectivity of VC-4 is high in spite of its poor smoothness and flatness characteristics.

We do not have sufficient data to show that no correlation between x-ray reflectivity and surface quality exists. Of the five data points in Figs. 1 and 2, three are bunched in a small region of smoothness/flatness space leaving, in effect, only three widely separated points to establish any correlation. Furthermore, one of these points is the VC-4 mirror which may not be as reliable as the other points. The material for the VC-4 mirror was acquired from a different source than the other mirrors. Its surface texture, clearly distinguishable from the other mirrors, has a milky and porous appearance. This may indicate that other bulk material properties are of equal or greater importance than smoothness or flatness properties in reflecting x rays. Also, as shown in Table 1, the spread in the two 90-deg flatness measurements is much greater for VC-4 than any of the other mirrors. More data, especially in the 30- to 100-Å smoothness range, would be helpful. Our

measurements indicate that the ratio ϵ/ϵ_0 measured to theoretical reflectivity is insensitive to the particular energy cutoff, at least between 1.5 and 3.25 keV. Therefore, new points can easily be added in or near this energy region. We intend to accumulate additional data as new mirrors become available. We wish to invite anyone who has such data and who is willing to have his mirrors characterized to contact us.

ACKNOWLEDGMENTS

We gratefully acknowledge the cooperation of LLL's Metrology and Optics Lab for mirror polishing and characterization. We are indebted to James Willis for the optical flatness measurements, Ron Blackman for mirror polishing, Phil Baker and Eberhard Prochnow for technical advice, and to Jim Bryan and Herman Hauschildt of Metrology Lab for advice and Talystep smoothness measurements.

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