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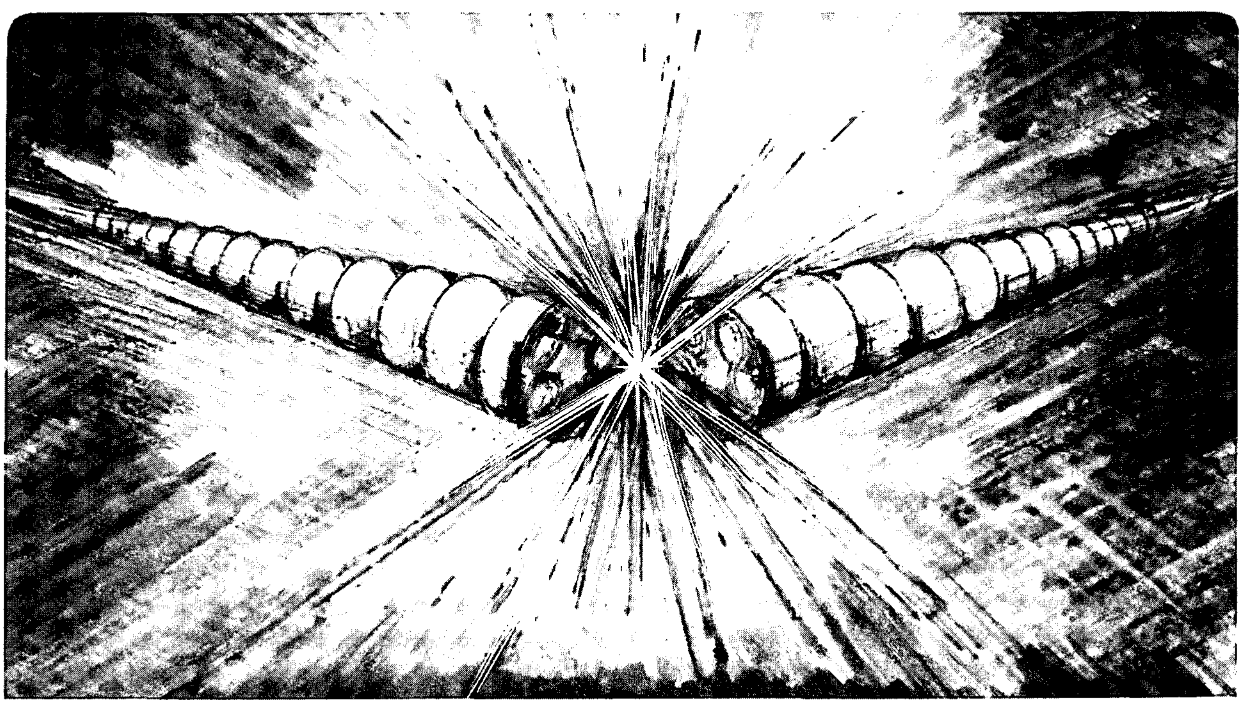

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DISCLAIMER

Stability of Tungsten/Carbon and Tungsten/Silicon Multilayer X-Ray Mirrors Under Thermal Annealing and X-Radiation Exposure

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ABSTRACT

The effect of thermal annealing and irradiation in an intense white synchrotron x-ray beam on the x-ray reflectance of tungsten/carbon and tungsten/silicon multilayers is reported. Thermal annealing at 400°C for 2 hours produces larger effects than irradiation of cooled multilayers in the white beam of a 20-pole hard x-ray wiggler with 0.94 Tesla peak field on the storage ring DORIS operating at 5.42 GeV and electron currents of 20-36 mA for 40 hours. Thermal annealing caused the period and first order reflectance of a W/Si sample to decrease, in contrast to a W/C sample whose period and reflectance increased on annealing. Of five actively cooled samples irradiated, one W/C sample showed significant change in reflectance. Pre-annealing of this multilayer stabilized it to radiation-induced changes. Irradiation effects also depend on multilayer period and constituent materials. Implications of these results for models describing multilayer reflectance and for multilayer applications in the new generation of synchrotron radiation sources are discussed.

Introduction

Multilayer x-ray interference coatings have been proposed for applications such as power filters which would position them as the first optical element in synchrotron radiation beam lines of unprecedented power density at the high-brightness synchrotron radiation facilities currently under construction.[1-3] Since multilayers are inherently metastable materials composites with nanometer-scale periodicities, questions arise regarding their microstructural stability and hence the stability of their reflectance in these intense x-ray beams. While the stability of multilayer-coated optics has not limited their application in white beams from existing synchrotron radiation bending magnet sources,[4-6] the unprecedented radiation densities from insertion devices at facilities currently under construction require investigation. Issues of multilayer stability in synchrotron x-ray beams are to some extent distinct from those of thermal deformation of mirrors or monochromator gratings and crystals, where primary concerns are degraded optical performance of specular reflection or Bragg diffraction due to differential thermal expansion resulting from thermal gradients.[7-9] While thermal distortions are also relevant to multilayer-coated x-ray optics, here we are concerned with the stability of the multilayer coating itself. Since synchrotron beams are pulsed at MHz rates we are interested in time-averaged effects. Reflectance changes in multilayers have also been observed to result from single pulses of intense soft x-rays from dense plasmas[10] and of 1.06 μm laser irradiation.[11,12]

Investigation of possible radiation-induced damage to multilayer reflectance must be made with consideration of thermal annealing effects on these metastable structures, since multilayers will experience at least moderate thermal annealing in high-flux applications. Previous studies of the effects of thermal annealing on W/C and W/Si multilayer structure and reflectance[12-20] help in placing limits on annealing conditions below which multilayers retain their layered microstructure and indicate the nature of structural rearrangements in this regime. In principle we can distinguish between multilayer structural rearrangements resulting from purely thermal effects and from non-thermal, radiation-induced effects. Non-thermal structural changes could manifest themselves, for example, as local interatomic structural rearrangements resulting from photoionization-induced changes in interatomic bonding, or other localized electronic defects. Such structural effects caused by x-ray absorption have not been reported in multilayers, to our knowledge, though have been observed in thin semiconducting and insulating membranes.[21-23] The metallic character of most multilayer x-ray mirrors may provide resistance to some types of radiation-induced damage mechanisms. In addition to possible non-thermal, radiation-induced changes, it is known that multilayer-coated mirrors placed in high-flux synchrotron x-ray beams can reach temperatures in excess of 500°C if not actively cooled.[3] While active substrate cooling limits surface temperature increases to tens of degrees, multilayer-coatings may experience moderate thermal annealing for prolonged periods while concurrently experiencing photoexcitation and relaxation by absorbing a fraction of the incident x-ray flux. Distinguishing between purely thermal effects, purely radiation-induced effects, and radiation-induced thermal annealing may in practice be difficult.

This paper presents results of early studies of the effect of intense x-ray exposure on the x-ray reflectance of cooled, sputtered W/C and W/Si multilayers, and compares these effects with those of thermal annealing. The intense white beam from the HARWI wiggler source at HASYLAB[24] was used for irradiation. We investigate the possibility[25] that annealing of multilayers might stabilize their microstructures to further changes which would otherwise occur in high-flux x-ray beams. After describing experimental methods, we present and discuss results of annealing and irradiation on the x-ray reflectance of a set of multilayer samples.

Experiment

The W/C and W/Si multilayer systems were chosen for initial study because both combinations of materials are widely produced and utilized in x-ray applications, especially in the hard x-ray regime. Samples were deposited by magnetron sputtering at the Lawrence Berkeley Laboratory and are summarized in Table 1, which gives the nominal period d and the number of periods comprising each sample. Tungsten and carbon were sputtered with dc input power while silicon was sputtered with rf power, all in an argon ambient of roughly 2 mTorr onto substrates rotating alternatively across the different targets at a distance of 10 cm. The number of periods ranged from 100 to 200 to yield a total thickness of roughly 0.4 μm, and the W-rich layers comprised roughly 0.4 of the total period based on the individual sputtering rates of the elements. These parameters are typical of those of multilayers used in hard x-ray applications. During each multilayer deposition several superpolished Si substrates (10 x 60 x 6 mm thick) were coated with identical multilayers for comparison of the effects of irradiation and thermal annealing on performance. These substrates were flat to 1/10 the wavelength of visible light and had an rms roughness of less than 2 Å as determined by a WYKO optical profilometer measuring a range of spatial frequencies from 0.8 μm to

0.6 mm.[26] Before exposure to the wiggler radiation, one W/C sample (89-150) and one W/Si sample (89-153) were annealed at 400°C for 2 hours in high vacuum.

Exposures utilized the white beam radiated from the 10 period, 0.94 Tesla wiggler on the electron synchrotron DORIS operating at 5.42 GeV in the HASYLAB facility.[24] This source radiates a beam with high power density in the hard x-ray region. The only filtering elements between the source and samples were 0.4 mm of carbon upstream of 3 mm of Be, significantly reducing flux in the spectral range below 2 keV. An optics thermal test facility[27] was used to monitor the temperature of the cooled optics during exposure with an infrared camera viewing the sample surfaces and a thermocouple attached to one sample. Six samples were installed adjacent to each other in the rough vacuum chamber with a liquid GaInSn alloy as a thermal conductor between the substrates and a water-cooled Cu thermal sink. Because the power per unit area absorbed in the relatively thin multilayer coating is maximized for incidence angles away from grazing, an incidence angle of 30 degrees was chosen. The average power density during irradiation was roughly 0.25 W/mm^2 at the sample. Roughly seven percent of photons with energy equal to the maximum critical energy of x-rays radiated from the wiggler (17.6 keV) are absorbed in the multilayer. Of these six samples installed, five were fully illuminated with the x-ray beam, and only results pertaining to these five samples are reported here. The time for exposure was limited and totaled roughly 40 hours spanning several fills of the storage ring.

The effect of x-ray exposure and annealing on multilayer performance was evaluated by measuring their specular reflectance, from which the precise multilayer period can be obtained as well as values for peak and integrated reflectance of the various Bragg orders. Reflectance measurements were made over six orders of magnitude using Cu $K\alpha_1$ x-rays monochromatized with a Ge (111) crystal from a sealed tube and a carefully aligned two-axis diffractometer. Accurate knowledge of the multilayer period is required since small changes in period are known to result from thermal annealing. The period d of each sample was determined by measuring the refraction-shifted positions θ_m of many orders of multilayer Bragg reflections in a symmetric θ-2θ geometry with a narrow slit defining angular divergence of the reflected beam. The apparent period d_m of the mth order is obtained from $m\lambda=2d_m\sin(\theta_m)$, and is related to the actual period d by a first order refraction correction[28] $d_m = d(1 - \delta\csc^2\theta_m)$ where $1-\delta$ is the effective real part of the complex index of refraction for the multilayer structure. Linear least-squares analysis of d_m vs. $\csc^2\theta_m$ for all measured orders provides a value of d together with the standard deviation, σ, for d. This is illustrated for the annealed W/C sample 89-150 in Figure 1, which shows both the measured reflectance data and fit from which d is determined. Other important measures of multilayer performance are peak and integrated reflectance values for the first order Bragg peak. These were obtained from rocking curves through the first order Bragg peak measured with the slit defining the reflected beam divergence wide open. Integrated reflectance values were obtained by integrating over a range of 5 times the peak FWHM centered at the peak.

Results and Discussion

A. Thermal annealing at 400°C

Vacuum annealing the W/C sample (89-150) and the W/Si sample (89-153) at 400°C for 2 hours resulted in significant changes in multilayer structure and reflectance. Certain aspects of the response to thermal annealing of the W/C system have been observed in many studies,[13-20] though the annealing response of the W/Si system has been less-well documented.[12,20] Annealing results are summarized in Tables 2 and 3 and Figure 2. Table 2 shows the multilayer period, d, the number of observed Bragg orders, m_{obs}, and the standard deviation of the determination of d, σ, for the as-prepared and annealed samples. Table 3 shows the peak reflectance, integrated reflectance, and FWHM of the first order Bragg peaks of these samples. Figure 2 shows the change in first order Bragg reflectance on annealing as measured from rocking curves.

Annealing of the W/C sample results in expansion of the multilayer period, an increase in peak reflectance and FWHM of the first order Bragg peak, and an increase in the number of observed Bragg orders. The annealing-induced expansion in period of W/C multilayers has been commonly observed,[12-15,17,20] though its explanation remains somewhat unclear. Early work suggested that agglomeration of the W-rich layers on annealing could explain this expansion,[16] though it has since become evident that expansion occurs on annealing W/C multilayers without agglomeration. The increase in reflectance on annealing in Table 3 is not consistent with agglomeration of the W-rich layers, which would result in degraded reflectance. Comparison of the annealing response of W/C and W/Si multilayers in this work (see below) is consistent with the suggestion[20,29] that expansion of the C-rich layers on annealing could account for the observed expansion in period. Other studies of the W/C system reveal structural changes specifically in the W-rich layers on annealing, including diffusional intermixing of C into the W-rich layers both in the amorphous state[17] and resulting in formation of polycrystalline W_2C.[19,14,29] The structural nature of both the as-prepared and annealed W-rich layers depends sensitively on their period,[17,18] their thermal history,[14,17-19] the relative thickness of the different layers,[14] and possibly on their preparation technique. Thus many factors must be considered in interpreting changes on annealing which would affect structure and reflectance. We believe that the observed increase in first order reflectance of sample 89-150 is real because it was observed for several different W/C samples having 3 nm period annealed under these conditions. Similar results have been reported previously for 4 nm sputtered W/C multilayers,[13] where an increase in reflectance was suggested to result from increased lateral uniformity in the layered structures and/or from sharper concentration gradients across the interfaces. Several additional factors should be considered in explaining the increased reflectance on annealing: expansion of the C-rich layers would lessen the optical density of those layers, diffusion of C into the W-rich layers would change the optical density of those layers, and the resultant change in relative thickness of the C- and W-rich layers would affect the intensities of the various orders. The later factor especially could also explain why the first order peak reflectance of some W/C multilayers is observed to increase on annealing, while for others is observed to decrease: the direction of change in reflectance could depend on the initial relative thicknesses of the C- and W-rich layers. Quantitative analysis of the annealing-induced changes in reflectance is beyond the scope of the this work. Detailed modelling of the entire measured specular reflectance profile, combined with knowledge obtained from microstructural characterizations as self-consistency checks, would help in understanding the origin of the changes observed on annealing.

Annealing the W/Si sample (89-153) results in changes in the opposite direction from those in the W/C system. As seen in Tables 2 and 3 and Fig. 2b, annealing causes the W/Si multilayer period as well as the first order peak and integrated reflectance to decrease. These features have been observed in previous annealing studies of W/Si multilayers.[12,20] Annealing causes an especially large decrease in the first order FWHM of this sample. Qualitatively the decrease in reflectance on annealing is more easily explained than the observed increase for the W/C sample, since diffusional intermixing across the interfaces or decreased lateral perfection of the layered structures are both likely consequences of annealing and would both be expected to result in decreased reflectance. The general behavior of the W/Si sample on annealing provides insight into the applicability of models for describing multilayer reflectance and its change on annealing. In particular, the annealing-induced decrease in both the first order peak reflectance and FWHM is not consistent with a Debye-Waller type model[30-32] in which the reflectance amplitude is reduced by the factor $\exp[-2(2\pi\sigma\sin(\theta)/\lambda)^2]$, where λ is the wavelength, θ is the reflection angle (from grazing), and σ describes an assumed rms interface roughness. Applying this model to describe reflectance changes on annealing by assuming that annealing increases σ predicts that the peak reflectance decreases, as observed, but does not predict the significant narrowing of the first order peak that is observed. For the W/C sample annealed here the first order FWHM increased, while other studies[3] have ovserved a decrease in FWHM for W/C on annealing. Thus models other than the Debye-Waller model should be investigated to relate multilayer structure to x-ray reflectance, especially in studying changes in these properties on annealing or irradiation.

B. Irradiation under the white wiggler beam

The effects resulting from irradiating the multilayers in rough vacuum in the white wiggler beam for roughly 40 hours are smaller than those resulting from vacuum annealing at 400°C for 2 hours. During irradiation maximum surface temperatures ranged from 55°C at 20 mA beam current to 74°C at 36 mA. The average power absorbed in the multilayers and their substrates was roughly 70 watts during this period. Only a small fraction of this power is absorbed within the multilayer coatings themselves, the remainder being absorbed in the Si substrates.

All exposed samples exhibited a contamination layer visible to the eye as a translucent brown film spanning the exposed area and observable in the x-ray specular reflectance as shown in Figure 3a. The frequency of the contamination oscillation can be accounted for by a carbonaceous film several tens of nanometers thick, although a calculation assuming a single homogeneous layer fails to adequately reproduce the envelope of the measured oscillation. These calculations indicate that the presence of such a contamination layer does not affect the positions of the various multilayer Bragg peaks significantly enough to affect the determination of the multilayer period within $\pm \sigma$. The formation of carbon deposits on synchrotron optics in poor vacuum is commonly observed, and is thought to result from cracking of hydrocarbons at the surface by photoelectrons from the optic.[33] While techniques have been developed to remove carbon contamination from synchrotron optics *in situ*,[34,35] care must be taken in cleaning contaminated multilayers by these techniques to avoid removing the thin, optically active multilayer coating in addition to the contamination.

The effects of irradiation on multilayer period and first peak reflectance are shown in Tables 4 and 5 respectively, and will be discussed separately. The effects are generally small, so that care must be taken both in measuring these quantities and in interpreting the significance of possible radiation-induced changes. Consider first the effect of irradiation on the multilayer period shown in Table 4. Of the five samples irradiated, one shows a change in period Δd which is significantly greater than the combined standard deviations σ of the separate d values. This is the as-prepared W/C sample 89-150 with nominal period 29.8 Å, which shows an expansion in period of 0.34 Å or 1.1% on irradiation. This change is 6 times the combined standard deviations of the individual measurements. Figure 3b shows high resolution θ-2θ scans across the first Bragg peak of these two samples, showing that this change in period results in a shift in the Bragg peak by roughly one-third of its width. The change in period in the direction of expansion is consistent with the expansion observed on annealing at 400°C. In Fig. 3b the irradiated sample appears to have a lower peak reflectivity than the unexposed sample, contrary to reflectivity data in Table 5. This is because the reflectance curves in Fig 3b were obtained with a narrow slit in front of the detector, which makes absolute reflectance measurement difficult, while the quantities in Table 5 were obtained with this slit wide open. The other four samples irradiated show changes in period of less than 0.2%, and while these changes are probably too small to be significant, we note that all changes are in the direction of smaller period. It is significant, however, that the period of the 89-150 sample which had been pre-annealed does not change on exposure. Thermal annealing of this W/C sample thus not only increased its reflectance, but also stabilized it against further changes in period on irradiation. Considering the lack of change in period of the as-prepared W/C sample 89–155 with 20 Å nominal period, we conclude that the effects of irradiation on period in the W/C system depend on the period. The lack of any significant change in period for either of the W/Si samples suggests that the effects of irradiation also are materials dependent.

Table 5 shows that changes in first order reflectance on irradiation are small and do not correlate with changes in period. In particular, the as-prepared 89–150 sample which shows a change in period does not show significant change in reflectance on irradiation. The reflectance quantities are not determined with as much precision as are the periods, and are estimated to be accurate to several percent. The data in Table 5 suggest a trend of a slight decrease on irradiation in overall reflectance for the W/C samples, and a slight increase on irradiation for the W/Si samples. The largest observed change after irradiation is a 10% increase in the peak reflectance of the annealed W/Si 89–153 sample, which is somewhat surprising considering that thermal annealing itself resulted in a 22% decrease in peak reflectance.

Summary

An important conclusion from this study is that actively cooled multilayers can undergo structural and reflectance changes on exposure to intense white beams from high intensity insertion device synchrotron radiation sources. This was observed for one W/C multilayer of five samples irradiated. Irradiation effects, like thermal annealing effects, apear to depend on multilayer period, constituent materials, and pre-annealing conditions. The effects resulting from irradiation under the conditions reported here are much smaller than those from thermal annealing at 400°C for 2 hours. We have not determined whether the changes in the irradiated W/C sample whose period expanded on annealing were the result of the moderate thermal annealing experienced, of non-thermal radiation-induced effects, or a combination of the two; clearly low temperature thermal annealing studies would be useful here. The complex response of the limited sample set studied suggests that further studies are warranted to better establish trends on irradiation. The microscopic details resulting in the response of the multilayers to both thermal annealing and irradiation are not fully understood at present. Analysis of absolute x-ray reflectance measurements from multilayers on high quality substrates, together with information regarding microstructural details from independant measurements, may aid in relating changes in structure to x-ray reflectance.

This study has implications for the utilization of multilayer-coated mirrors as the first optical element in the upcoming generation of synchrotron radiation sources where unprecedented power densities will be achieved, and for the continued research which may lead to these applications. The relative stability under intense irradiation exhibited by most of the cooled samples studied here supports the prospect of utilizing multilayers in harsh radiation environments. However, the absorbed power densities and exposure times encountered by these samples are far short of what would be encountered in an insertion device beam at the high-brightness 6-8 GeV synchrotron radiation sources currently under construction. More studies at increased irradiation levels and for longer durations are needed. The materials, period, and processing dependence of structure/reflectance relationships observed in this and other studies suggest that further research may result in multilayers with enhanced structural stability, both to intense x-radiation and to thermal annealing. The materials and irradiation conditions studied here are especially relevant to applications of multilayers in the hard x-ray region. In the soft x-ray region materials of choice for multilayer reflectors are different from those studied here, x-ray absorption lengths are shorter, and the relative probabilities of processes by which photo-excited atoms relax differ. Thus similar research specifically geared to the soft x-ray region may find different behaviors than those emerging in the hard x-ray region.

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Table 1. Summary of sputtered multilayer samples studied.

sample	materials	nominal d (Å)	no. of periods
89–155	W/C	19.7	200
89–150	W/C	29.8	150
89–153	W/Si	39.0	100

Table 2. Thermal annealing effects on multilayer period.

	as-prepared			annealed 400°C, 2hr			change	
sample	d (Å)	m_{obs}	σ (Å)	d (Å)	m_{obs}	σ (Å)	Δd (Å)	Δd/d
W/C 89–150	29.78	4	0.024	30.95	6	0.012	+0.34	+0.039
W/Si 89–153	38.96	7	0.081	38.00	7	0.050	−0.96	−0.025

Table 3. Thermal annealing effects on first order reflectance.

	as-prepared			annealed 400°C, 2hr		
sample	R_{peak}	FWHM† (mrad)	$R_{integrated}$ (mrad)	R_{peak}	FWHM† (mrad)	$R_{integrated}$ (mrad)
W/C 89–150	0.71	0.75	0.62	0.75	0.80	0.70
W/Si 89–153	0.68	1.01	0.78	0.53	0.72	0.45

†For comparison, calculations assuming ideally perfect structures corresponding to samples 89–150 and 89–153 have FWHM of 0.89 mrad and 1.1 mrad, respectively.

Table 4. Irradiation effects on multilayer period.

	unexposed		irradiated		change	
sample	d (Å)	σ (Å)	d (Å)	σ (Å)	Δd (Å)	Δd/d
W/C as-prep. 89-150	29.78	0.024	30.12	0.028	+0.34	+0.011
W/C annealed 89-150	30.95	0.012	30.93	0.026	-0.02	-0.0006
W/C as-prep. 89-155	19.66	0.002	19.65	0.007	-0.01	-0.0005
W/Si as-prep. 89-153	38.96	0.081	38.96	0.099	0.00	0.0
W/Si annealed 89-153	38.00	0.050	37.93	0.075	-0.07	-0.0018

Table 5. Irradiation effects on first order reflectance.

	unexposed			irradiated		
sample	R_{peak}	FWHM (mrad)	$R_{integrated}$ (mrad)	R_{peak}	FWHM (mrad)	$R_{integrated}$ (mrad)
W/C as-prep. 89-150	0.71	0.75	0.62	0.71	0.73	0.61
W/C annealed 89-150	0.75	0.80	0.70	0.74	0.79	0.68
W/C as-prep. 89-155	0.53	0.42	0.40	0.50	0.43	0.38
W/Si as-prep. 89-153	0.68	1.01	0.78	0.70	1.01	0.79
W/Si annealed 89-153	0.53	0.72	0.45	0.58	0.72	0.48

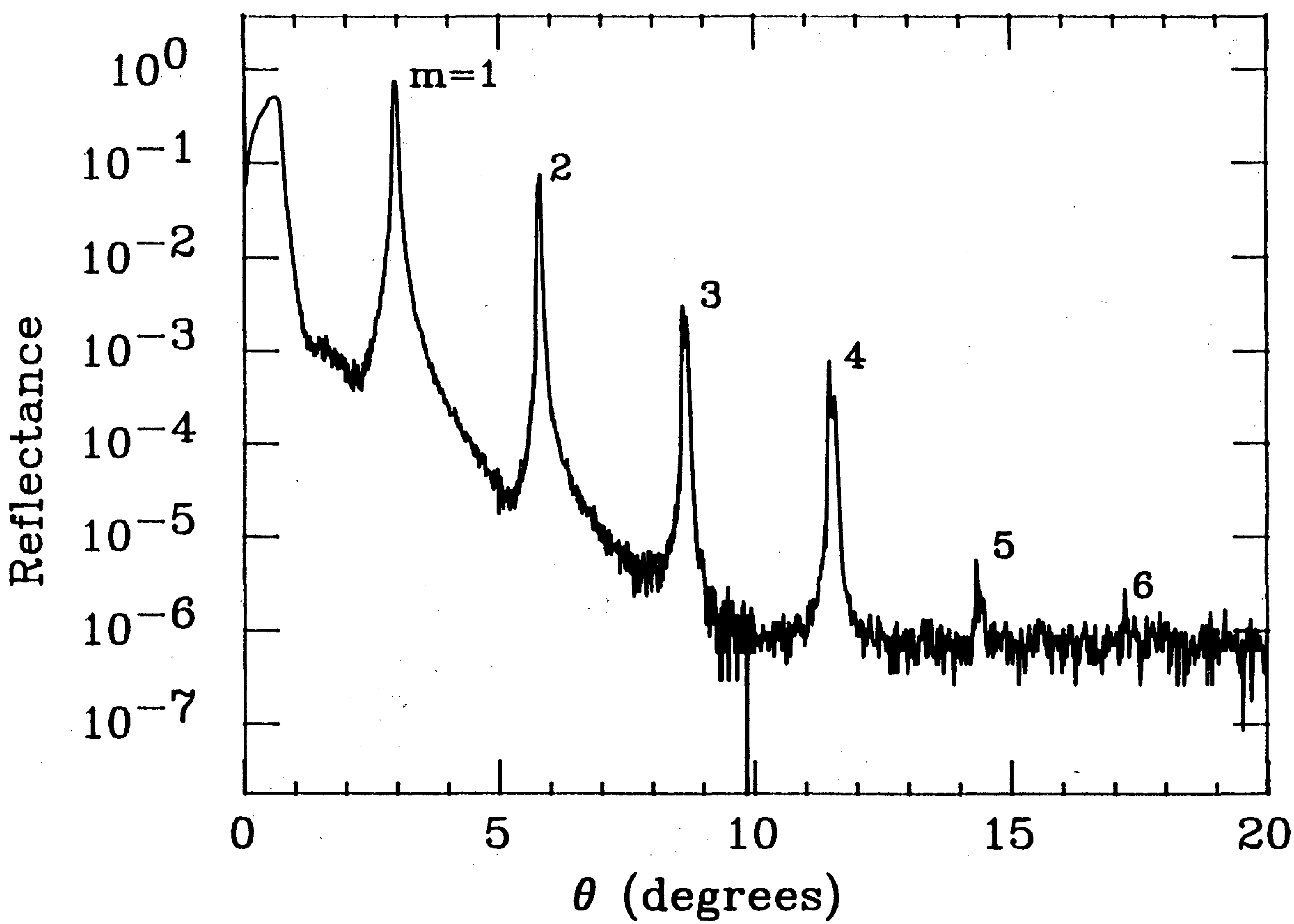
Figures Captions

Figure 1. High resolution specular reflectance profile of the annealed W/C sample 89-150 is in (a). (b) shows the linear least-squares fit of apparent period d_m vs. $\csc^2(\theta_m)$ which yields the refraction-corrected period of 30.95 Å with standard deviation $\sigma = 0.012$ Å. Data points represent the 6 measured orders of Bragg reflection.

Figure 2. Low resolution specular reflectance profiles (rocking curves) across the first order Bragg peak for the as-prepared (solid) and annealed (dashed) samples. For the W/C sample 89-150 in (a) annealing causes the period, peak reflectance, and FWHM to increase, while for the W/Si sample 89-153 in (b) the same annealing treatment causes these quantities to decrease.

Figure 3. High resolution specular reflectance profile of the as-prepared, irradiated W/C sample 89-150 in (a) shows the effect of a carbonaceous film on the reflectance. (b) shows that exposure to an intense white synchrotron x-ray beam can change the structure and hence reflectance of this cooled W/C multilayer.

Figure 1a



Reflectance
10^0
10^−1
10^−2
10^−3
10^−4
10^−5
10^−6
10^−7
m=1
2
3
4
5
6
0
5
10
15
20
θ (degrees)

Figure 1b

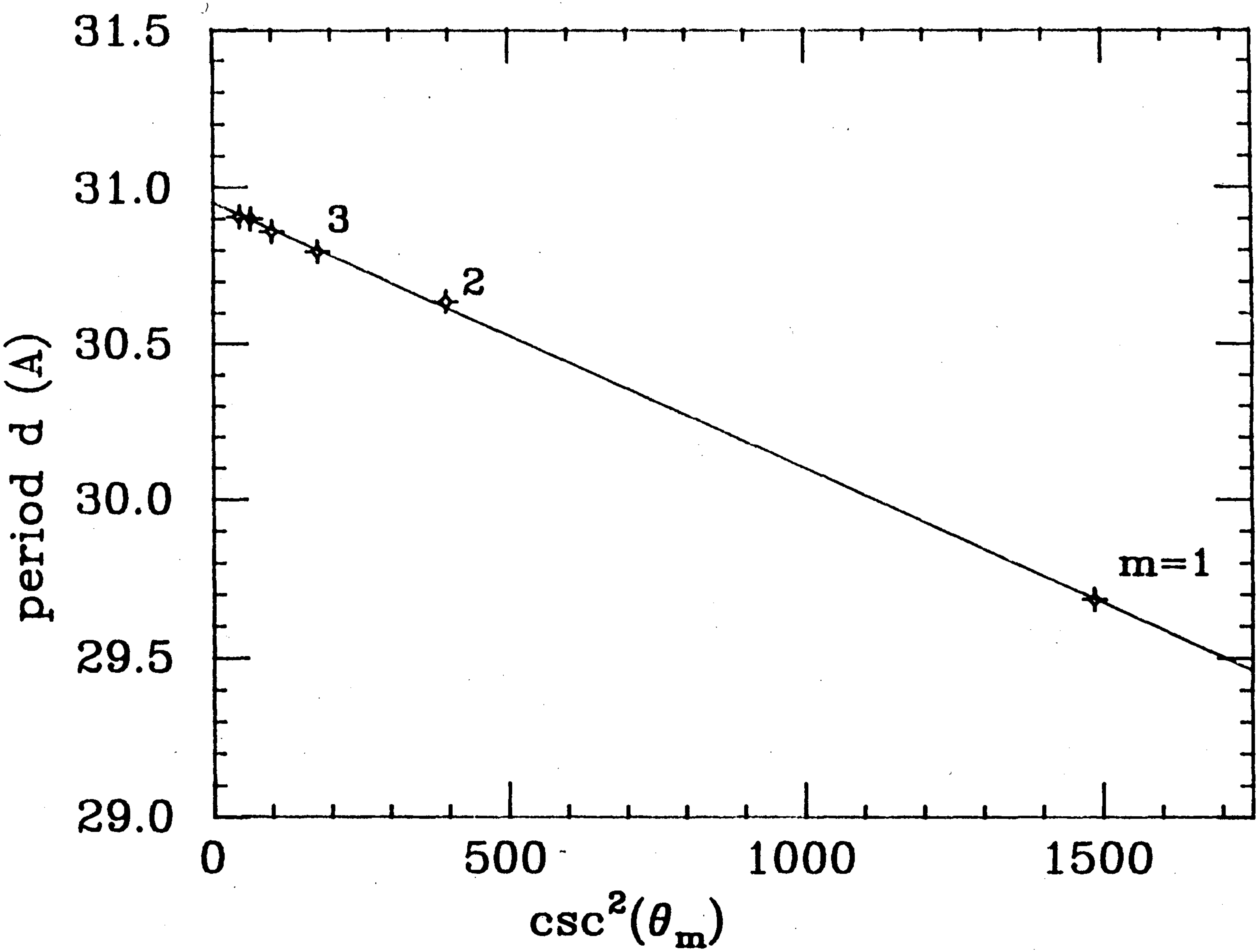
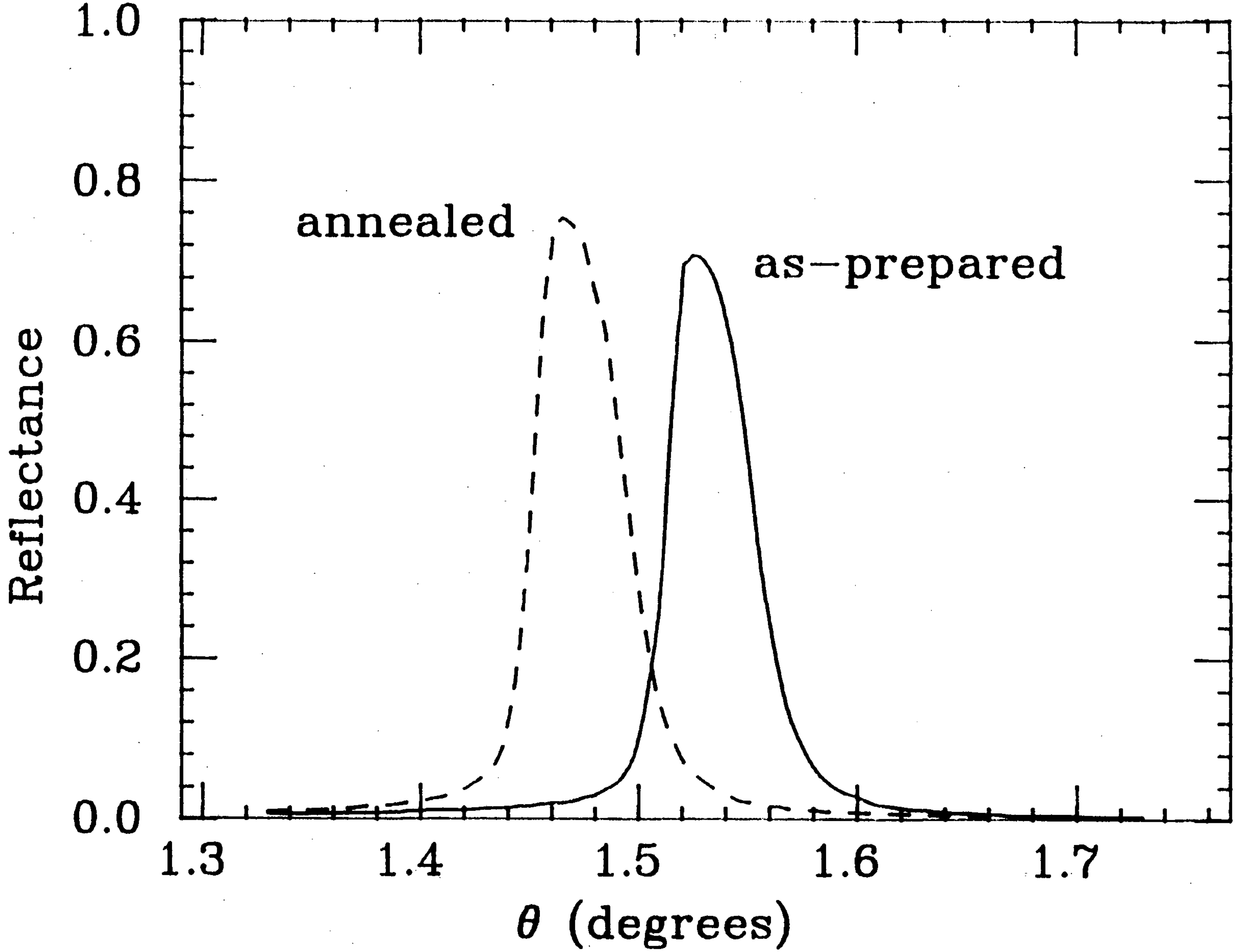


Figure 2a



annealed
as−prepared
Reflectance
1.0
0.8
0.6
0.4
0.2
0.0
1.3
1.4
1.5
1.6
1.7
θ (degrees)

Figure 2b

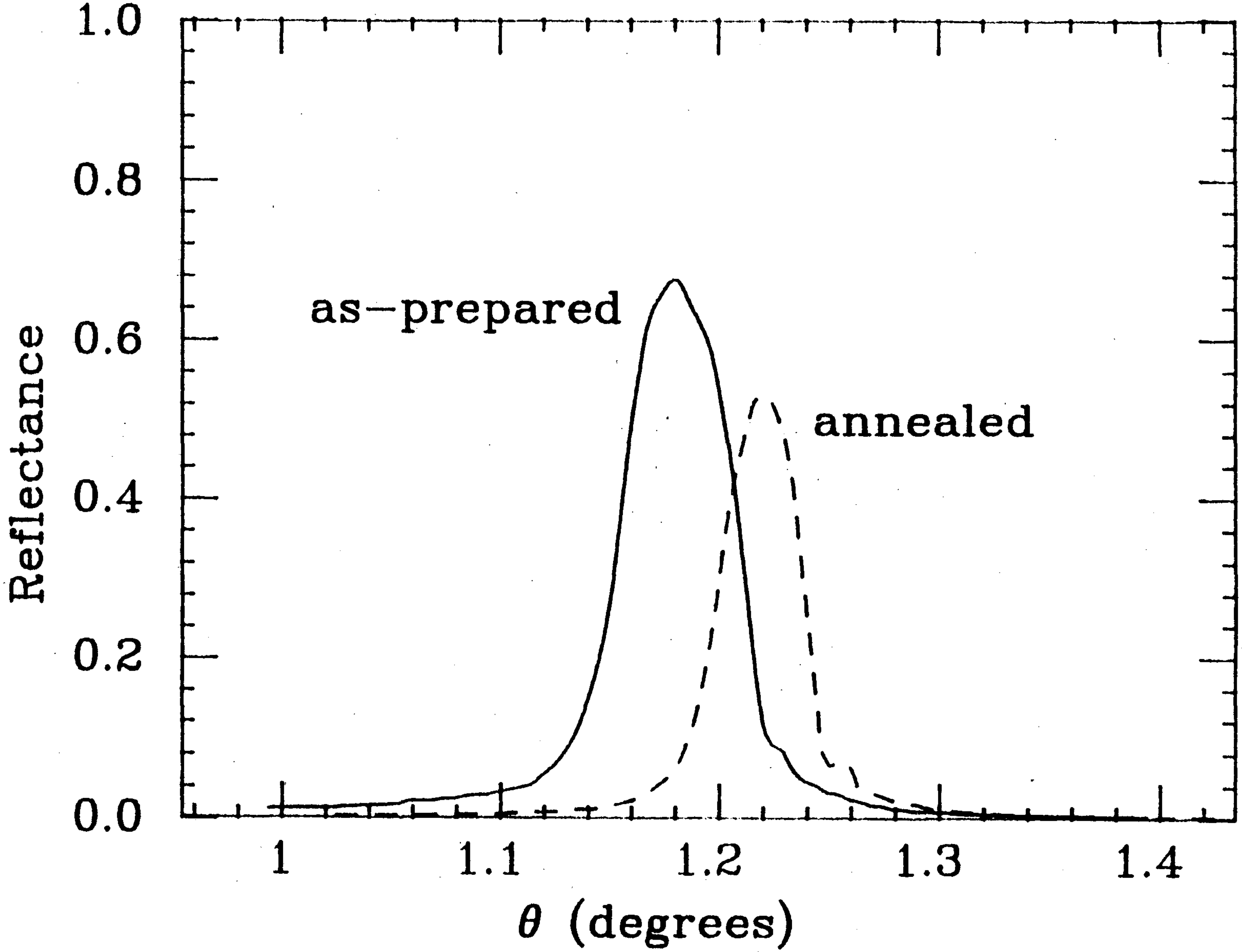
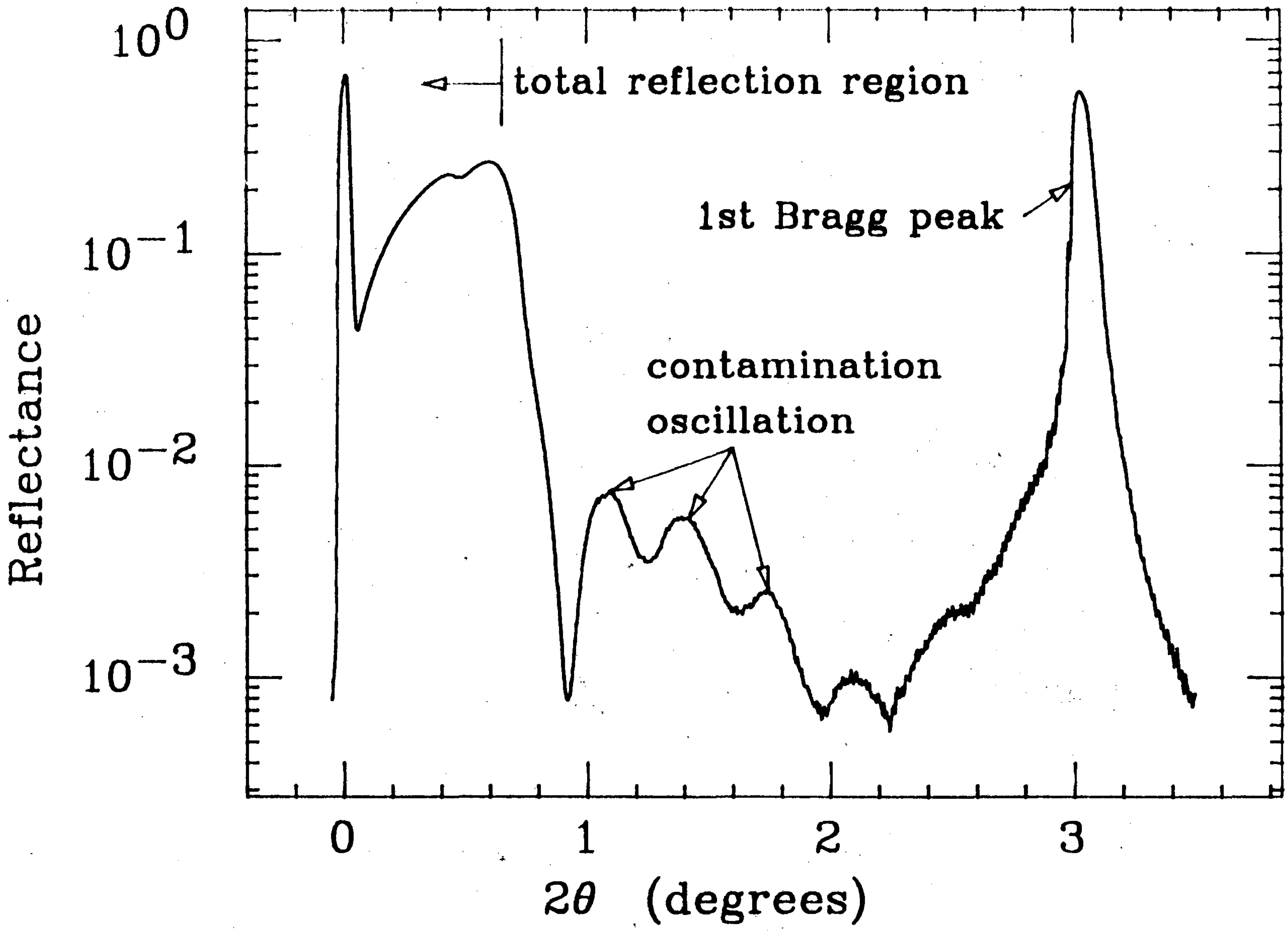
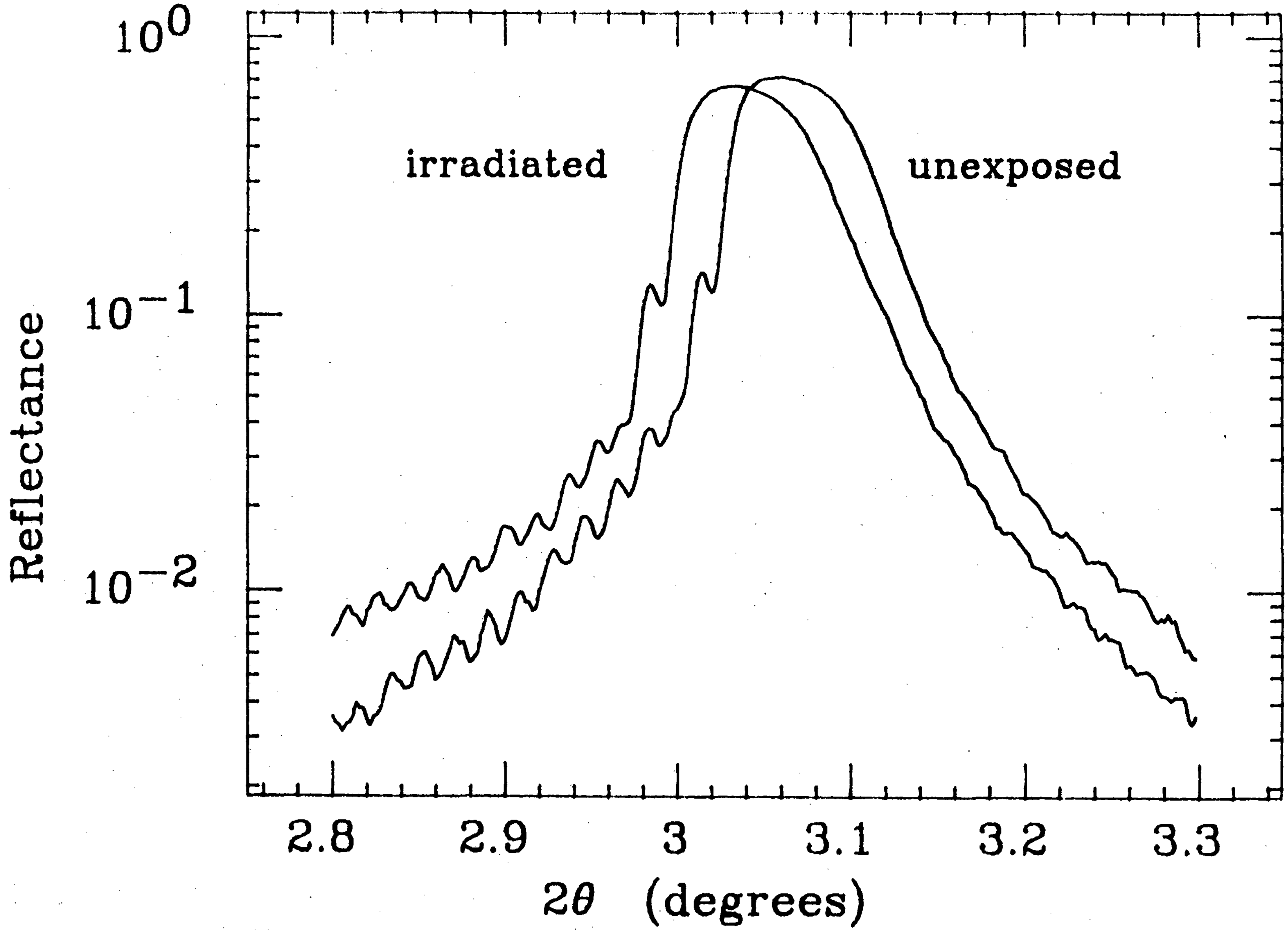

1.0
0.8
0.6
0.4
0.2
0.0
Reflectance
as−prepared
annealed
1
1.1
1.2
1.3
1.4
θ (degrees)

Figure 3a



10^0
10^{-1}
10^{-2}
10^{-3}
Reflectance
total reflection region
1st Bragg peak
contamination
oscillation
0
1
2
3
2θ (degrees)

Figure 3b



10^0
10^{-1}
10^{-2}
Reflectance
irradiated
unexposed
2.8
2.9
3
3.1
3.2
3.3
2θ (degrees)

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