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by

E. Spiller and A. Segmüller

IBM T. J. Watson Research Center Yorktown Heights

R.-P. Haelbich

Deutsches Elektronen-Synchotron DESY, Hamburg

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Eberhard Spiller and Armin Segmüller

IBM T. J. Watson Research Center Yorktown Heights, New York 10598

and

Rolf-Peter Haelbich

Deutsches Elektronen - Synchrotron DESY, D2000 Hamburg 52, West Germany and II. Institut fur Experimentalphysik, Universität Hamburg

Typed by Lorie A. Rinaldi (3277)

ABSTRACT

Multilayer coatings can in principle be used to obtain useful normal incidence reflectivity at any chosen wavelength and angle of incidence. For the practical realization, extremely smooth substrates and films and a good control of the deposition process are required. These requirements increase with decreasing wavelength; for wavelengths shorter than 50Å, atomically smooth films are required. We have fabricated multilayer coatings consisting of very smooth ReW and carbon films and tested them in the wavelength range λ=45-200Å and at λ=1.54Å. Our coatings show theoretical performance at normal incidence in the 150-200Å wavelength region. The films are sufficiently smooth to allow the fabrication of useful normal-incidence mirrors for wavelengths as short as 50Å. Improved thickness control has recently been obtained by measuring the soft x-ray reflectivity in situ during the film deposition.

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Grazing incidence mirrors[1] and zone plates[2] have been used as focussing elements for x-ray microscopes. Their resolution is limited by the resolution and tolerances in the fabrication of these elements. Normal incidence focussing mirrors require still tighter fabrication tolerances. However, the optical industry can fabricate spherical mirrors with about 50Å precision in the figure, a value that would immediately allow a resolution of about 500Å in a soft x-ray microscope. The 50Å tolerances in the figure of a mirror represent the limit of the testing methods presently used, and one can hope that an improvement of the testing capabilities in an optical shop might make further improvements possible.

All materials have very low normal incidence reflectivity in the soft x-ray region; some typical reflectivity values for the best reflectors are R=1% at λ=200Å, R=0.1% at λ=100Å and R=0.003% at λ=45Å.[3] Higher reflectivities can be obtained by using multilayer coatings, and theoretically such coatings can be designed to give a normal incidence reflectivity around R=30% for nearly any desired x-ray wavelength.[4,5] The practical realization requires that two materials with suitable optical constants are selected, which form stable, sharp boundaries. Haelbich and Kunz demonstrated that practically no diffusion occurs at the boundary between carbon and a heavy metal and obtained a reflectivity of R=3% at λ=190Å for a multilayer of gold and carbon.[6] The improvement of the reflectivity by the use of multilayer coatings at shorter wavelengths requires extremely smooth films and a well controlled deposition process, and we will describe our attempts to reach that goal in this paper.

MULTILAYER DESIGN

The performance of a multilayer coating can be calculated, if the thicknesses and optical constants of all layers are known.[7-10] For an optimized mirror (largest reflectivity with the smallest number of layers) one selects two materials with the largest possible difference in the optical constants. If the absorption of the films is very small, the thickness of each layer is adjusted such that all boundaries add in phase to the reflected wave, resulting in a periodic structure where each film is $\lambda/4$ thick (quarter wave stack). In the ultrasoft x-ray region, where only absorbing materials are available, absorption losses in a coating can be minimized by positioning strongly absorbing films close to the nodes of the standing wave produced by the superposition of the incident and reflected wave and by filling the space between the nodes (the antinodes) with a material of the smallest possible absorption.[4] Because the standing wave ratio changes throughout the depth of a multilayer coating, the design optimization leads to a nonperiodic design; the films of the strongly absorbing materials become thinner towards the top of a coating where the standing wave is more pronounced. Design of an optimized mirror coating is straightforward on a digital computer.

Figure 1 gives as an example the calculated reflectivity at $\lambda=190Å$ for an optimized multilayer coating of two materials with the optical constants $\tilde{n} = n-ik$ as indicated in the figure. The optical constants used are the values for Re given in Ref. 8 and those for carbon from Ref. 3. During the deposition of Re (full curves) the reflectivity increases. Slightly before a maximum in the reflectivity is reached, the Re deposition is terminated and carbon is deposited (dashed curves). The reflectivity decrease during the carbon deposition is smaller than the increase during the previous Re deposition due to the smaller reflectivity at the carbon-vacuum interface and due to the smaller absorption of carbon. If a material with $n=1$ and $k=0$ could be chosen as a spacer between the different Re layers, the reflectivity would not decrease during the deposition of the spacer material, and the reflectivity of the multilayer coating would be correspondingly larger with the limit $R\to 1$ for a large number of layers. The example in Fig. 2, calculated for $\lambda=45Å$ and an angle of incidence of 65° with the optical constants of Au and C given in Ref. 3, shows a smaller decrease in reflectivity during the carbon deposition than Fig. 1 due to the smaller absorption of carbon at this wavelength. For this graph we have deliberately introduced some deviation (in the 5% range) from the optimum thicknesses. The graph shows that these thickness errors have only a small influence on the reflectivity (see the decrease in reflectivity because the 9th and 11th layers are too thick). However, we have to note that the accumulated thickness error throughout an entire multilayer stack has to remain smaller than about $\lambda/10$. Therefore, during the fabrication we

expect no serious problem in the thickness control for each individual film but have to be careful about accumulated thickness errors for a coating with many layers.

SCATTERING

A surface has to be smoother than about $\lambda/20$ if we request that the amount of scattered radiation is small compared to the specularly reflected intensity near normal incidence. The simplest theory which describes the reduction of the reflectivity due to scattering gives for the reflectivity

$$R = R_0 \exp\left[-\left(\frac{4\pi\sigma\cos\alpha}{\lambda}\right)^2\right] \qquad (1)$$

where R_0 is the reflectivity of a smooth surface of the same material, σ is the rms height of the surface irregularities, which are assumed to be gaussian, α the angle of incidence and λ the wavelength. Equation 1 is known as the Deby-Waller factor in x-ray diffraction[9] and is used to calculate the intensity reduction due to the thermal motion of the atoms. A thorough theoretical treatment of surface roughness, especially with respect to microwaves, is given in Ref. 10. References 11-15 discuss the theory and its application for the spectrum of the visible light to the x-ray range and contain many more references. The theory for the scattering from multilayer coatings is still more complex.[16-20] For the simplest case - that the roughnesses of all boundaries is identical to each other and to the substrate (perfect replication) - the theoretical results are very close to those obtained for a single surface, and Eq. 1 represents a good approximation.[16,18] A boundary between two layers in a multilayer stack produces very little scattering if it is located near a node of the standing wave generated in a coating.[16] Therefore, a multilayer coating can be designed for minimal scattering in a similar way, as it can be designed for minimal absorption.[4]

For our work we were interested in knowing the influence of roughness on the reflectivity of a coating and wanted to be able to separate this influence from the influence of other parameters like thickness errors and uncertainties in the optical constants. For this purpose we define an effective roughness σ of our coating in such a way that Eq. 1 produces a best fit to the measured reflectivity. Roughness has very little effect on the reflectivity at very small glancing angles $\theta=90°-\alpha$ for any wavelength. Therefore, a measurement of the reflectivity as a function of angle of incidence at a properly chosen wavelength allows to obtain the effective roughness from Eq. 1 where R_o is obtained from the data in the region $\cos\alpha = \sin\theta \rightarrow 0$. The effective roughness σ summarizes the effect of all actual irregularities in our films on the reflectivity, and we use it as a convenient parameter to characterize a coating, especially to predict by use of Eq. 1 from measurements at one wavelength and within a certain range of angles of incidence, the performance at other wavelengths and angles of incidence.

EXPERIMENTAL

We fabricated multilayer coatings consisting of a ReW alloy and a spacer layer of C (and in some cases B) by electron beam evaporation in a conventional vacuum system with pressures around 10^{-6} torr. We found films of ReW to be smoother than films of pure Re or pure W and to be considerably smoother than thin films of gold.[21] The structure of the films is insensitive to the exact composition, to small contamination and to the substrate temperature in the region from 77°K to 500°K. The thickness of the films was originally only monitored with an oscillating quartz crystal, and the reflectivity of a coating was later tested either in a reflectometer for ultrasoft x-rays at DESY[22] or with a computer-controlled x-ray diffractometer at $\lambda=1.54Å$[23,24] Recently, we installed an additional x-ray monitoring system that measures the reflectivity of a coating for soft x-rays (most conveniently carbon radiation with $\lambda=44.8Å$) directly during the deposition of the film. The main advantage of this system is the immediate feedback of the results and the possibility to correct a thickness error made in one layer during the deposition of the next layer.

EXPERIMENTAL RESULTS

Figure 3 shows the near normal incidence reflectivity for a single ReW film and a 3, 5 and 7 layer coating of ReW and C. Theoretical curves calculated with the published optical constants of Re[8] and C[3] are also given for comparison. All multilayer coatings in Fig. 3 are deposited during the same run, and a shutter was moved between the source and the substrate after the deposition of layer 3 and 5 to mask part of the substrate. The fact that all coatings have their maximum at about the same wavelength indicates that thickness errors should have very little effect on the performance for this coating. Many of our coatings have a poorer performance than shown in Fig. 3. Figure 4 shows the maximum reflectivity obtained from a large number of coatings as a function of the wavelength of the reflectivity maximum. The theoretical curves for optimum design are again given for comparison. We attribute the scatter in the data to thickness errors in the film. A coating with poor performance shows usually a difference in the wavelength of the maximum between coatings of different number of layers produced during the same deposition run. The fact that some of our coatings have maximum reflectivities above the theoretical values indicates that the optical constants used do not fully describe our films.

Figure 5 is a reflectivity measurement at a wavelength $\lambda=1.54Å$ for glancing angles θ up to 4° for an 11-layer coating. This coating has a reflectivity maximum around $\lambda=190Å$ near normal incidence. At $\lambda=1.54Å$ the reflectivity maximum appears at a glancing angle θ slightly under 0.6°. The thicknesses of the ReW layers in this coating were not chosen to give maximum reflectivity but rather to produce strong higher order maxima which appear at larger glancing angles. We can determine the thickness of each layer by a best fit to a theoretical curve, such that the theoretical curve (dashed) describes the angular positions of all extrema in the experimental curve correctly. We see that discrepancies between the experimental and theoretical curve appear at larger glancing angles. If we assume that these discrepancies are due to a roughness, we can try to obtain a better fit by multiplying the theoretical curve with Eq. 1 and a properly chosen σ. For the example in Fig. 5, we have to select an effective roughness $\sigma=3Å$ to obtain good agreement.[21] Using Eq. 1 we can predict the influence of roughness at other wavelengths and angles of incidence. At the top scale of Fig. 5 we have indicated the wavelength at which we would observe the same reduction in reflectivity at normal incidence. The figure shows that the smoothness of the films should allow utilization of the interference effect for wavelengths as short as $\lambda=44$Å near normal incidence. To check the validity of Eq. 1 for this extrapolation, we had made with one coating the equivalent measurement as in Fig. 5 also at $\lambda=55$Å and obtained the same value for σ for both wavelengths.

From the measurements in Fig. 5 we cannot distinguish if the effective roughness of our coatings is due to the roughness of the substrate or if some roughness is generated during the deposition of the multilayer films. We believe, however, that the substrate is the main source for the observed effective roughness. One support for this belief is the observed trend that the effective roughness σ decreases for an increasing number of layers. This observation gives us hope that the deposition of multilayers might be used to smoothen substrates and make them this way more suitable for x-ray mirrors. The smoothest substrate which we found are 111 silicon wafers, and most of our coatings were deposited on this substrate. Superpolished quartz[12,25] gives considerably bigger values for σ. Figure 6 shows, as an example, the reflectivity at $\lambda=1.54$Å of a 9-layer coating deposited simultaneously on a Si wafer (full curve) and on a superpolished quartz substrate (dashed curve). At a glancing angle of 2° the quartz substrate gives more than a factor of 10 smaller reflectivities, and we cannot expect any useful reflectivity for a normal incidence mirror around $\lambda=50$Å on this substrate. We can determine the effective roughness of the quartz substrate from the ratio of the two curves in Fig. 6. With Eq. 1 and $\sigma=3$Å for the silicon substrate, we obtain a value of $\sigma=9$Å for our superpolished quartz.

For the construction of focussing elements for a future x-ray microscope, the results in Fig. 6 appear to be not very encouraging because spherical mirrors which are smoother than superpolished quartz are presently not available. Our main hope is that the multilayer deposition will have a smoothing effect, such that very low effective roughness will be observed for a coating with a large number of layers even for a substrate that was initially too rough. It appears that similar smoothing effects have already been observed by other authors.[26,27]

Figure 7 is a part of the strip chart recorder trace obtained with the recently installed in-situ reflectivity monitor system. Shown are the count rate of the flow proportional counter for photons with $\lambda=44.8$Å reflected from the sample at $\alpha=63°$ and the frequency shift of the quartz crystal oscillator. The pulses from a flow proportional counter are sent into a pulse heights analyzer which counts only pulses of the proper heights and eliminates most of the noise produced by the evaporation. This noise has still a small effect on the count rate, which can be recognized in Fig. 7 immediately after the shutter is opened for the deposition of the first film. We have marked in Fig. 7 the point after which the signal changes due to the deposition of the first ReW film and not due to a change in the noise background. The initial decrease in reflectivity is not visible in the corresponding theoretical plot of Fig. 2; it is, however, also obtained theoretically if proper optical constants for the substrate are used for the calculation. The expected increase of the reflectivity during the deposition of the ReW layers is clearly visible in Fig. 7, while the small decrease expected during the carbon deposition is within the noise of the system. Therefore, we cannot immediately deduce the proper carbon thickness, but have to deduce it from the change of the reflectivity during the deposition of the next ReW layer. We recognize, for example, from the initial decrease in the reflectivity during the deposition of layer 13, that the carbon thickness in layer 12 was slightly too small; and we can correct this error by making the next layer slightly thicker. Layers 20 and 21 show an example where the thickness error was too large for a subsequent correction in the next layer. The observed reflectivity at the end of each ReW deposition in Fig. 7 is compared in Fig. 8 to the theoretical values obtained from Fig. 2. Due to the uncertainties in the optical constants, the absolute values given in Fig. 8 can easily be a factor of 2 or more too large.

The monitoring of the thickness by means of the oscillations in the reflectivity can be made more reliable by a proper selection of the spacer material and the monitoring wavelength. Carbon produces very little change in the signal for carbon radiation because its optical constants for carbon radiation are very close to $n=1$ and $k=0$. We obtain a stronger decrease in the signal if we choose a more absorbing spacer material as for example boron. This will produce a lower total reflectivity at the monitor wavelength; however, the strong decrease during the boron deposition allows an immediate control of the thicknesses of the boron layers. If the mirror is later used at a wavelength $\lambda>67$Å where boron has little absorption, reflectivity even higher than that possible with a carbon spacer might be obtained. On the other hand, if a coating is to be used around $\lambda=45$Å where a carbon spacer is desirable, a monitor wavelength different than $\lambda=45$Å, as for example $\lambda=67$Å (BK_α) or $\lambda=13$Å (CuL), might offer an advantage. Using carbon radiation with $\lambda=44.8$Å for monitoring, we have produced a ReW-B multilayer coating without any noncorrectable thickness error. An earlier coating with 51 layers, which still contained some thickness errors, gave a normal incidence reflectivity of 1.6% at $\lambda=100$Å compared to $R<0.05\%$ for a single film of ReW in a test at the DESY reflectometer.

SUMMARY

We have reported the present state of our efforts to fabricate normal incidence reflectors consisting of evaporated multilayer films. To obtain high normal incidence reflectivity in the soft x-ray region, very smooth substrates and films and very good thickness control during the deposition are required. The installation of an in-situ reflectivity monitor using $\lambda=44.8$Å has greatly improved film thickness control and made it possible to evaporate up to 50 layers, all adding in phase to produce enhanced normal incidence reflectivity around $\lambda=100$Å. We deduce that the effective roughness of multilayer films on 111 silicon substrates is around $\sigma=3$Å. Some indications that coatings with more layers tend to become smoother give us hope that also curved mirror substrates which are not sufficiently smooth to reflect soft x-rays near normal incidence, might be smoothened by the deposition of multilayer coatings such that they become useful as focussing elements. We are confident that further progress will allow to use multilayer coatings in combination with normal incidence mirrors for ultrasoft x-ray microscopy down to the carbon K-edge.

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Fig. 1 Calculated normal incidence reflectivity at $\lambda=190$Å for a multilayer coating as a function of total thickness as it might be observed during the deposition of the coating. The optical constants indicated are values published for Re[8] and Au[3]. During the deposition of Re the reflectivity increases (full curves) and decreases during the subsequent C deposition (dashed curves). Thickness of each layer starting at the substrate with Re: 53, 54.5, 47.3, 56.9, 44.3, 58.9, 42.6, 61, 40.2, 62, 41Å.

Fig. 2 Calculated reflectivity Rs, Rp for s and p polarization at $\lambda=45$Å and an angle of incidence of 65°. The optical constants are those published for Au and C. The full curves represent the deposition of the heavy material (Au), the dashed that of the light material (C). Thickness of each layer: 28, 28, 27, 28, 26, 28, 26, 28, 28, 29, 25, 29, 24Å.

Fig. 3 Measured and calculated reflectivity of a single 400Å thick ReW film and of a 3, 5 and 7 layer coating of ReW and C. The 3 and 5 layer coatings were obtained by shadowing part of the wafer by a shutter during the evaporation. The optical constants of Re (Ref. 8) are used for the calculation, thickness of each layer starting at the Si substrate with ReW are: 56.9, 60, 49.9, 62.8, 47.3, 64.6, 49.4Å.

Fig. 4 Measured peak reflectivity R_{max} versus wavelength of the reflectivity maximum λ_{max} for a large number of fabricated coatings (points) and theoretical values for the peak reflectivity for optimum designs (curves). The scatter in the experimental data is due to thickness errors during the deposition.

Fig. 5 Measured (full curve) and calculated (dashed curve) reflectivity versus glancing angle at $\lambda=1.54$Å for an 11-layer coating of ReW and C. Thickness values for the calculated curve, starting at substrate: 22.2, 70.8, 16.2, 72.3, 15, 25.6, 14, 86.3, 12.2, 80, 11.2Å; refractive index of ReW and C: $1-4.4\times10^{-5}-i\cdot6.8\times10^{-6}$ and $1-6.8\times10^{-6}-i\cdot0.11\times10^{-6}$. Smooth films are assumed for the theoretical curve.

Fig. 6 Measured reflectivity at $\lambda=1.54$Å versus glancing angle for 9-layer coatings of ReW and C deposited simultaneously on a silicon (full curve) and a superpolished quartz (dashed curve) substrate.

Fig. 7 Recorder traces of the reflectivity at $\lambda=44.8$Å, $\alpha=63°$ and the frequency shift of a quartz oscillator obtained during the deposition of a 21-layer ReW-C multilayer coating. Preparation time between the deposition of the different layers has been cut out. Note the change in scale for the reflectivity after layer No. 2, 8 and 17.

Fig. 8 Theoretical (...) and measured reflectivity (xxx) at the end of the deposition of each ReW layer in a multilayer coating. The theoretical points correspond to the maximum in Fig. 2; the experimental points are obtained from Fig. 7 and given in arbitrary units.

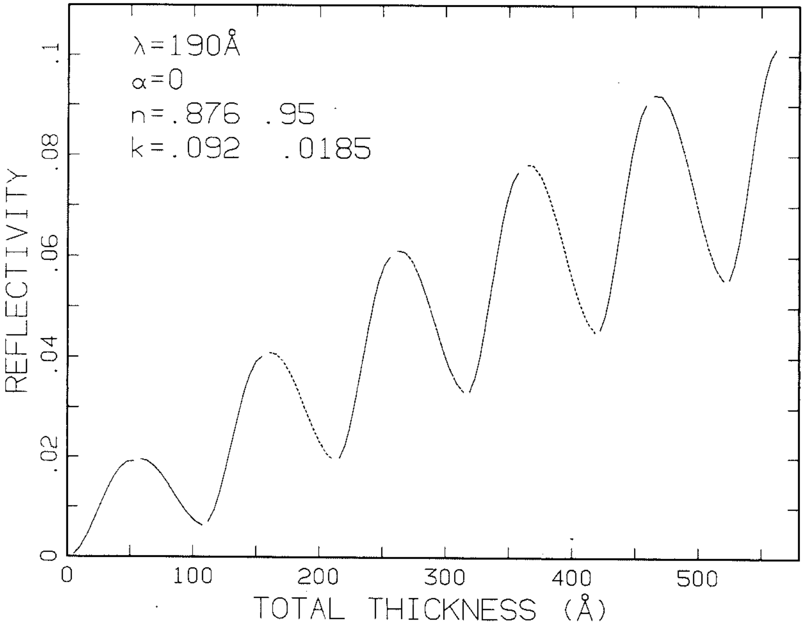

Fig. 1

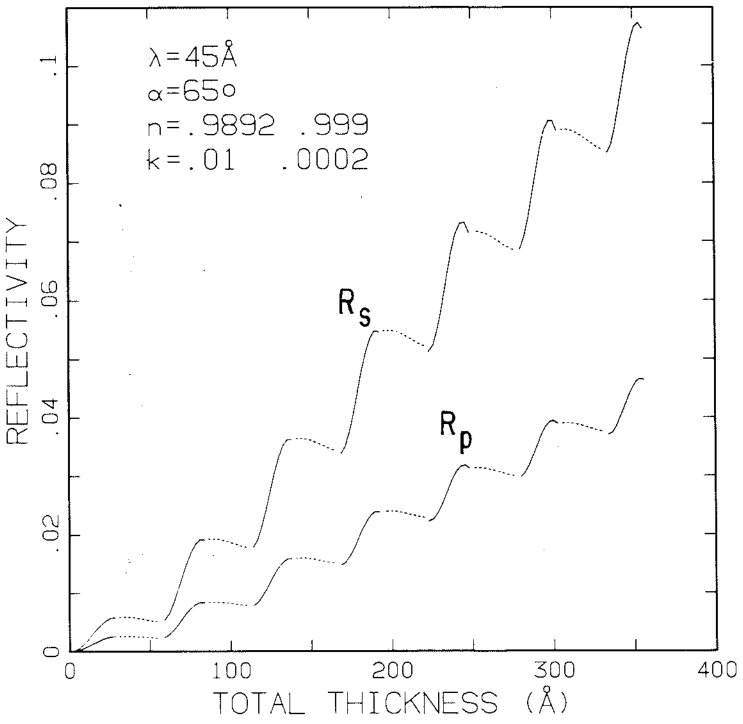
λ=45Å
α=65°
n=.9892 .999
k=.01 .0002
R_s
R_p
REFLECTIVITY
0
.02
.04
.06
.08
.1
0
100
200
300
400
TOTAL THICKNESS (Å)

Fig. 2

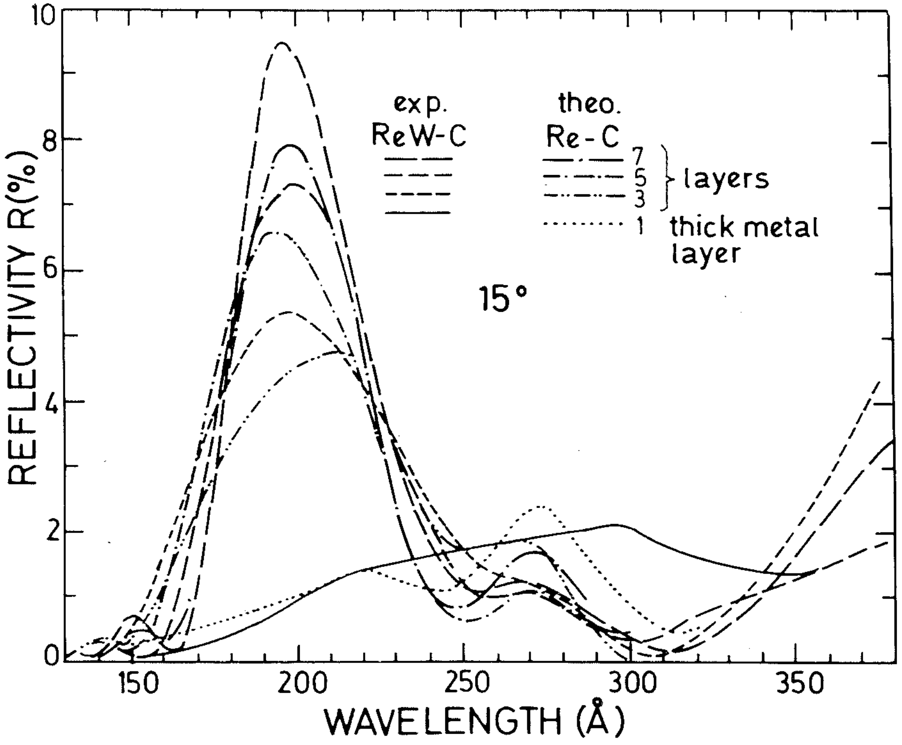
exp.
ReW-C
theo.
Re-C
7
5
3
layers
1
thick metal layer
15°
REFLECTIVITY R(%)
0
2
4
6
8
10
150
200
250
300
350
WAVELENGTH (Å)

Fig. 3

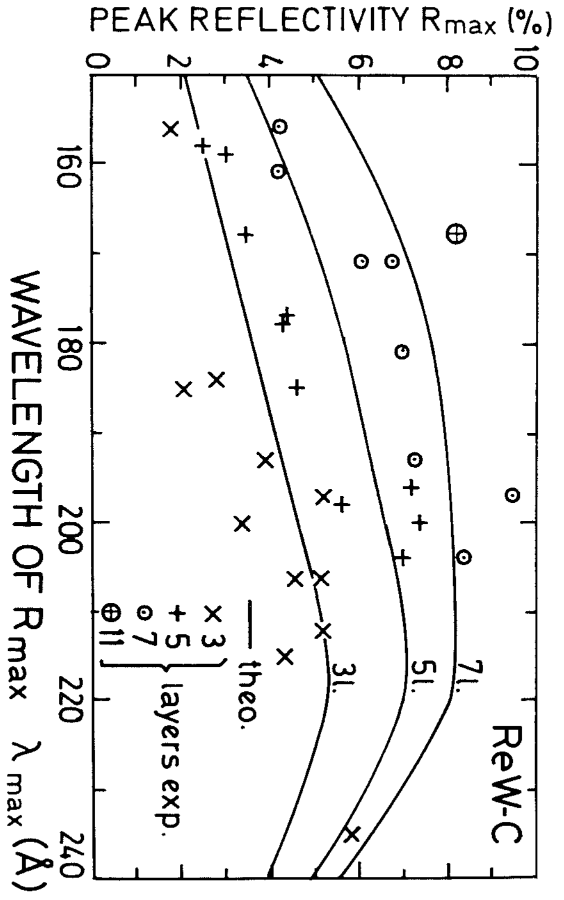
PEAK REFLECTIVITY R_{max} (%)
0
2
4
6
8
10
WAVELENGTH OF R_{max} λ_{max} (Å)
160
180
200
220
240
ReW-C
7l.
5l.
3l.
— theo.
× 3
+ 5
⊙ 7
⊕ 11
layers exp.

Fig. 4

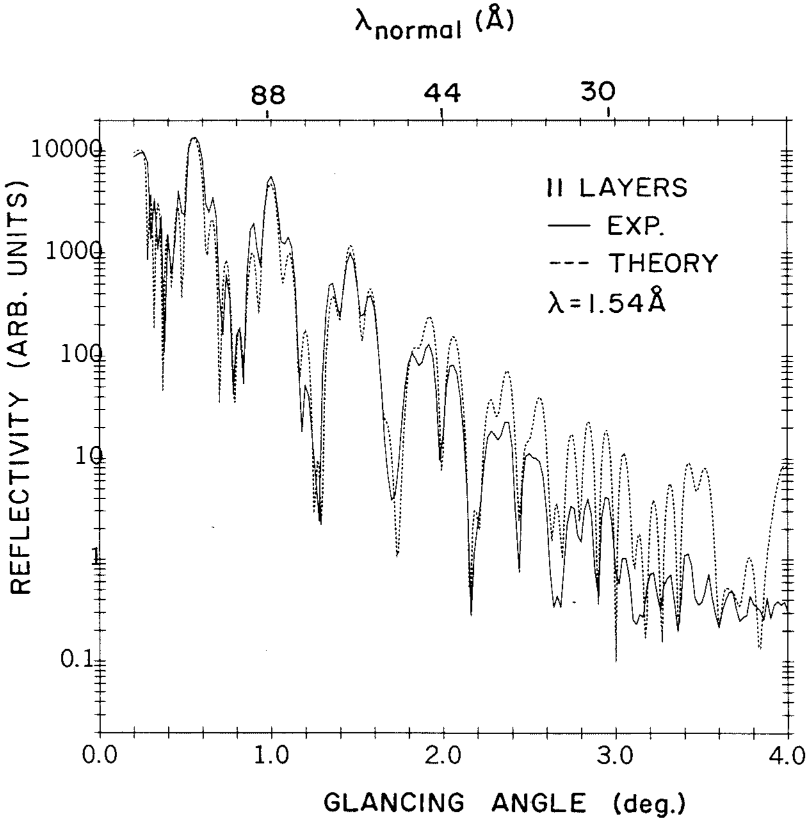
λ_{normal} (Å)
88
44
30
REFLECTIVITY (ARB. UNITS)
10000
1000
100
10
1
0.1
0.0
1.0
2.0
3.0
4.0
GLANCING ANGLE (deg.)
11 LAYERS
— EXP.
--- THEORY
λ = 1.54 Å

Fig. 5

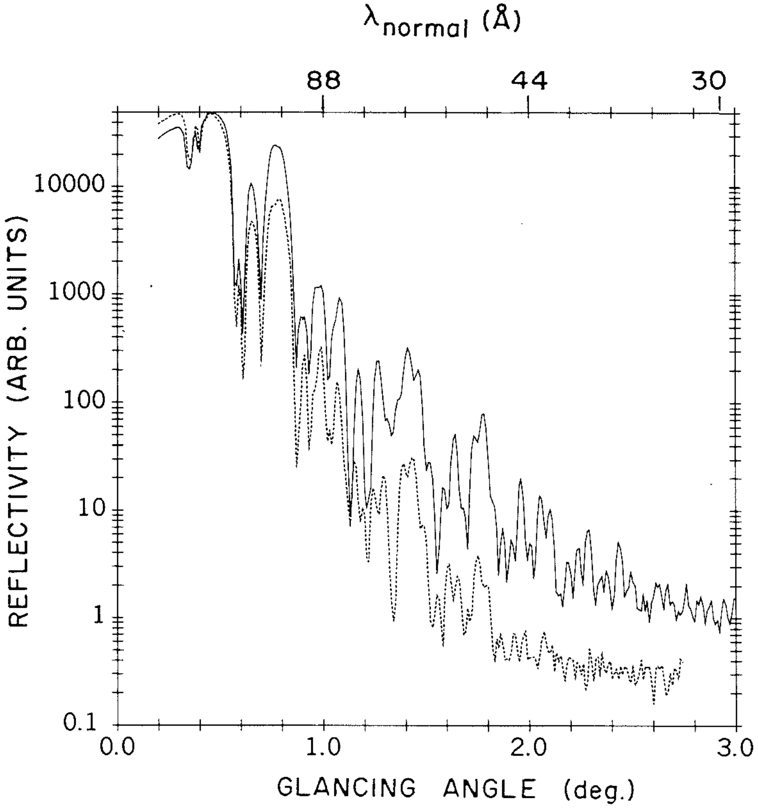
λnormal (Å)
88
44
30
10000
1000
100
10
1
0.1
REFLECTIVITY (ARB. UNITS)
0.0
1.0
2.0
3.0
GLANCING ANGLE (deg.)

Fig. 6

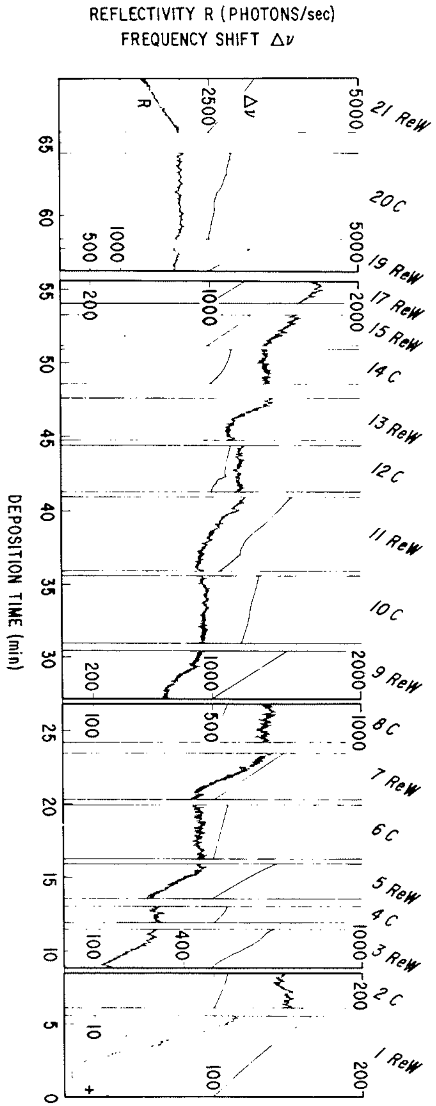
REFLECTIVITY R (PHOTONS/sec)
FREQUENCY SHIFT Δν
DEPOSITION TIME (min)
R
Δν
21 ReW
20 C
19 ReW
17 ReW
15 ReW
14 C
13 ReW
12 C
11 ReW
10 C
9 ReW
8 C
7 ReW
6 C
5 ReW
4 C
3 ReW
2 C
1 ReW

Fig. 7

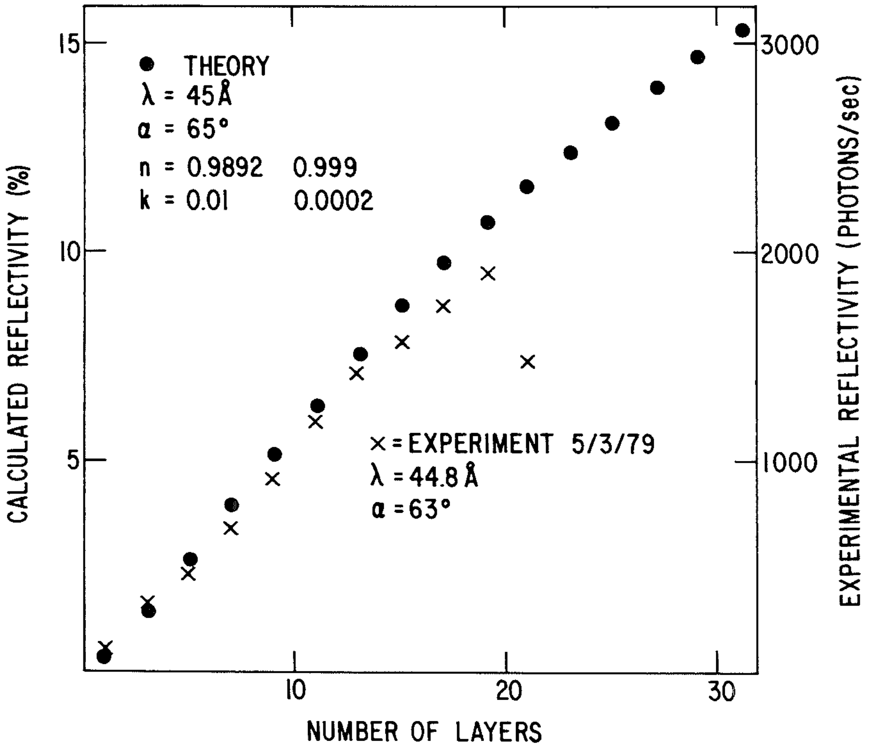
● THEORY
λ = 45Å
α = 65°
n = 0.9892 0.999
k = 0.01 0.0002
× = EXPERIMENT 5/3/79
λ = 44.8 Å
α = 63°
CALCULATED REFLECTIVITY (%)
EXPERIMENTAL REFLECTIVITY (PHOTONS/sec)
NUMBER OF LAYERS
5
10
15
10
20
30
1000
2000
3000

Fig. 8