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TITLE SYNCHROTRON BASED MEASUREMENTS OF THE SOFT X-RAY PERFORMANCE OF THIN FILM MULTILAYER STRUCTURES

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SYNCHROTRON BASED MEASUREMENTS OF THE SOFT X-RAY PERFORMANCE OF THIN FILM MULTILAYER STRUCTURES

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INTRODUCTION

It has been demonstrated by a variety of groups that thin film multilayer structures or layered synthetic microstructures (LSMs) are high reflectivity x-ray coatings.(1-4) These coatings have found applications in astrophysics,(5) high temperature plasma diagnostics,(6) x-ray flourescence analysis,(7) and x-ray physics.(8) These and future applications require an accurate way to measure the performance of structures that can be fabricated, and to guide improvements in the fabrication program. Using synchrotron radiation, we have developed a measuring system to test the performance of LSMs from 50 to 500 eV. In this paper we will review our measurement techniques and compare our results to theoretical predictions of LSM performance.

MEASUREMENTS

A synchrotron provides broad band radiation from the infrared to the hard x-ray range. In the soft x-ray range the radiation has a small divergence and is strongly polarized. A high resolving power monochromator is be used to define a small continuously tunable spectral band across the soft x-ray range. It is this tunability feature that is most important for our measurements. These experiments do not require large radiation fluxes. Therefore they can take advantage of operating conditions not suitable for other experiments. We have performed measurements at two synchrotrons, the Stanford Synchrotron Radiation Lab (SSRL) with a grasshopper type monochromator (9) and at the National Synchrotron Light Source using a plane grating monochromator.(10)

All of our measurements are performed in an ultra high vacuum environment (10^{-9} Torr). The sample chamber holds up to 16 samples per load (see Fig. 1). The sample angle (θ) and the detector angle are independently controlled. The measurements were performed by first setting the sample angle and then independently setting the detector angle, using the appropriate energy radiation to set the position of the detector. The monochromator is scanned across the energy range of interest. We have used two methods of normalization. In both cases after a scan of the reflected radiation the detector is positioned in the incident beam and an identical (normalization) scan is made of the reflected radiation. To reduce the effects of synchrotron intensity fluctuations, i.e. the decay of the storage ring electron beam current or movement of the electron beam relative to the monochromator, a monitor was used in all experiments to minimize the effects of these variations. Two methods were used in our experiments. One, all x-ray signals were normalized to the electron current in the storage ring at the time of measurement. This reduces the effect of electron beam decay but has no effect on beam wander. Two, an in situ x-ray beam monitor was used. This detector uses a screen as a proto-cathode which transmits most of the incident radiation to monitor the x-ray flux while a scan is being taken. Both methods have produced excellent and consistent results.

Both detectors use aluminum oxide photocathodes to produce electrons which are detected by a channeltron detector and counted with pulse counting electronics. The detectors are biased to reject externally generated electron signals. It is interesting to note that the use of ion pumps with such a detector can cause a large background signal. We have been successful in operating this experiment with very low background levels.

Our experiment consists of measuring the absolute reflectivity of LSMs at a variety of fixed angles as a function of energy. In the rest of this paper we will review some of our measurements and their relationship to calculations of the LSM performance using standard models.(11) We have examined a large variety of samples with individual layer thicknesses, d, from 10 to 100A, with 14 to 160 layerpairs and made with more than fifteen different materials. We are indebted to Energy Conversion Devices (J. Keem), IBM (E. Spiller), and Stanford University (T. Barbee) for supplying the multilayers used in all of our experiments.

RESULTS AND ANALYSIS

Fig. 2 is an example of a diffraction profile of a Ni-C LSM with an experimental reflectivity of 15 percent at 173 eV. At an angle of incidence of 30 degrees this reflectivity represents a significant improvement over any other method of reflecting soft x-rays at non-grazing angles. This and all other sample reflectivities we have measured are less than one would predict from an idealized theory,(1-3, 11, 12) i.e. uniform layer thicknesses and densities, well known optical constants, perfectly smooth and sharp interfaces, and samples that are flat. Many of the discrepancies between theory and experiment can be explained if one or more of these conditions are not met. We have attributed the reduced experimental reflectivity to sample roughness and the structure in the wings of the profile to changes in the layer thicknesses as a function of depth into the sample. These issues will be discussed later in this paper.

With the tuneability of a synchrotron radiation source monochromator combination we can examine the reflectivity of any sample as a function of energy for s polarized light. In Fig. 3 we show the peak reflectivitiy of several samples as a function of energy. We have compared these results to idealized theory and to empirically corrected theory for surface roughness, which is considered constant for a given sample. This correction R_R has the form (1, 13)

$$R_R = \exp[-2\pi\sigma/d]^2 \quad . \tag{1}$$

Table I shows the inferred roughness from our data and equation (1). The magnitude of the inferred roughness and the sample deviations indicate that a simple correction of this form may be a reasonable interpretation of the failure of the LSMs to reach theoretical peak reflectivities.

TABLE I

Calculated Roughness

LSM	Calculated Roughness (Å)	Sample Deviation (Percent)
Fe-C	4	20
Co-C	8	30
V-C	4	15
Cr-C	6	12

Like all diffracting elements a LSM will reflect higher order radiation as well as the fundamental. Fig. 4 shows the first three orders of a ReW-C multilayer with 17 layerpairs and a 2d spacing of 175A. Idealized theory predicts that the higher order reflectivity will fall off as n^2 where n is the order number. In addition the resolving power, E/dE, where dE is the FWHM of the diffraction profile, should improve linearly with increasing n. Any discrepancies in these values can be related to imperfections in the LSM and changes in the optical constants with energy. The latter feature also manifests itself as a departure in the energy position of the peak reflectivity from an integral multiple of the fundamental energy.

TABLE II

Harmonic Reflectivity and Resolving Power

n	R	$R*n^2$	E (eV)	E/n (eV)	E/dE	E(dE*n)
1	0.06	0.06	87	87	17	17
2	0.015	0.06	166	83	33	17
3	0.002	0.018	248	83	41	14

Table II is based upon the data presented in Fig. 4, and demonstrates these features for a large 2d spacing ReW-C sample. We see excellent agreement in second order for the normalized (corrected for order number) reflectivity, $R*n^2$ and normalized resolving power, E/(dE*n). The normalized energy position of the peak in reflectivity, E/n, has shifted in second and third order due to changes in the optical constants with energy. The normalized data in third order shows a degradation in performance and indicates the presence of imperfections in the structure of the LSM.

The resolving power of a LSM is proportional to n, the number of layerpairs participating in the diffraction process. The number of pairs participating may be a strong function of energy, i.e. the penetration of the radiation into the LSM may be limited by reflective or absorptive losses. Table III demonstrates this effect in a ReW-C LSM where absorption has reduced the resolving power by a factor of over 2.5 as a result of the increase in absorption for energies greater than 280 eV, i.e. above the carbon edge.

TABLE III

Resolving Power as a Function of Energy

LSM: ReW-C, 2d = 40 A, 64 layer pairs

E (eV)	E/ΔE
170	64
200	75
400	25

If p-polarized light is used a strong dip in the reflectivity can be observed at Brewester's angle. For x-rays the index of refraction is approximately one, therefore Brewster's angle is 45 degrees. This phenomenon can be easily observed in Fig. 5 as a strong depression in the reflectivity near 45 degrees.

We have also been able to test the long term stability of LSMs. A Fe-C sample was retested after storage for 12 months at ambient laboratory conditions. No change in performance of this sample could be detected.

With the success of individual x-ray mirrors a natural extension of this activity is to fabricate more complicated structures. One such structure is a Fabry-Perot etalon, two LSMs separated by a spacer material. Fig. 5 shows the diffraction profile of such a structure, the shape of this structure is closely related to a two slit diffraction profile modulated by the reflectivity envelope of the LSMs comprising the structure.[14, 15] This is a direct result of the large absorption and small reflectivities in the soft x-ray range which limits the number of reflections that can participate in the interference pattern.

SPUTTERING VERSUS EVAPORATION

Evaporation affords the fabricator the chance to monitor the LSM while forming the layers and maximize the reflectivity during the fabrication process. This process produces a LSM with a variable layer spacing which results in the structure in the wings of the diffraction profile. Samples with large 2d spacings made by this process yield excellent peak reflectivities. A recent test of samples with small 2d spacings produced by evaporation did not show the high reflectivities that were expected based upon the large 2d spacing results. LSMs produced by sputtering yield very symmetric diffraction profiles with little structure in the wings. The peak reflectivities in these samples are very good, but typically are lower than those of evaporated samples with similar menumbers of layerpairs and large 2d spacing. (We must note that we have never compared two identical structures made with these two techniques). Sputtering has been very successful at producing high reflectivity in small 2d spacing samples. These are general conclusions based upon our limited experience with sputtered and evaporated multilayers.

CONCLUSIONS

Layered synthetic microstructures can be used as high reflectivity x-ray coatings. These coatings are stable under laboratory conditions for a long period of time. Idealized models can be used to quantitatively predict their performance. More accurate results can be obtained if the effects of nonideal behavior such as roughness and layer thickness variations are included. A synchrotron allows one to measure the performance of these structures at arbitrary soft x-ray energies and make direct comparisons to theory.

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FIGURES

1. A schematic of the sample chamber used in all of the experiments described in this paper. Sixteen samples may be placed on the sample wheel at a time inside the ultra high vacuum chamber.

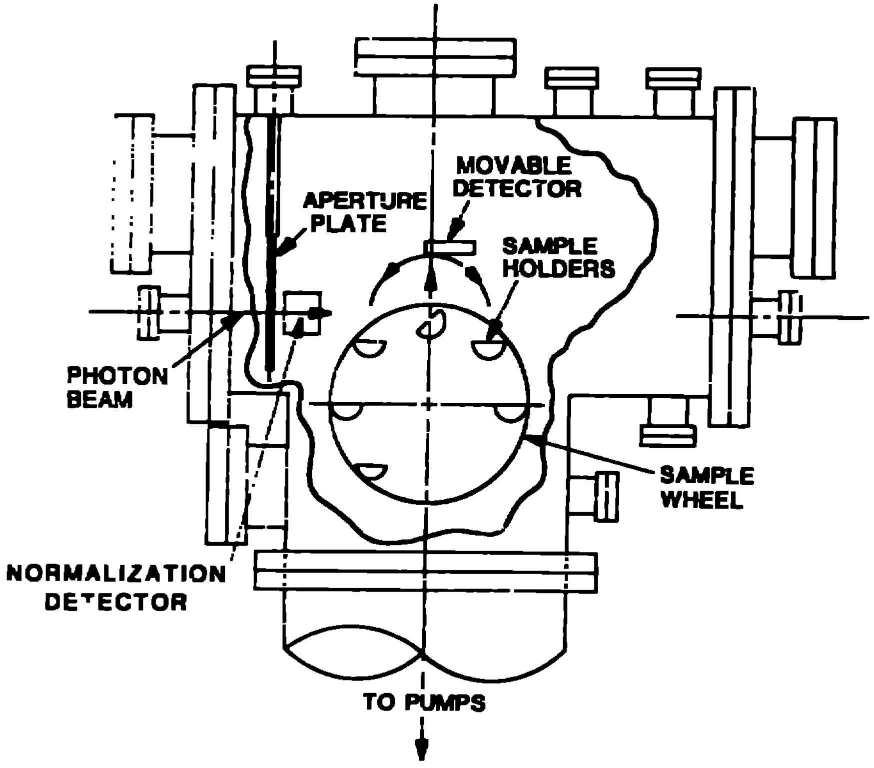
2. A diffraction profile for a 16 layer pair Ni-C multilayer with a 2d spacing of 143A. The .... are the experimental points which are normalized to a theoretical calculation (solid line) of the reflectivity using regular lattice and the optical constants indicated in the figure. The peak experimental reflectivity of 15 percent has been matched to the theoretical peak reflectivity of 25 percent.

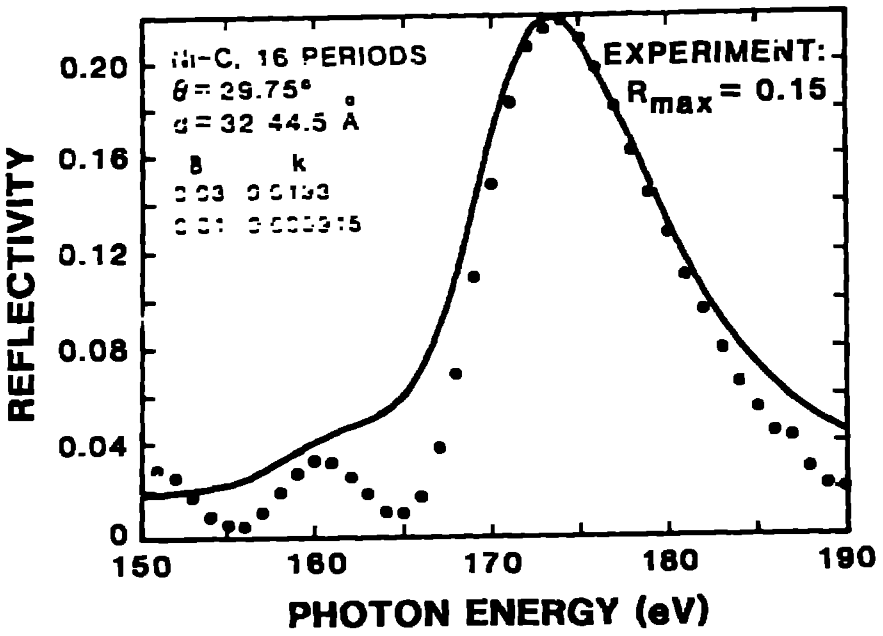
3. Peak first order reflectivity vs. energy for a variety of transition metal-carbon multilayer systems. The microstructures had approximately 17 layerpairs with a 2d spacing of 140A.

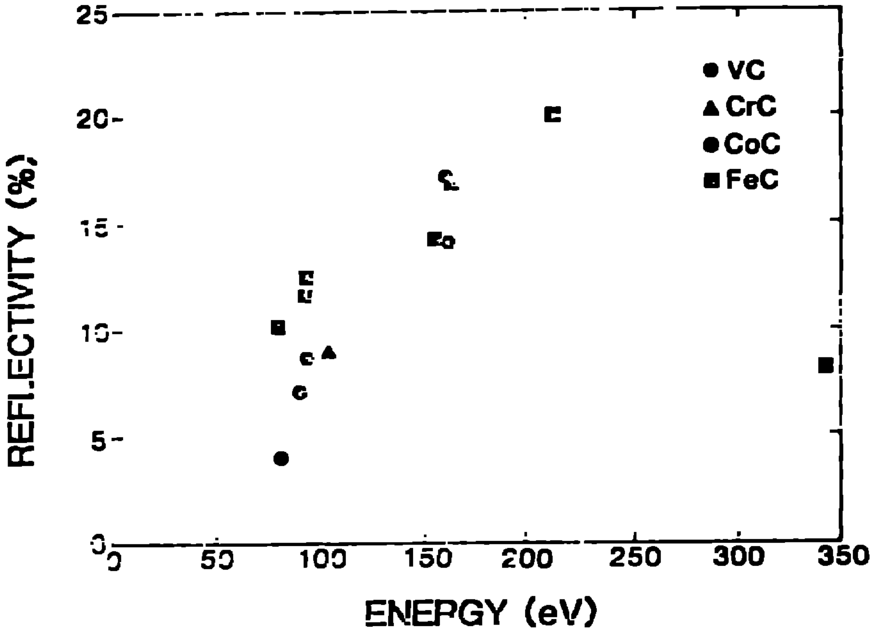
4. The reflectivity vs. energy for three harmonics of a ReW-C multilayer with a 2d spacing of 175A at a fixed angle.

5. The effect of p-polarized light on the reflectivity of a LSM near Brewster's angle (45 degrees) in the soft x-ray regime.

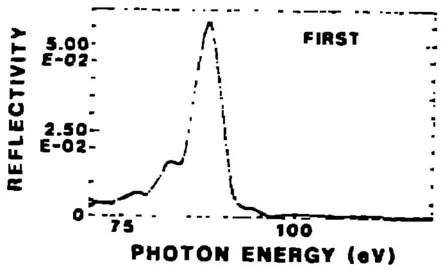
6. Measured reflectivity vs. energy for a soft x-ray Fabry-Perot etalon which exhibits a two slit type diffraction pattern modulated by the reflectivity of the LSMs in the structure.

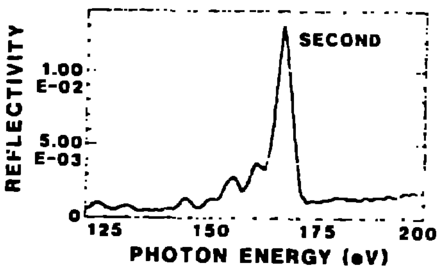

MOVABLE DETECTOR
APERTURE PLATE
SAMPLE HOLDERS
PHOTON BEAM
SAMPLE WHEEL
NORMALIZATION DETECTOR
TO PUMPS

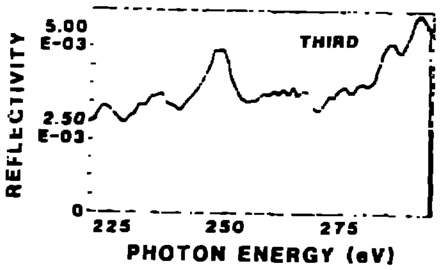

Ni-C, 16 PERIODS
θ = 29.75°
d = 32 44.5 Å
δ k
0.03 0.0193
0.01 0.000315
EXPERIMENT:
R_{max} = 0.15
REFLECTIVITY
0
0.04
0.08
0.12
0.16
0.20
150
160
170
180
190
PHOTON ENERGY (eV)



REFLECTIVITY (%)
25
20
15
10
5
0
0
50
100
150
200
250
300
350
ENERGY (eV)
● VC
▲ CrC
● CoC
■ FeC



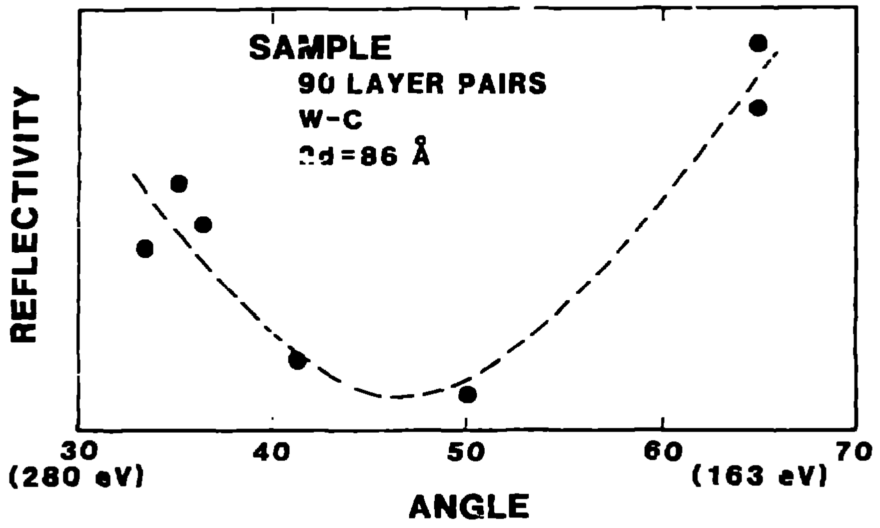


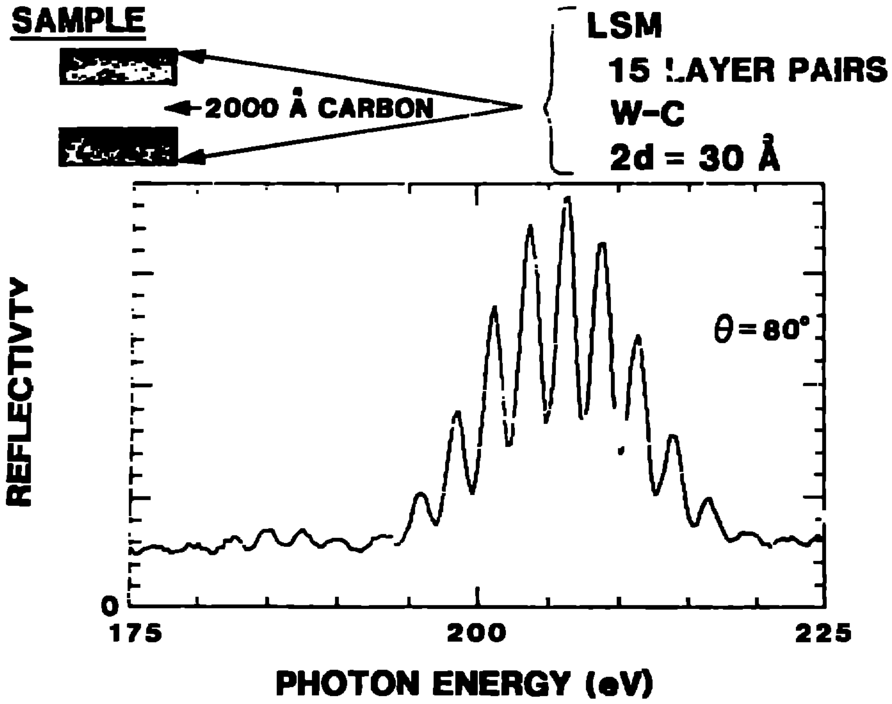


SAMPLE

17 LAYER PAIRS
ReW-C
2d = 175 Å

P-POLARIZATION




SAMPLE
2000 Å CARBON
LSM
15 LAYER PAIRS
W-C
2d = 30 Å
θ = 80°
REFLECTIVTY
0
175
200
225
PHOTON ENERGY (eV)