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X-Ray Performance of Multilayer Diffraction Diagnostics

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X-Ray Spectroscopy is an established diagnostic for the Nuclear Test Program. The high diffraction response of multilayer structures can make important contributions as X-ray diffraction analyzers for UG testing. The soft X-ray performance of a number of commercially deposited multilayers was determined in our laboratory and compared with diffraction theory predictions.					
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X-RAY PERFORMANCE OF MULTILAYER DIFFRACTION DIAGNOSTICS

I. INTRODUCTION

The use of x-ray spectroscopy as a diagnostic tool for scientific and industrial applications has increased appreciably this decade. There is also potential application in the Nuclear Weapons Testing Program for x-ray diagnostics. The heart of the x-ray spectroscopic technique is the diagnostic element that must satisfy the Bragg relationship for x-ray diffraction. Nature provides only a limited number of good crystals that are suitable for use as efficient diffraction elements. The Naval Research Laboratory (NRL) has had a history of acquiring and evaluating the x-ray performance of Bragg diffraction elements, either for planar or curved optics (1). In the early 60's convex-curved crystals were utilized in compact spectrograph's (2) suitable for acquiring x-ray spectra in UG tests. An outgrowth of this work was increased emphasis on an extensive program for laboratory testing of natural and grown crystals (3-5) at NRL and Langmuir-Blodgett films at Pomona College (6,7) for soft x-ray analysis and field applications. Bragg crystals grown from acid phthalates were found to have good x-ray performance for low energy x-ray spectrochemical and field application. In recent years synthetic crystals have been produced by the deposition of alternating layers of high and low-atomic number (Z) elements with selectable interatomic spacing. These synthetic multilayer structures (MLS) have exhibited excellent diffraction efficiencies, generally an order of magnitude better than natural or grown crystals (8-10). However, for many uses other factors in addition to high diffraction efficiency, such as the diffraction uniformity and the diffracted linewidths, are important performance parameters for successful UG diagnostics tool.

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In this work, we evaluated the soft x-ray performance of a number of different types of multilayers chosen to manufacture from the predicted diffraction properties. The x-ray characteristics of multilayer structures have been evaluated using both single- and double- crystal spectrometers. Multilayer specimens were prepared by vacuum deposition on a variety of substrates whose surface roughness varied from supersmooth (1-2 Å) to deliberately roughened surfaces (formed by microetching silicon wafers).

The x-ray performance was evaluated by comparing first order and higher order diffraction efficiencies, rocking curve widths, and the variation in diffraction characteristics over the surface of the specimens. The MLS's were measured in the 8-27 Å region at discrete x-ray characteristic wavelengths, 8.3 Å (Al K α) and 9.9 Å (Mg K α) in single-crystal spectrometers using fluorescers, and 8.3 Å, 17.6 Å (Fe L α) and 27.4 Å (Ti L α) in a double-crystal instrument with the anode of a Henke tube as the x-ray source. Classical diffraction theory for multilayers predicts nearly equal integral reflection coefficients (R-values) for W/C and W/Si multilayers structures having nearly the same bi-layer thickness with a nearly equivalent tungsten layer fraction. Such multilayers were prepared on pairs of equal roughness substrate for x-ray evaluation. The surface roughness of both the substrates and multilayers was measured by optical profilometry. The multilayer surface roughness for some samples was also determined by scanning tunneling microscopy, STM, and by atomic force microscopy, AFM, which have lateral spatial resolution more commensurate with the x-ray diffraction correlation length.

For low-Z multilayers, theory predicts lower diffraction efficiency but better energy resolution. Because more layer pairs are involved in the diffraction process, a beneficial result would be an improvement in rocking curve

width and potentially a higher heat load capacity under high x-ray flux. Multilayers of selected medium or low-Z elemental pairs Ni/Si, $\text{Si}_3\text{N}_4/\text{Si}$ and C/B C were manufactured for evaluation. As predicted, the diffraction efficiencies are smaller for the low-Z multilayers, but are of the same order of magnitude as acid phthalate crystals.

II. BACKGROUND

The x-ray performance of crystals can be evaluated from the line intensities, linewidths, and the integral of intensity under the diffraction curve obtained by scanning through the Bragg peak in an x-ray spectrometer. The relation between the peak intensity, P , the linewidth, W , and the integral diffraction coefficient, R , are illustrated in Fig. 1. The diffraction uniformity over the entire specimen is important for achieving spectral resolution, particularly in field applications. The x-ray topography was determined for selected multilayers.

Both single x-ray crystal spectrometers, SXTLS, (Fig. 2) and a double x-ray crystal spectrometer, DXTLS, (Fig. 3) were used to evaluate the multilayers. The SXTLS's at Ovonic Company and at NRL used fluorescers and the DXTLS at NRL had Al, Fe, or Ti anodes. The instrumentation used at NRL has been described previously (11). X-ray topographs were acquired in the DXTLS by placing a small aperture between the monochromator crystal and the specimen being analyzed.

The multilayer specimens were deposited by sputtering onto flat substrates. Substrates varying from polished Si wafers to fused quartz and superpolished Zerodur were used. Multilayers were deposited onto Si wafer substrates and cleaved to rectangular sections 3.2×7.6 cm that could be accommodated by the crystal holder of commercial spectrographs. Prior to deposition, substrate roughness was evaluated with noncontact scanning profilometers capable of

determining roughness to 1-2 Å. Multilayers of W/C and W/Si were produced as pairs on similar substrates. The multilayer surface was remeasured after deposition. In general the multilayers surface had about the same surface roughness as the substrate, except it was noted that for depositions made on the supersmooth quartz and Zerodur substrates that the multilayer surfaces were slightly smoother than their respective substrates.

The substrate roughness was thought to be an important factor in the MLS performance. As noted in previous work, classical transport model predictions of the integrated diffraction intensity are 5-10 times higher than measured values for W/C multilayers (12). By analyzing higher order diffraction data, the differences observed between the measured and predicted R-values were correlated to the substrate roughness determined by optical profilometry (13). The object of this current work was to quantify the correlation. Profilometer results gave a variety of surface roughness for the current substrates and multilayers from 1 Å to 22 Å RMS.

Natural or grown crystals with the largest interplanar spacings are acid phthalate crystals with 2d-spacings of about 26 Å. These crystals are limited to x-ray measurements below about 20 Å because of the nonuniform response at the oxygen K edge (23.2 Å). Acid phthalates with potassium, rubidium, or thallium cations have moderate efficiencies for soft x-ray diffraction. KAP Crystals have nearly a uniform diffraction response between 8-20 Å with R-values less than 0.1 mr. KAP crystals have unique properties for curved-crystal spectrographs and have found utility in the DNA x-ray simulation program (14) (15).

In the 60's, Burt Henke's laboratory began making and evaluating large interatomic synthetic crystals, by the Langmuir-Blodgett (LB) dipping process (6). They have moderate diffraction efficiency and moderate resolving power.

The LB crystals have 2d-spacings only at specific values limited to 70 Å (Pb laurate), 80 Å (Pb myristate), 100 Å (Pb stearate), 130 Å (Pb lignocerate) and 160 Å (Pb melissate). Therefore, large-interatomic spacing synthetic crystals can be made by the Langmuir-Blodgett process; however, the possible 2d spacings are limited. They have good diffraction efficiency but only moderate resolving power.

Multilayers fulfill requirements for 2d-spacings between 26 and 70 Å for soft x-ray experiments. Multilayer structures (MLS) formed by deposition have been manufactured with continuous 2d spacing as small as about 30 Å. MLS's fulfill the requirement of good collection efficiency for soft x-ray experiments.

In this work, multilayers were prepared with 2d-spacings of about 60 Å from a variety of different components on different types of substrates. With this 2d-spacing, the multilayers are usable as soft x-ray diffraction elements in the 0.25-1.5 keV (50-8 Å) region.

Preliminary to the choice of multilayers to be manufactured, x-ray reflectivities were predicted theoretically for many alternating pairs of both high-Z/low-Z and low-Z/low-Z components. Fig. 4 shows results for multilayers with 2d-spacings (thickness of two bi-layers) of about 60 Å. Calculations were performed for W/Si and W/C multilayers having the same W thicknesses equal to about 25% of the bilayer structure. In selecting elements one must take into account the x-ray absorption edges. Tungsten and carbon have edges about 7 and 44 Å corresponding to the tungsten M edges and the carbon K edge respectively, while for silicon the K-edge is 6.7 Å. The W/Si multilayer is seen to have a smooth and nearly constant R-value response between 10-55 Å. Generally, near the absorption edges, the integral reflection drops sharply. Further at large Bragg angles, the R-values increase as the Bragg diffraction wavelength

approaches the d-spacing limit. The predicted R-values for the high-Z/low-Z multilayers (C and W/Si) are an order of magnitude larger than for acid phthalate crystals.

Nickel and silicon were chosen to form a medium-Z/low-Z elemental multilayer. Nickel has a L-edge at 14.6 Å that results in a dip in the response curve; however, at wavelengths greater than about 17 Å the calculated R-value is smooth and greater than 0.5 milliradians.

One low-Z/low-Z combination of interest is silicon/silicon nitride (Si/Si₃N₄). The predicted response is found to have a slight dip at the nitrogen K-edge at 31 Å but is relatively smooth from 10-60 Å with R-values above 0.1 mr.

Several elemental and compound pairs were examined as good low-Z multilayer candidates. Two such pairs were silicon/carbon and carbon/boron nitride. Both were found to have low, nonuniform diffraction response curves. Another candidate considered for a low-Z/low-Z multilayer is carbon/boron carbide C/B₄C. The predicted response of this multilayer was constant and lower than that of Si/Si₃N₄ below 30 Å, but has a increased diffraction efficiency above 30 Å. The predicted R-values of C/B₄C are comparable to potassium acid phthalate crystals below 26 Å.

III. RESULTS

A. X-ray performance of high-Z multilayers

The R-values were measured over a large surface area of the multilayer specimens in the single crystal spectrometers. The 2d spacings were determined using Mg K α radiation. The double crystal spectrometer was used both as a broad source for the determination of the overall integral diffraction coefficients and, with an aperture, the variation over the surface. The monochromator was a W/C multilayer whose rocking curve had been determined in previous work. With

a small aperture the x-ray performance was determined at selected positions along the multilayer surface. Up to 6 measurements were made on the 7.6 cm long specimens.

The R-values and line widths measured for a number of W/C and W/Si multilayers are given in Table 1. For the single crystal results at 9.9 Å, the R-values for the W/C multilayers on Si wafers vary from 0.56-0.67 mr while the W/Si multilayers on Si substrates have R-values of 0.83-1.06 mr. The measured 2d spacings of these MLS's were 51.8-55.0 Å. R-values were calculated for similar atomic spacing multilayers. Values predicted for W/C with a 2d spacing of 51.3 Å and a W layer thickness equal to 28% of the interatomic spacing were 1.6 mr at 8.3 Å and 2.1 mr at 17.6 Å. The R-values predicted for W/Si multilayers were 1.5 and 1.92 mr at 8.3 and 17.6 Å, respectively for a W thickness 23% of the d-spacing. In summary, the linewidths and R-values were essentially the same magnitude for the W/Si multilayers deposited on silicon substrates whose optically-determined surface roughness varied from 5-22 Å RMS.

The R-values were measured at 17.6 Å across the specimen surface in the double crystal spectrometer. The R-values were 0.8-1.0 mr for the W/Si specimens compared to values of 0.45-0.65 mr for the W/C multilayers. In most cases, the W/Si multilayers had a uniform diffraction response and only one R-value is listed. An exception was the M015B-1 specimen (W/Si) whose R-value varied from 0.83-1.0 mr. The polished Si wafer used as the substrate for this multilayer had a large surface roughness of 22 Å. The R-values from the multilayers deposited on supersmooth quartz and Zerodur substrates were noticeable lower than the W/Si multilayers on silicon substrates.

The linewidth measured in the double crystal spectrometer were between 0.5-0.6 degrees for W/Si and 0.5 to 1.3 degrees for W/C; however, the W/Si

multilayers were uniform for most of the specimens measured while the W/C multilayers were nonuniform. For the multilayers on the quartz substrates, the line widths were about the same for the W/C and W/Si multilayers, different than on silicon substrates. Both had uniform line widths and R-values. The resolving power of the multilayers varied from 20-100.

The X-ray topography for high-Z multilayers on silicon substrates is shown in Fig. 5. The diffraction line profiles for the W/Si were about equivalent across the specimen surface. For the W/C multilayer, the linewidths were nonuniform. The narrower linewidths generally correspond to about the center of the specimen. Diffraction peaks for multilayers on quartz substrates are shown in Fig. 6. The R-values for W/C were about 2/3 of the those for W/Si; however, the linewidths were about equal. The uniformity of the W/C on quartz was better than 10% as was the W/Si specimen; but, the R-values for the W/Si multilayers were about 1/3 lower than for W/Si on silicon substrates.

B. X-ray performance of low-Z multilayers

Medium-Z and low-Z multilayers were evaluated in both spectrometers. The specimens were manufactured with nominal 2d spacings of 60 Å, except for a Si/Si₃N₄ multilayer where thin (2 Å) carbon depositions were made at the interfaces. This was intended to improve the diffraction efficiency by limiting possible interface diffusion or chemical reactions during the deposition process. The results of the x-ray measurements are given in Table II for Ni/Si, Si/Si₃N₄, and C/B₄C multilayers on various substrates. In general, lower R-values were observed than predicted by about an order of magnitude. Some slight improvement in line resolution was achieved with the specimens on substrates with low surface roughness (polished silica). It is noted that having low-Z interface boundaries

(2 Å of carbon) in the Si/Si₃N₄ did not lead to an improvement in the x-ray performance. Diffraction peaks for the multilayers are shown in Fig. 7.

Multilayers of C/B₄C low-Z components have been deposited on silicon wafer substrates and are presently under evaluation.

C. Predicted multilayer intensities

The theoretical integral reflection coefficients for high-Z and low-Z multilayers are presented in Tables III and IV, respectively, for three orders of Bragg diffraction. From a comparison with the measurements at 17.6 Å in the double-crystal spectrometer, the experimental first diffraction order R-values for the W/C multilayers are about a factor of four lower than predicted and for W/Si are a factor of two lower than predicted by diffraction theory. This data would indicate that the W/Si multilayers have a smoother surface than the W/C multilayers.

D. Surface roughness measurements

Preliminary data has been obtained for the high-Z multilayers by a scanning tunneling microscope (STM) and by an atomic force microscope (AFM). Sections of the SP15-2 W/C multilayer were scanned by the STM. The upper carbon layer of the bilayer structure had characteristic rows of steep ridges. The surface roughness of one of the sections was 12-15 Å RMS. STM images were not obtainable from the W/Si multilayer because of the surface oxidation of the upper silicon layer.

Linear scans were performed by AFM for sections of the SP15-3 W/Si multilayer. The cantilever force that yielded intensity data showed a smooth surface with peak-to-valley variations of 2-3 Å.

CONCLUSIONS

- ☐ W/Si multilayers have higher R-values than W/C multilayers.
- ☐ W/Si multilayers have significantly narrower rocking curves than W/C multilayers.
- ☐ R-values and rocking curve widths were uniform across the surface of W/Si samples.
- ☐ R-values were uniform across the surface for W/C samples.
- ☐ Rocking curves widths varied with position for W/C multilayers.
- ☐ Soft x ray diffraction properties did not correlate with optical measurements of substrate roughness.

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Table I. X-ray performance of high-Z (tungsten) multilayers.

SPECIMEN/ SUBSTRATE	MULTI- LAYER TYPE	DOUBLE-CRYSTAL MEASUREMENTS					SINGLE-CRYSTAL MEASUREMENTS			
		ROUGHNESS		Fe L			Mg K			
				W(meas)	dE/E	R-Value	2-D	dE/E	R-Value	
		Substrate (Å)	Surface (Å)	Deg. Theta	(%)	(m.)	(Å)	(%)	(m.)	
SP10-2/ silicon	W/C	4.8	5.6	0.53	1.14	0.53	54.40	5.28	0.63	*
				0.80	3.10	0.53				*
				1.03	4.40	0.45				*
										*
SP10-4/ silicon	W/Si	5.5	5.4	0.59	1.71	0.91	51.80	3.32	0.97	*
										*
										*
										*
SP15-2/ silicon	W/C	6.0	6.0	0.57	1.53	0.65	54.80	5.71	0.56	*
				1.01	4.28	0.55				*
				0.85	3.41	0.62				*
				0.48	0.56	0.58				*
SP15-3/ silicon	W/Si	5.6	5.8	0.55	1.35	0.88	52.80	5.57	0.83	*
				0.59	1.71	0.84				*
				0.65	2.17	0.87				*
				0.59	1.71	0.86				*
MO15G-3/ silicon	W/C	8.4	11.7	1.32	5.91	0.46	54.40	5.81	0.67	*
				0.95	3.94	0.56				*
				0.85	3.41	0.55				*
				0.69	2.45	0.56				*
MO15G-1/ silicon	W/Si	5.7	10.1	0.54	1.24	0.90	54.00	3.42	1.02	*
										*
										*
										*
MO15B-4/ silicon	W/C	19.0	22.0	0.89	3.62	0.51	54.20		0.65	*
				1.10	4.77	0.33				*
				0.97	4.07	0.41				*
				0.87	3.51	0.45				*
MO15B-1/ silicon	W/Si	22.0	22.3	0.91	3.72	0.47	55.00	3.43	1.06	*
				1.14	4.98	0.41				*
				0.57	1.53	0.93				*
				0.56	1.44	0.83				*
VOCQ-2/ quartz	W/C	2.9	1.8	0.55	1.35	0.44				*
				0.59	1.71	0.41				*
				0.62	1.95	0.48				*
				0.59	1.71	0.41				*
VOFS-2/ quartz	W/Si	2.9	1.8	0.57	1.53	0.66				*
				0.62	1.95	0.62				*
				0.59	1.71	0.63				*
										*
GOR-1/ zerodur	W/Si	1.9	1.4	0.61	1.83	0.46	54.60	3.72	0.48	*

Table II. X-ray performance of medium- and low-Z multilayers.

SPECIMEN/ SUBSTRATE	MULTI- LAYER TYPE	SINGLE-CRYSTAL SPECTROGRAPH MEASUREMENTS (MeV K)				
		ROUGHNESS		2-D (Å)	dE/E (%)	R-Value (mr)
		Substrate (Å)	Surface (Å)			
GO-2/ silica	Ni/Si	2.0	2.0	53.00	5.39	0.100
MO15-3/ silicon	Ni/Si	13.4	8.7	53.20	6.71	0.081
GO-4/ silica	Si/Si ₃ N ₄	2.1	2.1	63.60	4.05	0.005
SP-1/ silicon	Si/Si ₃ N ₄	2.8	3.6	65.80	4.25	0.029
MO-2/ silicon	Si/Si ₃ N ₄	9.1	8.1	67.60	4.30	0.037
MO15-5/ silicon	Si/C/Si ₃ N ₄ /C	3.2	7.7	64.40	5.11	0.012

Table III. Predicted soft x-ray reflectivities for high-Z multilayers at 17.6 Å.

Components	Layer composition (percent)	R-value (mr)		
		First	Second	Third order
W/C	28:72	2.07	0.349	0.222
W/Si	23:77	1.92	0.432	0.289

Table IV. Predicted soft x-ray reflectivities for low-Z multilayers at 17.6 Å.

Components	Layer composition (percent)	R-value (mr)		
		First	Second	Third order
C/B ₄ C	25:75	0.026	0.004	0.002
C/B ₄ C	50:50	0.048	2×10^{-4}	0.004
C/B ₄ C	75:25	0.022	0.004	0.002
Si/Si ₃ N ₄	50:50	0.356	0.000	0.017
Si/C/Si ₃ N ₄ /C	39:11:39:11	0.422	0.116	0.119

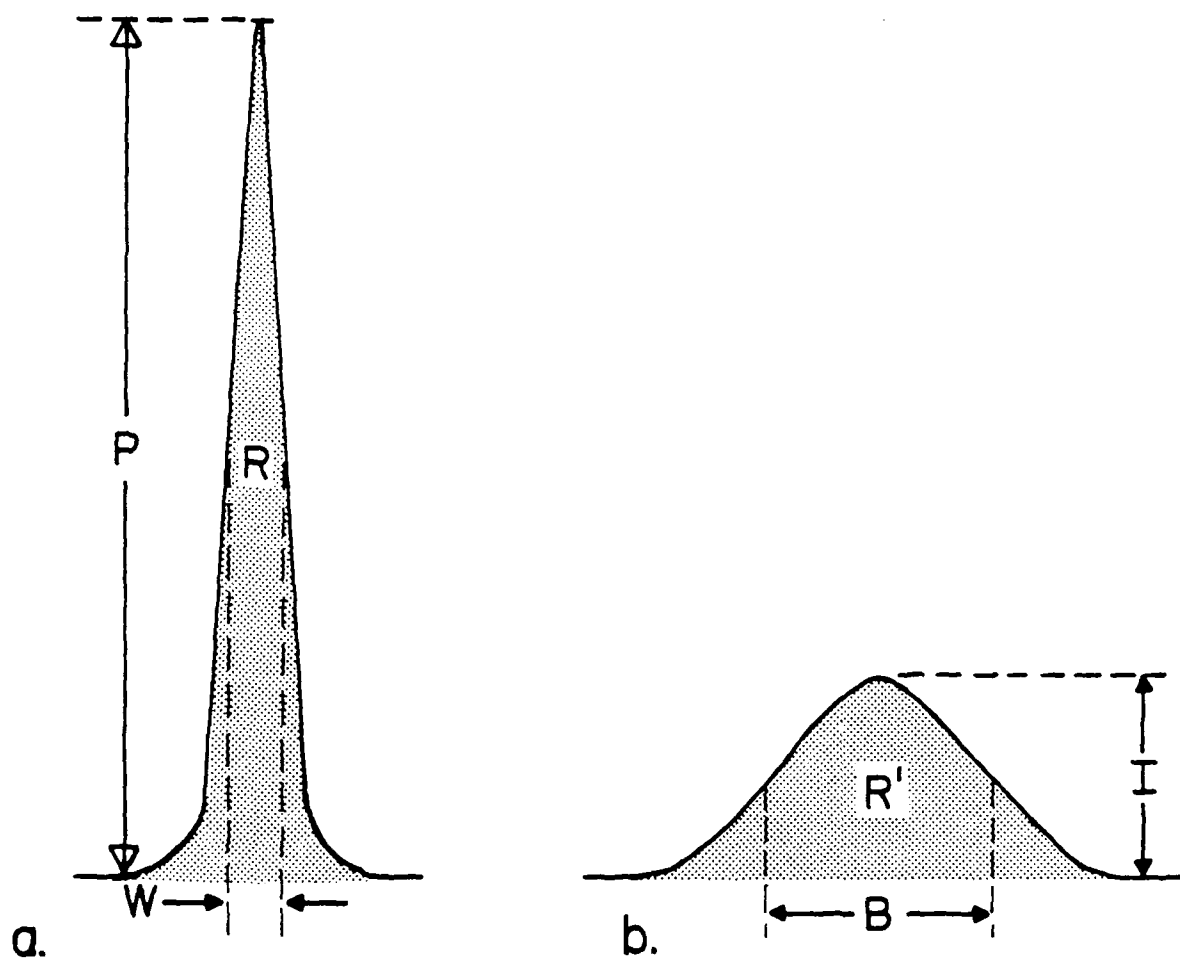


Fig. 1 — Bragg-diffracted x-ray line characteristics: a. Diffraction rocking curve of a crystal: P is the peak diffraction coefficient, W is the full width at half-maximum of the curve, and R is the integral reflection coefficient. b. Line shape from part a as it would be measured in a typical single crystal spectrometer: line intensity (I) is not equal to P , linewidth (B) is not equal to W , but R' exactly equal to R .

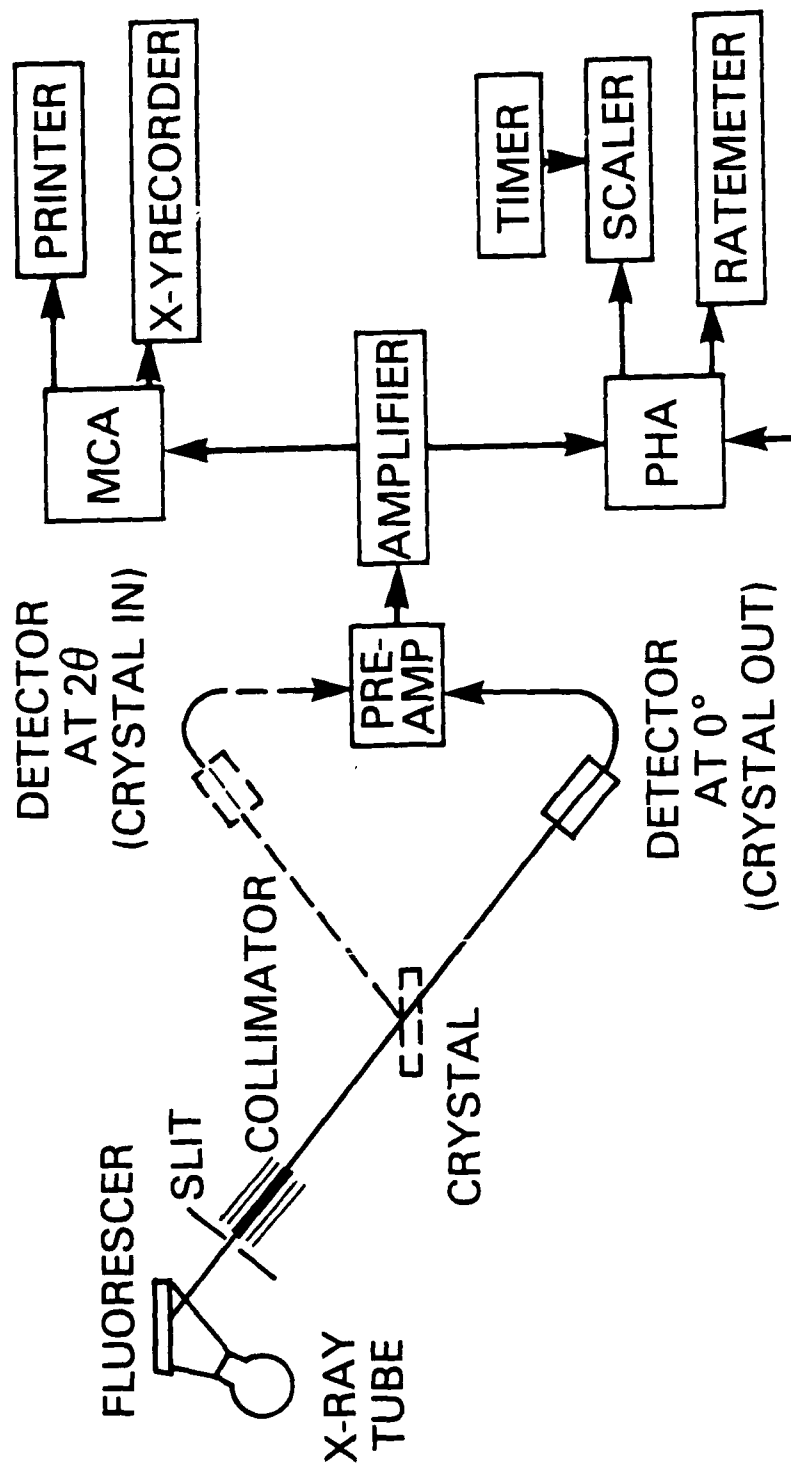


Fig. 2 — Schematic of single x-ray crystal spectrometer (SXTLS)

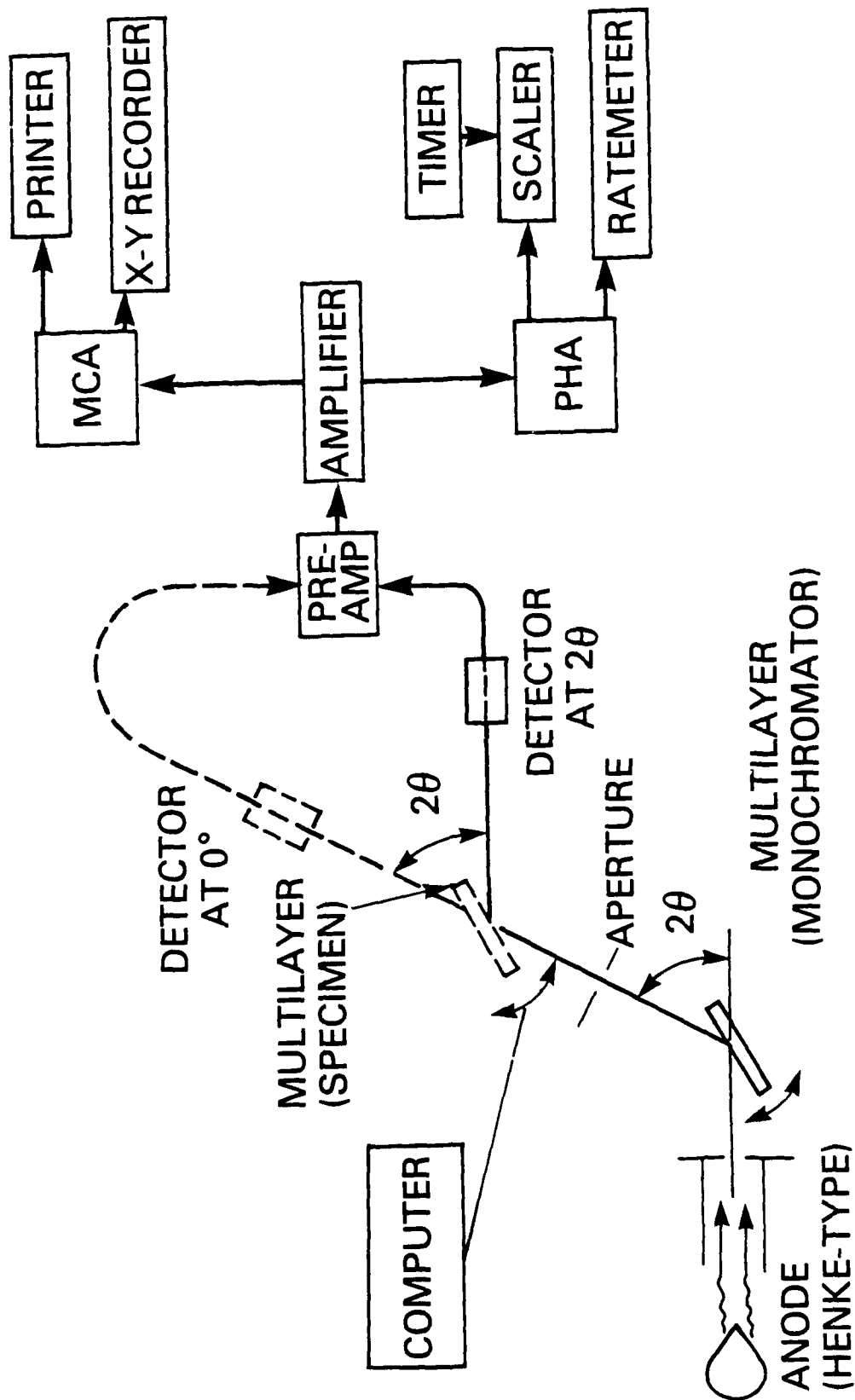


Fig. 3 — Schematic of double x-ray crystal spectrometer (SXTLS)

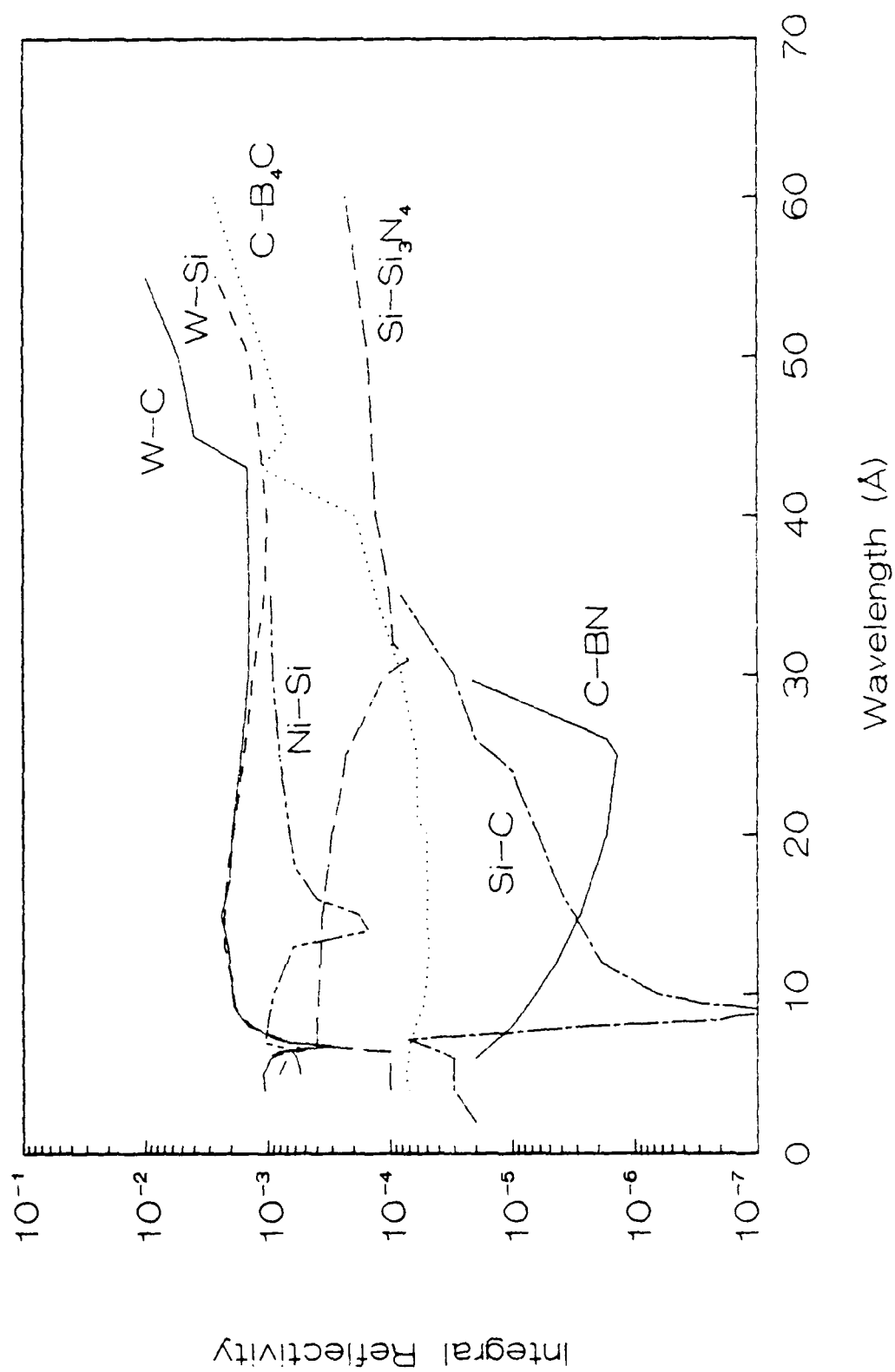


Fig. 4 --- Soft x-ray integral reflection coefficient, R-values, calculated by a diffraction model code

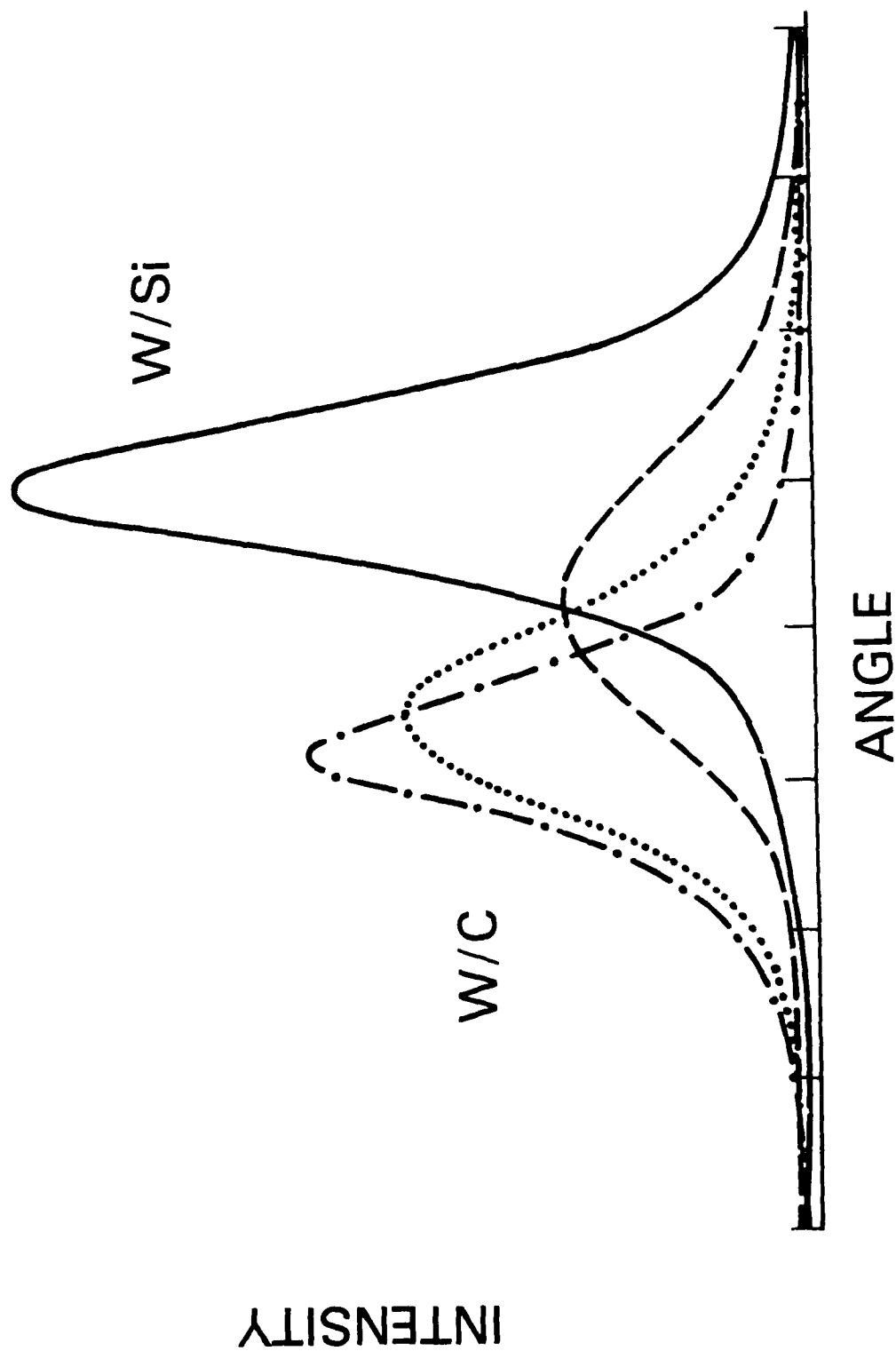


Fig. 5 — Diffracted line profiles for high-Z multilayers on silicon substrates.
The three lower intensity curves are topographic measurements across the specimen surface.

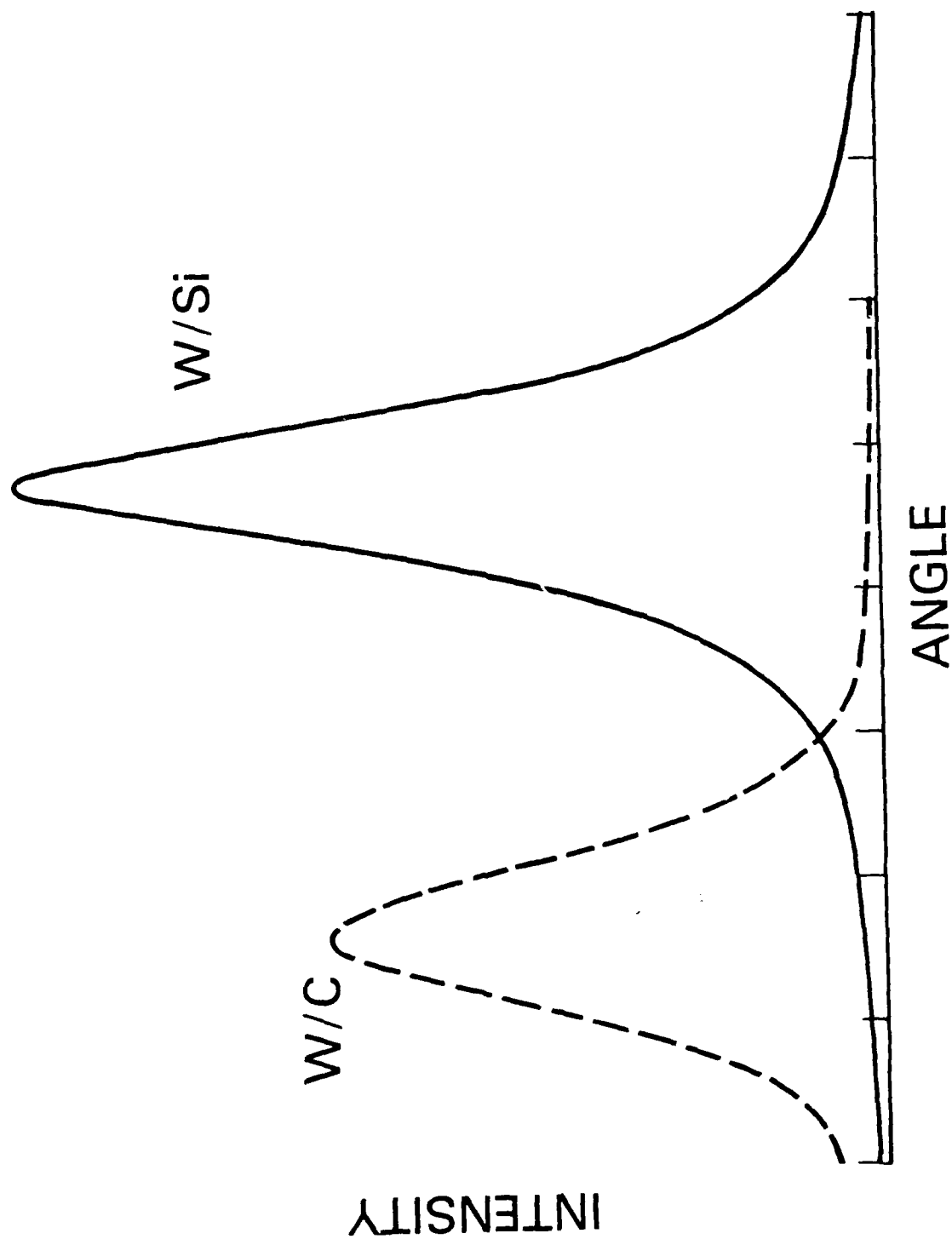


Fig. 6 -- Diffracted line profiles for high-Z multilayers on quartz substrates

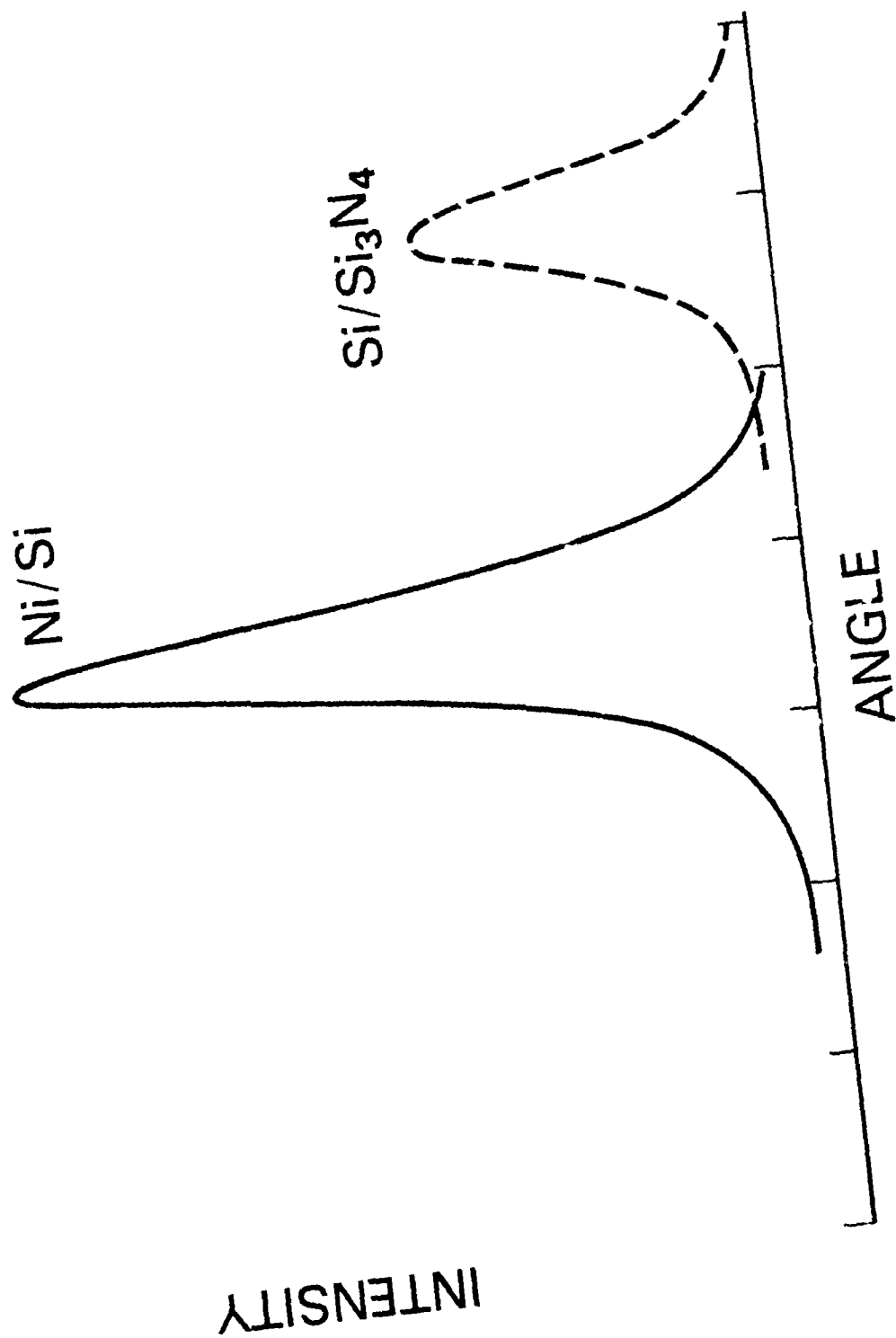


Fig. 7 — Bragg-diffracted profiles from medium- and low- Z multilayers