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X-Ray Reflectivity of a 200 Layer $W_{.25}Si_{.75}$ Multilayer Crystal

J. Als-Nielsen and F. Grey

Risø National Laboratory, DK-4000 Roskilde, Denmark
January 1990

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CH = Switzerland
DD = German Democratic Republic
XE = Commission of the European Communities (CEC)
XC = European Organization for Nuclear Research (CERN)
NL = The Netherlands
DK = Denmark
SE = Sweden

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X-Ray Reflectivity of a 200 Layer $W_{.25}Si_{.75}$ Multilayer Crystal

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ABSTRACT

X-ray reflectivity studies of a synthetic 200 layer $W_{.25}Si_{.75}$ crystal on a Si wafer with a d-spacing of 25 Å yielded the following results:

At a fixed wavelength of 1.54 Å the reflectivity for grazing incidence below a critical angle of 5.1 mrad exceeds 70%. The peak reflectivity of the first order Bragg reflection at the Bragg angle of 31 mrad is 78% of the totally reflected beam, and the relative band width of the peak is 2.1%. Both these numbers are in good agreement with the simplest form of dynamical scattering theory as outlined in the text.

The reflectivity of higher harmonics is less than a few per cent, with the fundamental set for reflection at 1.54 Å. Crystals of this quality are very useful optical elements in synchrotron radiation instrumentation, in particular when adequate cooling methods are fully developed.

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1. INTRODUCTION

The reflectivity of a multilayer[1] crystal of $W_{.25}Si_{.75}$ with d = 25 Å has been studied both at a fixed wavelength of 1.54 Å in the angular range from 0.3 mrad to 72 mrad and at a fixed grazing angle of 31 mrad for wavelengths between 2.5 and 0.5 Å.

The set up is shown schematically in fig. 1. Distances, slit dimensions, precision of 2θ and ω and the translation stage t are given in the appendix.

The fixed wavelength around the Cu K-α doublet was selected by a discriminator window in the MCA spectrum from the Ge detector. Although the relative bandpass of this window is 9% the intensity is dominated by the K-α doublet with an intrinsic splitting of about 0.25% which is ten times smaller than the relative bandpass of the multilayer crystal as we shall see.

With the low energy part of the spectrum from the rotating anode attenuated by a 0.5mm Al foil the spectra of the direct beam and of the beam reflected from the crystal at a glancing angle of 31 mrad were also compared. More details and results are given in the following section.

In section 3 we compare the results to the simplest possible dynamical diffraction theory (the Darwin-Prins formalism) and we find very good agreement between the observed peak reflectivity and the relative band pass for the fundamental reflection, whereas the higher harmonics experimentally are considerably suppressed compared to the idealized model, presumably due to imperfections in the precise sequence of layer thickness.

2.RESULTS

2.1 Fixed Wavelength Cu-K-α = 1.54 Å

The anode was operated at 20 kV, 10 mAmp.

The direct beam intensity was measured at $2\theta = 0$ and $\omega = 0$ with the sample translated out of the beam.

The sample was oriented and centered at $2\theta = 0$ by intercepting the direct beam to 50% of full intensity and optimizing ω in an iterative procedure. The estimated accuracy from the data is 0.02° on ω and 0.01 mm on the translation by this method. With the sample centered, the (0,0,1) peak was optimized in 2θ and ω and the computer value of ω changed to exactly half of 2θ. The accuracy in the setting of ω is then 0.004 °.

With the sample aligned in this way the wavevector transfer perpendicular to the surface, Q_z, was scanned from 0.01 to 2.10 in units of the Bragg reflection occurring at $Q_z = 2\pi/d$, d being the lattice spacing 25 Å. Results are shown in fig.2 with the reflectivity on logarithmic scale. The insets show the fundamental (0,0,1) and the second harmonic (0,0,2) reflections on expanded linear scales.

The rocking curve (ω-scan) of the (0,0,1) and (0,0,2) peaks had a FWHM of 0.041°.

We note that the peak height of the fundamental reflection is 78% of the maximum of the totally reflected intensity and the widths of both peaks are around 0.02 in units of the (0,0,1) reciprocal lattice vector.

2.2 Energy Dispersive Spectrum at Fixed Glancing Angle

With the crystal oriented to (0,0,1) Bragg reflection for Cu-K-α we registered the reflected spectrum as well as the direct beam spectrum. In order to attenuate the dominating K-α peak in the spectrum and also not to saturate the detector we inserted a 0.5mm Al foil in the direct beam from the anode operating at 30 kV for these measurements. The reflected beam spectrum divided by the direct beam spectrum is shown vs. energy in fig. 3. By comparing the integrated intensities of the (0,0,1), (0,0,2) and (0,0,3) peaks we conclude that the reflectivity for second and third harmonic is 1.6% and 0.07%, respectively with the crystal set to reflect a fundamental wavelength of 1.54 Å.

Note the distinct interference phenomenon in the wings of the fundamental Bragg reflection between the Bragg reflected beam and the totally reflected beam: constructive interference on the low Q-side and destructive interference on the high Q-side. This is similar to the observations in surface induced smectic layering in liquid crystals[2].

3.THEORETICAL MODEL.

3.1 Reflectivity of one W layer imbedded in Si

The *amplitude* of the specular reflected wave from a thin layer, relative to the amplitude of the incident wave, must be a dimensionless complex number, r. It must be proportional to the scattering amplitude for one electron, the Thomson scattering length $r_o = 0.282 \times 10^{-4}$ Å. It must also be proportional to the number of electrons/unit area perpendicular to the incident beam, i.e. to $\rho_{el}\ \delta z/\sin(\theta)$ where δz is the layer thickness and θ the grazing angle. That exhausts the dependence on sample parameters. In order to get a dimensionless ratio it must also be proportional to the wavelength λ, i.e.

$$r = c\ \rho_{el}\ r_o\ \{\lambda/\sin(\theta)\}\delta z \qquad (1)$$

The complex number c turns out to be $i = \sqrt{-1}$, c.f. see Warren[3] eq. 14.4. From Bragg's law the factor $\lambda/\sin(\theta) = (2/m)d$ with $m = 1$ for first order, $m = 2$ for second order etc. The tungsten layer is assumed to be 25% of the lattice spacing d, so δz is d/4, or more general xd for crystal composition W_xSi_{1-x}. However, we must take into account the finite thickness of the W layer. The ray reflected from the top of the layer is not exactly in phase with the ray reflected from the bottom of the layer. Near Bragg reflection the phase difference is $2m\pi$ for a distance d, i.e. $2m\pi x$ for the thickness of the W layer. For $x = .25$ the scattering power is reduced by the average of $\cos\phi(\phi)$ between $-\pi/4$ and $+\pi/4$ for first order, and between $-\pi/2$ and $\pi/2$ for second order, i.e. by a factor 0.9 and 0.64, respectively. We call this reduction factor f (for form factor) and get, in the notation used by Warren:

$$r = iq = i\ \rho_{el}\ r_0\ d^2(2x/m)\ f \qquad (2)$$

The electron density is of course the contrast density between W and Si:

$$\rho_{el} = \{N_A\ Z/A\ \rho_{mass}\}_W - \{N_A\ Z/A\ \rho_{mass}\}_{Si} \qquad (3)$$

N_A being Avogadro's number, and Z and A atomic number and weight.

It is also of interest to calculate how much the wave is attenuated from one tungsten layer to the next. The path length is $d/\sin(\theta)$, so the intensity is

attenuated by μ d/sin(θ) and the amplitude is attenuated by half of that. In Warrens notation the attenuation per layer is denoted h:

$$h = \mu d/(2\sin(\theta)) \tag{4}$$

In evaluating h we must take the sum of h for W and Si using distances d/4 and 3/4d respectively.

Inserting numbers, we find:

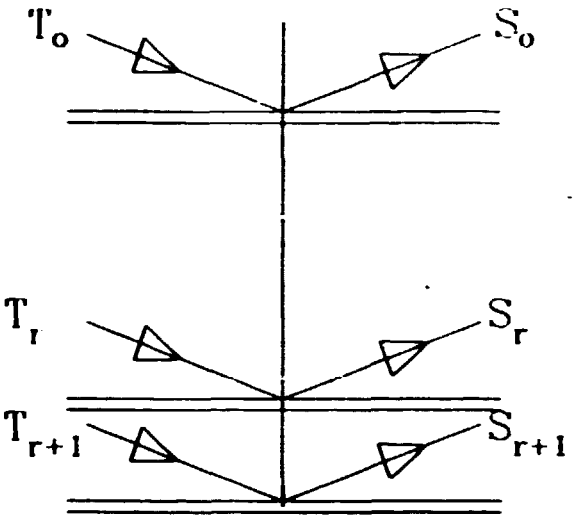
	q	h
1st order	3.2×10^{-2}	0.39×10^{-2}
2nd order	1.1×10^{-2}	0.19×10^{-2}

These simple considerations are quite instructive for order of magnitude estimates. Since q is around 3% for first order we should not expect more than about the first 33 layers to be "active" in first order reflection which must have a relative bandpass of the order of q, whereas the top 100 layers are "active"' in reflecting second order. Bearing in mind that in addition to 3 times more layers, the phasing for a certain depth must be twice as accurate for 2 nd order, it may not be surprising that we find a much lower 2 nd order reflectivity than predicted by idealized models. That is *not* necessarily a drawback of the crystal, it may actually be an advantage that it produces a monochromatic beam with very little higher order contamination.

It is also clear from the table above that scattering dominates over absorption at the considered wavelength 1.54 Å.

3.2 Darwins Formulation of Dynamical Scattering

We follow Warren's exposition in chapter 14, and consider an incident wave T_r and the specular reflected wave S_r from layer r, and the corresponding quantities from layer (r + 1). Sr is composed of specular reflection of T_r (i.e. $T_r(iq)$) and the wave S_{r+1} from the layer below, properly attenuated and phased by the factor $(1-h+iq)\exp\{-i\phi\}$.



$$\phi = 2\pi d\sin\theta = \pi(Q/\tau) = \pi(m+\zeta)$$

$$S_r = T_r(iq) + (1-h+iq)S_{r+1}e^{-i\phi}$$

$$T_{r+1} = (1-h+iq)T_r e^{-i\phi} + iq\,S_{r+1}e^{-i2\phi}$$

Trial: $T_r = T_o(1-\eta)^r \;,\; \eta \ll 1$

Solution: $\eta = (q^2 + (h+iv)^2)^{\frac{1}{2}}$ with $v = n\zeta$

$$\frac{S_o}{T_o} = \frac{iq}{(h+iv) \pm \eta} = \begin{cases} \dfrac{ig}{h + (q^2-h^2)^{\frac{1}{2}}} \xrightarrow{q \gg h} i(1-h/q) \text{ for } v = o \\ = \dfrac{iq}{iv \pm (q^2-v^2)^{\frac{1}{2}}} \rightarrow \text{FWHM}(\zeta) = \dfrac{(3/\sqrt{2})q}{n} \end{cases}$$

Inserting numbers for first order reflection we find:

Peak reflectivity = $(1-h/q)^2 = 78\%$
FWHM in $Q_z/\tau = 0.0216$

These two numbers are without any adjustable parameters in excellent agreement with the experimental findings.

However, with the same model applied to 2nd order we find an enormous difference. A very likely reason for the discrepancy was outlined in the end of the previous paragraph.

4. DISCUSSION

The present crystal quality is sufficient for substantial improvement of existing synchrotron radiation experiments.

4.1 Liquid Surface Spectrometry[1]

As an example we take the present liquid surface spectrometer at HASYLAB at beam line D4, i.e. unfocused 1 mrad bending magnet radiation 22 m from the source. In the experiments a monochromatic beam is deflected downwards at an angle α towards the liquid surface. A natural unit for α is the critical angle α_c for total reflection from the liquid surface and accordingly we define the dimensionless quantity $q = \alpha/\alpha_c$. Three kinds of experiments are performed:

(i) Specular reflectivity (XR) yielding information about the average density profile across the liquid-air interface e.g. the molecular conformation in a Langmuir film of amphipilic molecules on a water substrate. Typical q-range from 0.5 to 30

(ii) Grazing incidence fluorescence (GIF) to examine the depth variation of certain atomic species by their fluorescence yield as the evanescent wave penetration depth is varied. Typical q-range from 0.5 to 3.

(iii) Grazing incidence diffraction (GID) where the lateral structure is examined by the diffraction pattern in the horizontal plane using the evanescent beam as the incident beam. Typically q fixed around 0.8.

In the present set up monochromatization is obtained by Bragg reflection from a Ge(111) crystal giving a band width of 0.025%. The beam deflection is obtained by tilting the monochromator crystal[2]. In practise the beam width on the sample is limited to 5 mm compared to the the width of 22 mm of the 1 mrad S.R. beam 22 m from the source. The height of the beam incident on the sample is limited by the maximal footprint on the liquid surface, typically 50 mm. For $q = 0.8$ and $\lambda = 1.54$ Å this implies a beam height of only 0.1 mm, compared to the S.R. beam height of 2.5 mm. It is therefore obvious that a considerable improvement may be obtained by using focusing as well as a broader band pass.

4.2 Focusing Double Monochromator with Multilayer Crystals

We suggest the use of a double crystal monochromator with multilayer crystals as described in this report. The lattice spacing of the two crystals should be slightly different, e.g. 24 and 25 Å, to provide an exit beam direction 2 mrad downwards

from horizontal corresponding to q = 0.8. This beam can then be used directly in GID. The double monochromator should provide focusing in the horizontal as well as in the vertical plane by sagital and tangential bending of the crystals. A 1:4 demagnification of the source can be obtained by locating the double monochromator around 17 m from the source. The first crystal could be cooled (power load is 30 W from a DORIS Bending Magnet) and mounted with a tunable tangential radius of curvature in the hundreds of meter range. The second crystal could provide the sagital focusing with radius of curvature around 50 cm. With this kind of focusing the full beam width can be utilized and 1/5 of the beam height for the 0.1 mm beam. In comparison with present conditions it means that the gain obtained by focusing is around 25 times.

In searching for 2-dimensional powder Bragg peaks from a mono-molecular film in a Langmuir trough a resolution of 2% may actually be adequate. The gain from the band pass is thus 80, or altogether a potential gain in signal of 2000 times! The signal to background ratio is by and large determined by the sample, not by the source or by the parameters of the instrument. In a typical favourable case the ratio is 1:1. Presently the count rate is so low that peaks with a ratio less than 0.2:1 are not detectable. However, with a thousandfold increase in intensity the statistical uncertainty of the background level could be reduced to 0.1% within reasonable counting times, so signals which are only 1% of the background level should be detectable. The present GID patterns contain only 1-3 Bragg peaks from which all the lateral structure information must be derived. Clearly a data set with say 10 Bragg peaks would be much more informative.

In cases where a more narrow bandpass is needed one can just insert a standard double monochromator of Ge (111) before the sample with, of course, the corresponding sacrifice in intensity.

Let us now turn to XR and GIF with the suggested monochromator. The remaining problem is the beam deflection.

4.3 Beam Deflector

We suggest a beam deflector consisting of a multilayer crystal followed by a conventional, flat gold coated mirror as shown in fig. 4. The two components

contribute to q by the amounts of q_{ml} and q_{mi}, respectively, and these are discussed in turns.

The multilayer crystal deflects the beam by a *fixed* amount $2\theta_B \simeq \lambda/d$ or in terms of q by $q_{ml} = \lambda/d/\alpha_c$. The critical angle $\alpha_c = \lambda (r_o \rho_{el}/\pi)^{\frac{1}{2}}$. For water $\rho_{el} = 0.33$ electrons per Å^3, so $\alpha c = \lambda(Å) \times 1.72$ mrad, or $q_{ml} = 581/d(Å)$.

The mirror deflects the beam by $2\theta_{mi}$. The mirror has a sharp upper cut-off at the critical angle which is larger than α_c by the square root of the ratio of electron densities of the gold layer and water, i.e. by approx. 3.6 times. This means that the maximal value of q_{mi} is 7.2. The minimal value is set by the footprint on the mirror. Since the mirror is a few times longer than the sample and therefore has a correspondingly longer allowable footprint we assert q_{mi} as *continuously variable* between 2 and 7.2.

In order to cover the full q-range of 0.5 to 30 required for XR one obviously needs a set of d values for the multilayer crystal. We suggest a *striped* multilayer crystal. The first stripe could be a 100 Å thick W layer. When this stripe is inserted in the beam the multilayer crystal acts like a mirror which can deflect the beam either downwards or upwards corresponding to $4 < q_{ml} < 6$ or $-4 > q_{ml} > -6$, respectively. The second stripe has d = 64 Å or $q_{ml} = 9$. The third stripe has d = 32 Å or $q_{ml} = 18$ and the fourth stripe has d = 23 Å or $q_{ml} = 23$. For a chosen stripe of the multilayer crystal the q-range is swept continuously by turning the mirror from $(q_{ml} + 2)$ to $(q_{ml} + 7.2)$.

Beam monitors may be inserted between the multilayer crystal and mirror and after the mirror to ensure optimal alignment.

5. ACKNOWLEDGMENT

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J. Als-Nielsen in "Physicochemical Hydrodynamics" p. 639 (Ed. M.G. Verlarde) Plenum Publ. Corp. 1988

7. FIGURE CAPTIONS

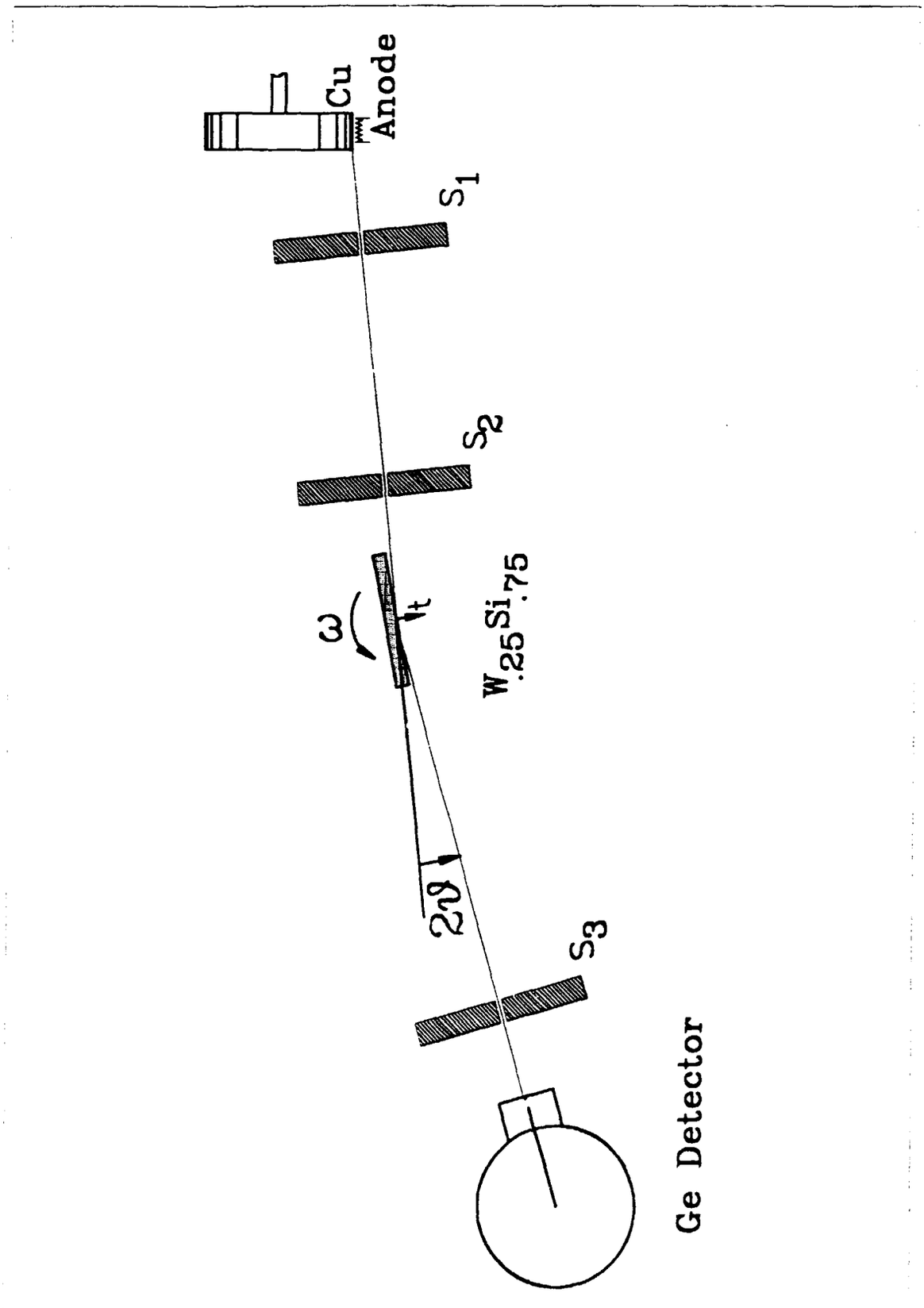
Fig.1 Schematic set up. Note that the X-ray path is defined by slits only, and the detected wavelength is selected in the Ge detector.

Fig.2 Angular scan of the specular reflected beam for a wavelength of 1.54 Å, the Cu-K-α doublet. Insets: Detailed line profiles of the (0,0,1) and the (0,0,2) reflections. Intensities are normalized to 100 at the maximum of the total reflected beam. The fundamental Bragg reflection reaches 78% of the total reflected beam.

Fig.3 Ratio between the energy spectra of the reflected and the direct beam at a fixed glancing angle of 31 mrad. Both spectra were measured with a 0.5 mm Al attenuator. The reflectivity of the 2nd (3rd) harmonic is less than 2% (0.1%) of the

fundamental at this fixed glancing angle. Intensities are normalized to 100 at the (0,0,1) reflection.

Fig.4 Schematic beam deflector using a multilayer crystal followed by a conventional, flat Au plated mirror. The multilayer crystal can function as a mirror as well and can also reflect the beam upwards.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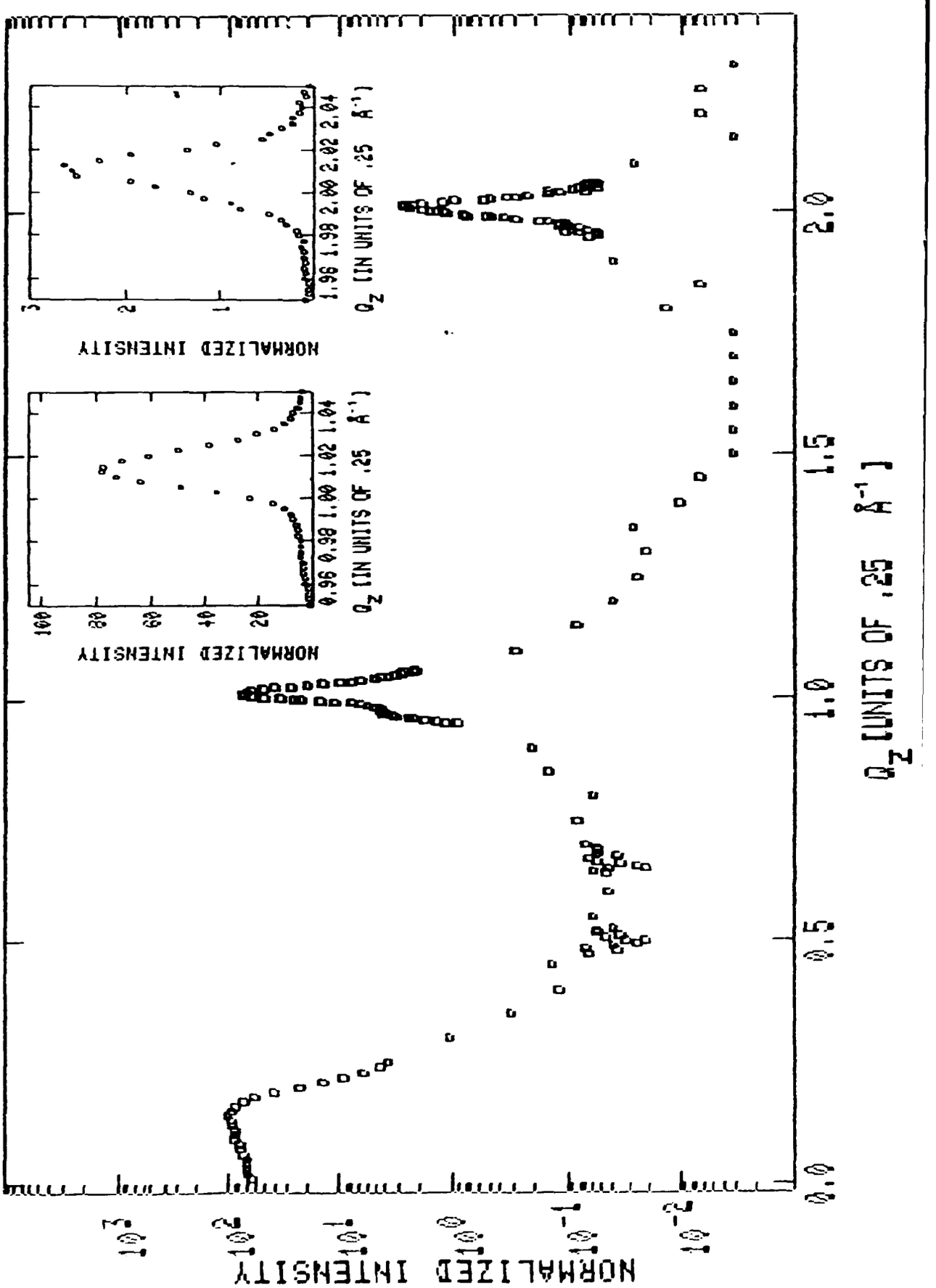
Anode
S_1
S_2
S_3
$W_{.25}Si_{.75}$
t
ω
2θ
Ge Detector


NORMALIZED INTENSITY
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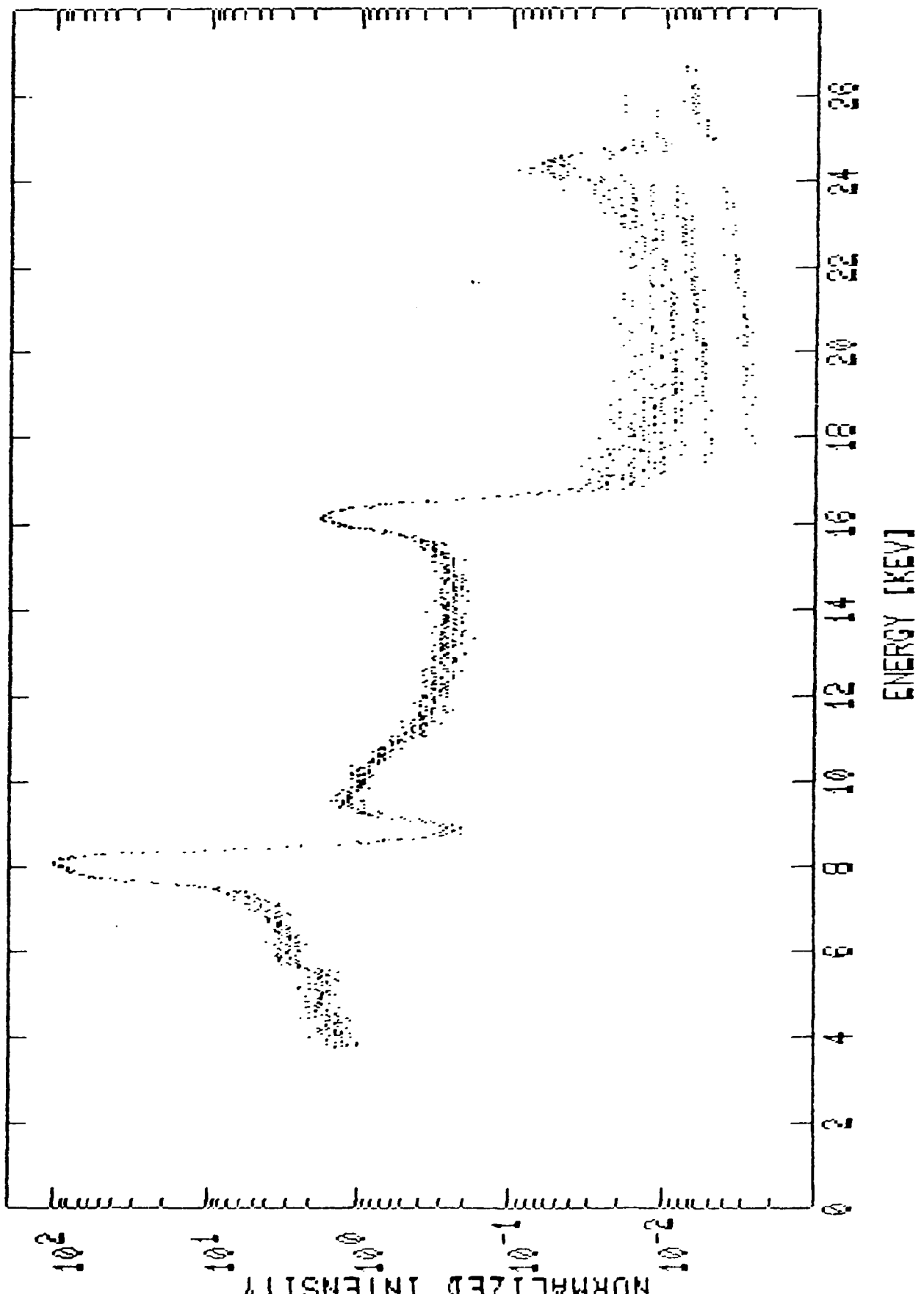
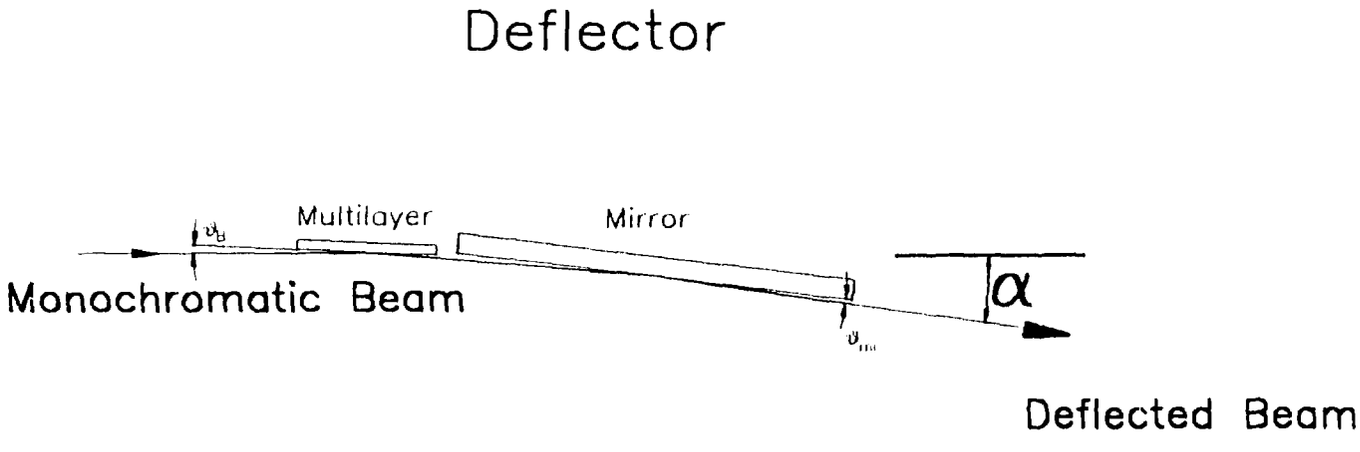
10^{-2}
10^{-1}
10^{0}
10^{-1}
10^{-2}
0.0
0.5
1.0
1.5
2.0
Q_z [UNITS OF .25 Å^{-1}]
NORMALIZED INTENSITY
100
80
60
40
20
0.96 0.98 1.00 1.02 1.04
Q_z [IN UNITS OF .25 Å^{-1}]
NORMALIZED INTENSITY
3
2
1
1.96 1.98 2.00 2.02 2.04
Q_z [IN UNITS OF .25 Å^{-1}]


NORMALIZED INTENSITY
ENERGY [KEV]


Deflector
Multilayer
Mirror
Monochromatic Beam
α
Deflected Beam

APPENDIX

Technical Description of Set Up

1. Distances from source and slits (width × height in m m):

Source: 0 (1.0×0.5)

S1: 140 (0.1 × 10)

S2: 710 (0.1 × 1)

Sample: 790 (70 × 35)

S3: 1190 (2 × 10)

2. Motors:

M4: Sample translation perpendicular to beam 400 steps/mm

M1: ω - Sample rotation, 2000 steps/degree

M5: 2θ - Detector arm rotation, 2000 steps/degree

3. Detector:

Solid State Ge Detector with MCA (NUCLEUS in P.C.)

Cu-K-α channels # : 628 peak

600 and 653 for low and high channel integration limits. In program TAS use following input:

CHL1 = 600 ; CHU1 = 653 ; LCHA = 599; UCHA = 654;

In Subroutine XMSC one has substituted INT1 for I in XSC

Settings:

H.V. = -1000 V.

Canberra 2020: 1.5 usec. Fine Gain 6.25 Coarse Gain 1k

Input Polarity: +, Restorer: AUTO

Unipolar Output to MCA

4. Sample

Ovonic Synthetic Materials Company, Inc.

W-Si, 200 layers, 25% W, XRO #2467-10

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