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AN IN-SITU X-RAY DIFFRACTION METHOD FOR THE STRUCTURE OF AMORPHOUS THIN FILMS USING SHALLOW ANGLES OF INCIDENCE

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A technique for the structural characterization of thin amorphous films employing synchrotron radiation parallel beam X-ray optics at grazing angles of incidence is detailed. At incident angles near to the critical angle for total external reflection, sampling of specimens may be achieved via the evanescent mode.

The parallel beam geometry allows the use of a technique in which a 2θ detector , incorporating a parallel plate collimator , scans diffraction data for a given incident angle. For a specified wavelength , the incident angle chosen will determine the penetration of the radiation into the sample (~ 10 - 1000Å). The data must be corrected for significant peak shifting resulting from X-ray refraction, as well as for the effects assosciated with conventional θ:2θ scans.

Preliminary data resulting from the first application of this technique to amorphous hydrogenated silicon:carbon thin films, deposited onto crystalline silicon substrates, will be presented and discussed. Conventional θ:2θ powder diffraction data will also be presented as a comparative standard .

INTRODUCTION

Amorphous thin films have shown themselves to be of contemporary interest for the wide range of properties they display, many of which have potential technological applications. These properties are defined by the microscopic structure of the material and are determined by the parameters involved in the production process. X-ray diffraction, incorporating a technique which involves shallow angles of incidence, may be used for in-situ structural characterization of amorphous thin films deposited onto crystalline substrates. The central characteristics of the glancing angle technique, in relation to the study of crystalline iron oxide layers on glass substrates, have been detailed by Lim et al [1].

The refractive index of materials at X-ray wavelengths is less than unity, consequently at incident angles below a critical value total external reflection of the X-rays occurs. At such incident angles limited penetration of the material is achieved via the evanescent mode. Thus a study of the as-deposited material eliminating the effects of substrate scattering is, in principle, possible. For the samples concerned the critical angle is of the order of 0.2°. The intrinsically highly parallel nature of the beam provided by a synchrotron radiation source such as that at the SRS, Daresbury Laboratory, U.K. is of advantage over focussed laboratory X-ray sources for this technique in that geometric aberration effects are greatly reduced. Further, the high intensity beam provided is suitable for the relatively weak scattering from the small volume of material sampled.

Penetration into the sample increases with the incident angle and decreases with the wavelength of the radiation. Thus for a given wavelength a number of sampling depths may be achieved by varying the incident angle. Below the critical angle for total external reflection penetration of the X-rays normal to the sample surface is exponentially damped and, in principle, sampling depths of the order of 10Å to 1000Å may be achieved.

Above the critical angle penetration increases rapidly with incident angle and is limited by photoelectric absorption.

INSTRUMENTATION

The experiments detailed were carried out on station 9.1 at the SRS. The parallel beam produced by the SRS allows the use of a technique in which a 2θ detector, uncoupled from the sample axis, scans the diffracted intensity for a given fixed wavelength and incident angle. The 2θ detector scans in a vertical plane, the sample being offset from the horizontal by the incident angle.

A schematic diagram of the instrumentation used is given in Figure 1. The incident X-ray wavelength is selected from the white beam spectrum using a water cooled, channel cut silicon (111) monochromator, CM [2]. A silicon powder standard (S.R.M. 640b) was used to determine the wavelength and the zero angle of the diffractometer detector, D. The incident monochromatic beam intensity is constantly monitored during the experiment using scattering from an inclined kapton foil in the beam (KF). The footprint, l, of the radiation on the sample is determined by the width of the entrance slit, S2, where $l = S2/\sin\alpha$, α being the incident angle. For grazing angles of incidence S2 is in general required to be small (1~ μm) to restrict the incident beam to the sample surface and avoid scattering from the sample mounting. Thus a high intensity X-ray source is required to ensure that a suitable flux is incident on the sample surface.

The diffracted beam is collimated by a set of 330mm long horizontal parallel slits, HS, giving a 2θ resolution of approximately 0.07^{o}. This is neccessary for the large area of sample irradiated to avoid significant smearing effects. Divergence off axis is similarly limited by a set of parallel vertical slits, VS.

The increased intensity in the wavelength range of interest to this experiment (~ 0.6Å) produced by the wiggler magnet on

line 9 at the SRS enables viable count rates to be achieved for the relatively low scattered intensity involved. The use of the hard X-rays available on this line is of advantage in the study of amorphous materials as it provides access to an extended wavevector transfer range and thus increased real space resolution from the X-ray diffraction data.

For glancing angles of incidence the zero angle, θ_0, determined for the sample mounted on the diffractometer is clearly important. In determining this angle an ion chamber is used in place of the detector and slits. Having determined the ion chamber reading without the sample in the beam, the diffractometer sample stage, set at the nominal diffractometer zero angle, was raised normally into the beam until the sample in the beam reduced the ion chamber reading by a half. The sample was then rocked about the θ axis to either side of the nominal θ_0 and a transmission curve taken at the ion chamber. The zero angle of the diffractometer was then reset to the angle at the centre of the peak in the transmission curve. Several iterative transmision curves were taken until a sharp peak occured in the transmission at the zero angle of the sample. The accuracy of this value is eventually limited by any curvature of the sample (- curvature occurs as many thin films are in compressive stress).

THEORY

The critical angle for total external reflection of the X-rays by the sample, α_c, is given by [1, 3]

$$\alpha_c = (2\delta)^{1/2} = 1.6 \times 10^{-3} \rho\lambda$$

where $\delta = n-1 = 1.3 \times 10^{-6} \rho \lambda^2$, n being the real refractive index of the material. ρ is the material density in gcm^{-3} and λ the incident wavelength in Angstroms.

Below the critical angle the penetration depth of the X-rays into the sample, t, is related to α_c and the incident angle α as follows :

$$t = \lambda / [2\pi (\alpha_c{}^2 - \alpha^2)^{1/2}]$$

At or above the critical angle t is given as :

$$t = \mu / \sin \alpha$$

where μ is the linear absorption coefficient.

In the case of diffraction data collected at glancing angles of incidence peak shifting resulting from X-ray refraction by the material may become significant. As the refractive index of materials for X-rays is less than unity a positive shift in the peak positions $\Delta 2\theta$ occurs. This shift is given by

$$\Delta 2\theta = 2\delta / [\sin (2\theta_{obs} - \Delta 2\theta)] + \delta / \alpha$$

where $2\theta_{obs}$ is the observed scattering angle. The scattering data must also be corrected for the effects assosciated with conventional θ:2θ scans such as geometrical, background and absorption factors [4], and Compton scattering [5] to obtain the coherent scattering relating to the structure of the material.

RESULTS

Preliminary glancing angle diffraction data is presented for a thin film of amorphous hydrogenated silicon:carbon (a-Si:C:H) deposited onto a silicon substrate. The sample, of contemporary technological interest in the photovoltaics industry, was produced using a glow discharge technique from a silane and hydrocarbon precursor gas mixture diluted with hydrogen. The thickness of the sample was approximately 1μm.

Conventional θ:2θ powder diffraction data is also presented. The atomic composition of the powder was found to be $Si_{0.7}C_{0.1}H_{0.2}$ from UPS and Rutherford Backscattering studies.

Figure 2a. shows the structure factor for a thin film of a-Si:C:H as determined by glancing angle X-ray diffraction. The diffraction data was taken at a wavelength of 0.6Å and an incident angle of 0.1° . This is below the critical angle for the sample at this wavelength. The accuracy of the calculated critical angle is limited by the density value given to the thin film; from the known value of the bulk density of a powder sample a lower limit may, however, be assigned to the film density , the density of a thin film always being greater than that of a corresponding powder sample. Thus, a lower limit may be assigned to the critical angle of the film.

Figure 2b. shows the structure factor for the powder sample of a-Si:C:H obtained from a conventional θ:2θ diffraction geometry experiment. The experiment was carried out using an incident X-ray wavelength of 0.6Å.

In each the case the initial diffraction data has been normalized to the incident intensity and standard corrections applied. The refraction shift for the thin film sample is 0.05°, and thus below the resolution of the data collected.

A significant degree of correlation may be seen between the structure factors in figures 2a. and 2b. The sharp peaks in the X-ray diffraction data for the thin film in fig. 2a. at Q = 10Å^{-1}, 16Å^{-1} may be attributed to residual contributions from the silicon substrate.

Figures 3a. and 3b. give the corresponding pair distribution factors for the thin film and powder samples respectively. Features may be seen in the g(r) for the thin film corresponding to the C-C sp^3 bond length (1.54Å), the Si-Si bond length (2.35Å), and Si-Si second shell (3.82Å). The latter two features are also apparent in the g(r) for the powder sample.

CONCLUSIONS

The use of a SRS for X-ray diffraction at glancing angles of incidence has been shown as a viable technique for the analysis of thin amorphous films on crystalline substrates. Futher work remains to be done on the data , but significant correlations with data collected by conventional θ:2θ powder diffraction data has been shown.

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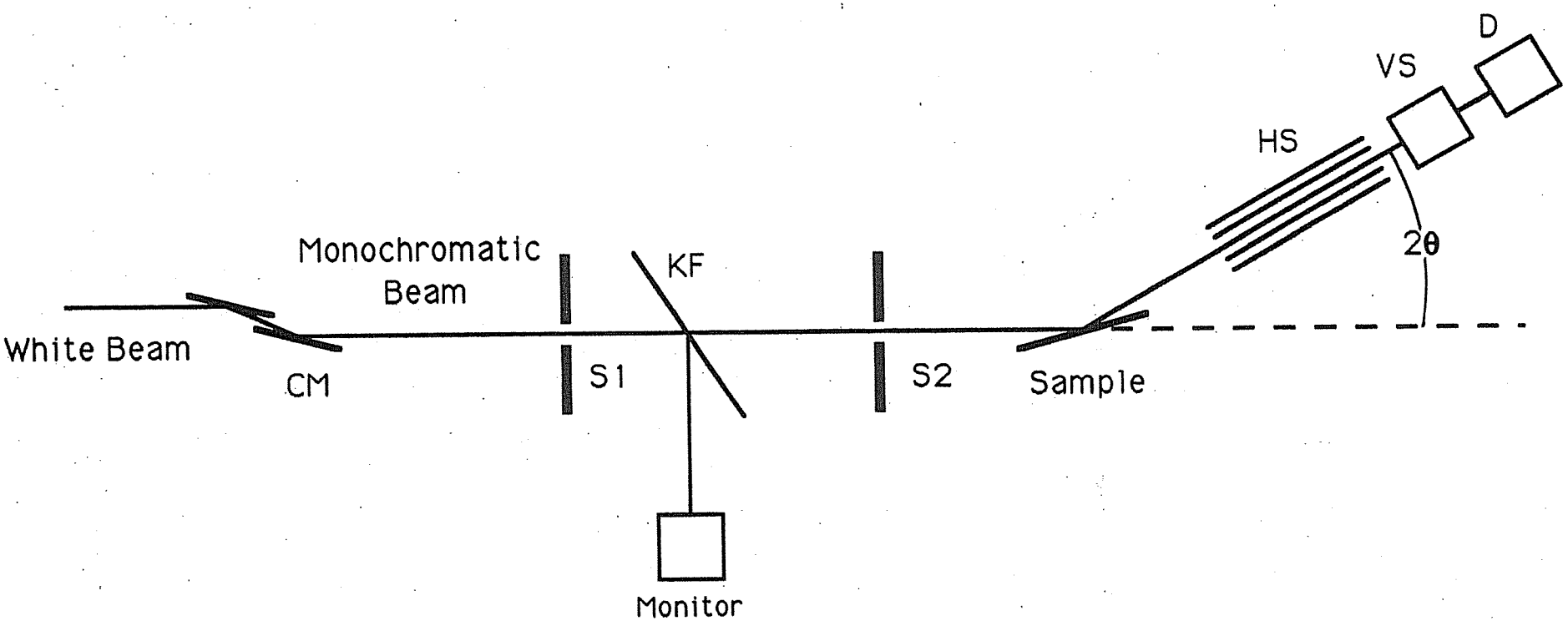

FIG.1 Schematic Diagram of Instrumentation For Glancing Angle X-ray Diffraction

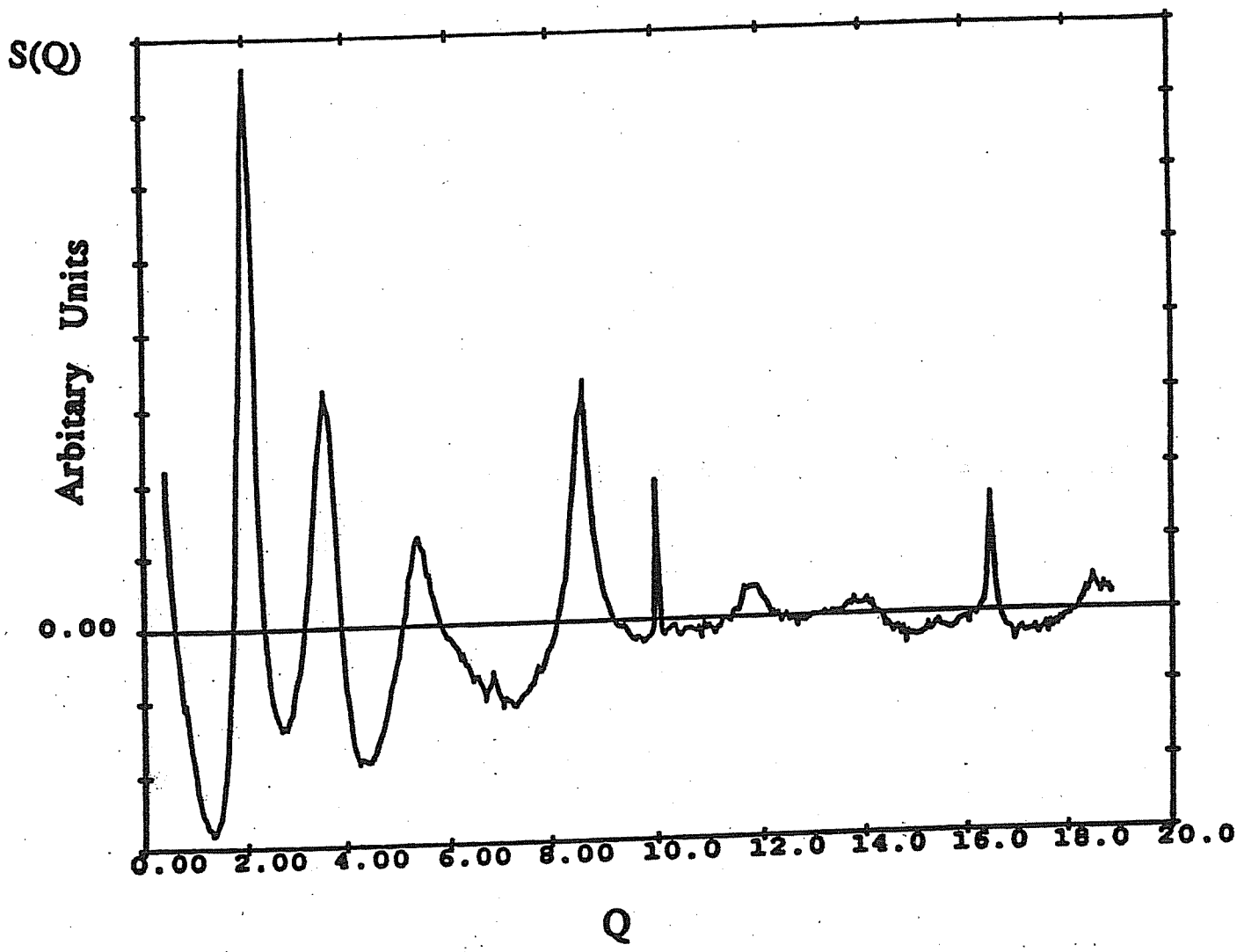

Fig.2a Structure Factor for Amorphous Hydrogenated Silicon: Carbon Determined by Glancing Angle X-ray Diffraction

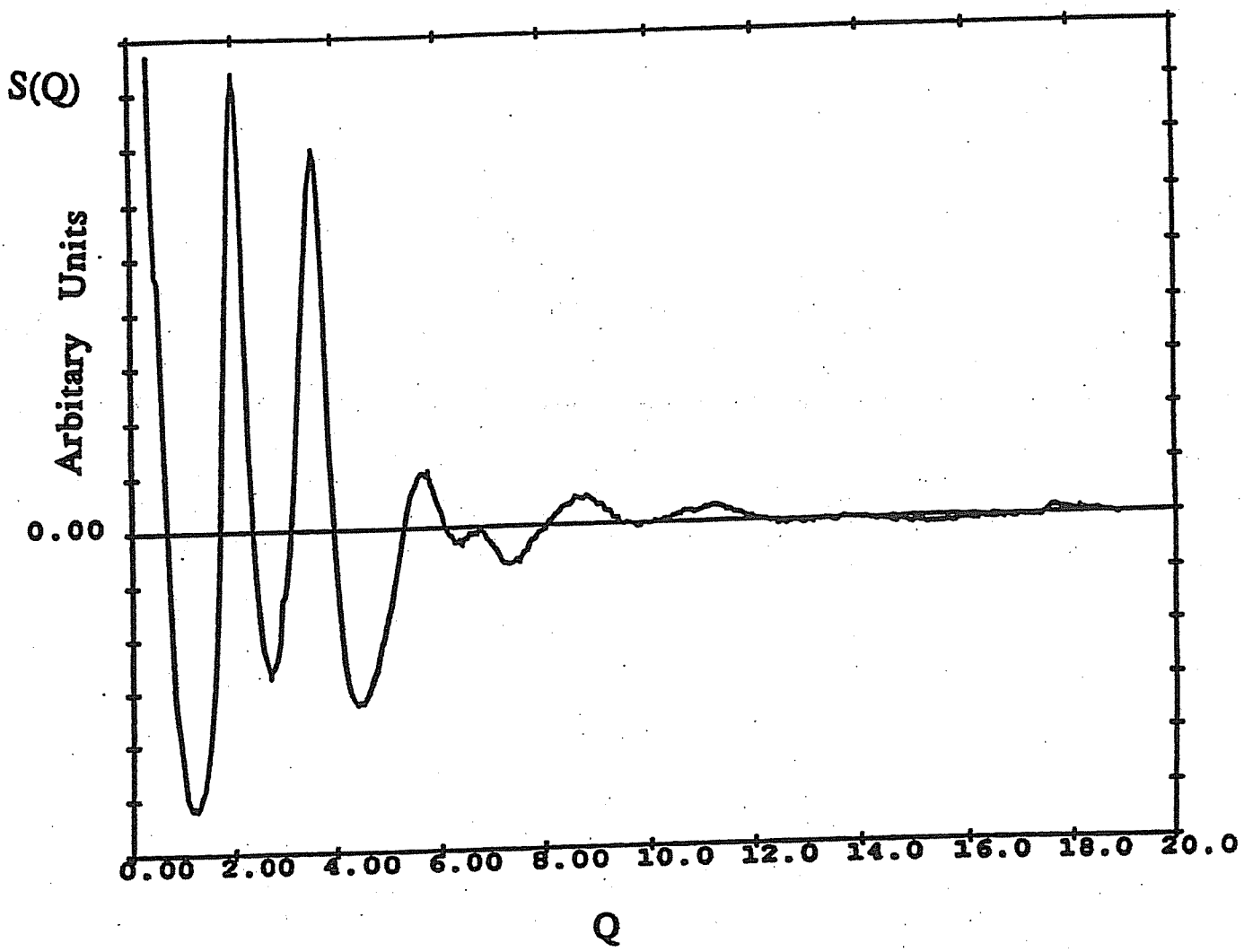

Fig.2b Structure Factor for Amorphous Hydrogenated Silicon: Carbon Determined by θ:2θ Powder Diffraction

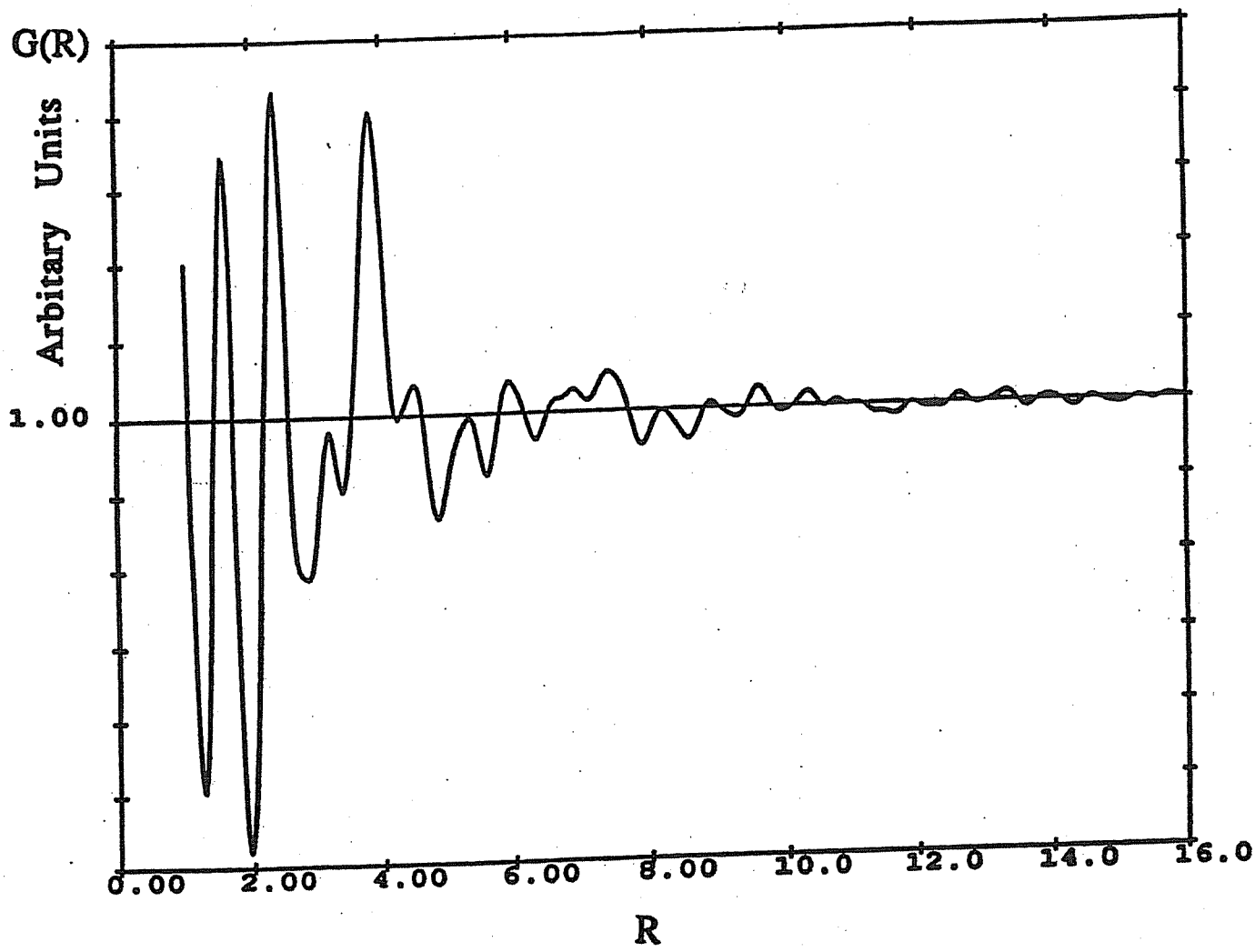

Fig.3a Pair Distribution Function for Amorphous Hydrogenated Silicon: Carbon Determined By Glancing Angle X-ray Diffraction

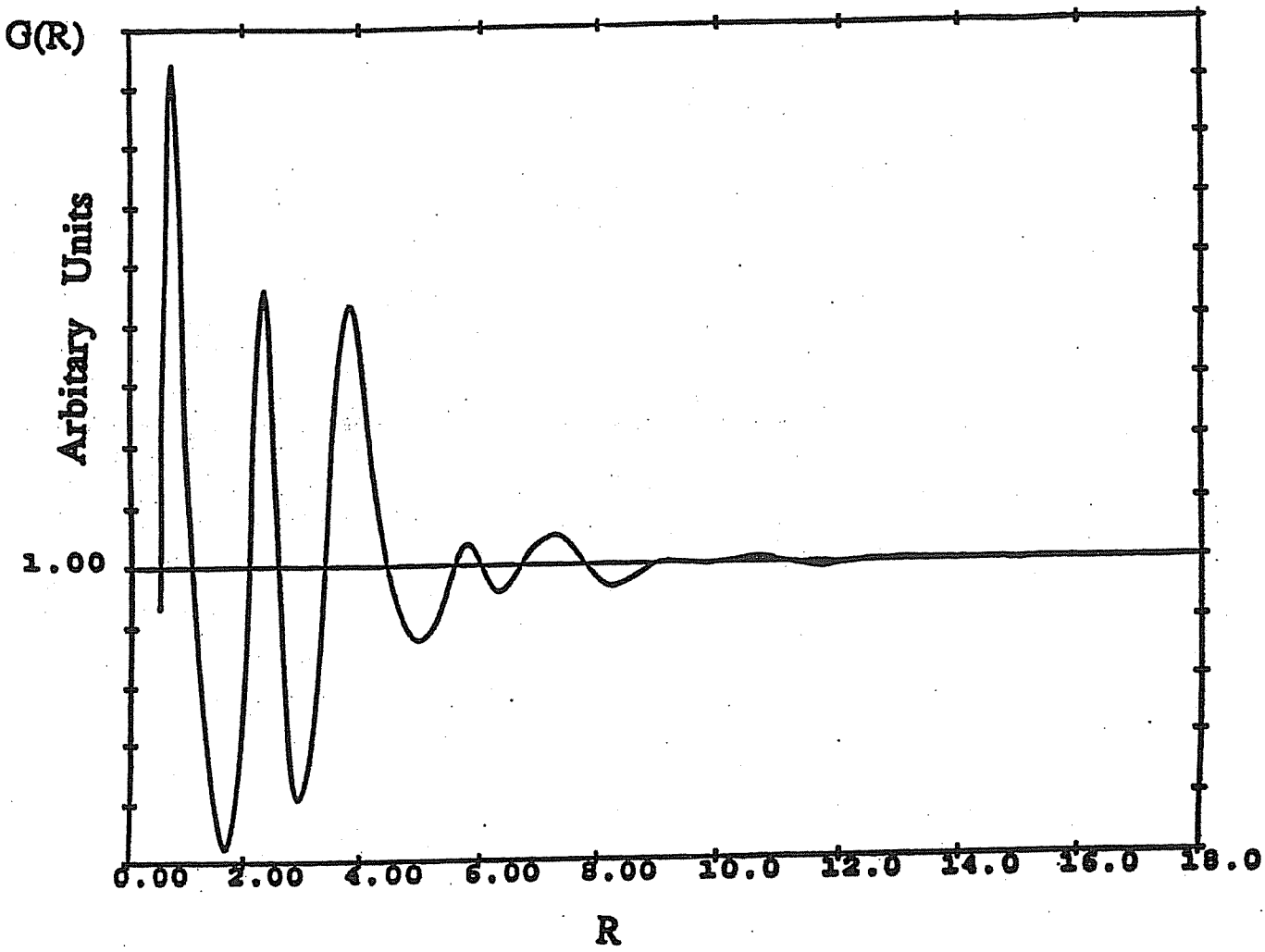

Fig.3b Pair Distribution Function for Amorphous Hydrogenated Silicon: Carbon Determined by θ:2θ Powder Diffraction