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A. Gibaud ([1,2,*]), J. Wang ([2]), M. Tolan ([3]), G. Vignaud ([4]) and S.K. Sinha ([2])

([1]) Équipe de Physique de l'État Condensé (**), Université du Maine, Faculté des Sciences, BP 535, 72017 Le Mans Cedex, France
([2]) Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439, USA
([3]) Institut für Experimental Physik, Christian-Albrechts-Universitat Kiel Olshausenstrasse 40, 2300 Kiel 1, Germany
([4]) E.S.R.F., BP 220, 38043 Grenoble Cedex, France

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Abstract. — An X-ray scattering experiment at small angle of incidence from the laterally modulated structure of a grating is reported. The attention is mainly focused on the determination of the linewidth of the different diffracted orders observed in transverse scans parallel to the surface of the grating. It is shown that the width of the different orders decreases when the incident angle increases and that the evolution of the width is consistent with the evolution of the instrumental resolution. It is also shown that if the spatial coherence of the beam is defined by the angular apertures of the incident and outgoing beams a identical behavior is expected at least when the diffracted orders are well separated.

Résumé. — L'étude par diffraction des rayons X aux petits angles de la structure modulée latéralement d'un réseau est présentée. Notre attention a été portée plus particulièrement sur la détermination de la largeur à mi-hauteur des différents ordres de diffraction observés dans des mesures faites parallèlement à la surface du réseau. Nous montrons que la largeur à mi-hauteur des ordres de diffraction décroît avec l'augmentation de l'angle d'incidence du faisceau sur la surface ce qui est en accord avec l'évolution de la fonction de résolution instrumentale. En prenant en compte la divergence angulaire du faisceau incident et du faisceau diffracté pour définir la cohérence spatiale du faisceau, nous obtenons une loi d'évolution identique ce qui nous permet de conclure que, pour des pics de Bragg bien séparés, les effets de cohérence et de résolution sont indiscernables.

X-ray diffraction by laterally modulated surfaces is presently one of the very up-to date topics in X-ray diffraction at small angle of incidence. The relevant surfaces are usually the surfaces of etched mesoscopic gratings some of which present a nearly perfect 1D mesoscopic structure of period, d, varying from 1 μm to several hundreds of microns. The use of such surfaces

(*) Author for correspondence
(**) URA N°807 du CNRS.

at small angles of diffraction is particularly appreciated to investigate the coherence length of X-rays. The periodic structure of the grating produces a periodic diffracted pattern when scans are performed parallel to the stepped surface of the material. The location of the various orders of diffraction is given by $2\pi/d$. A few studies have already been reported in which the spatial coherence length of X-rays has been investigated either with help of gratings or with pinholes [1–5]. In the case of gratings, the spatial coherence length is deduced from the width of the different diffracted orders. The first experiment reported by Tolan *et al.* [1, 3] was performed by rocking the grating at a fixed position of the detector whereas Salditt *et al.* [4] performed similar experiments at fixed incident angle but moving the detector. In such experiments the grating is mounted in the vertical plane and the reflected intensity measured in the horizontal plane. The lateral period of the grating can be modified by rotating the grating about an axis normal to its surface, *i.e* about q_z. The stepped surface has then an apparent periodicity in the q_x direction which is related to the period d of the grating and to the angle η of rotation by $d_x = d/\cos\eta$ (a similar effect is observed in the perpendicular direction). The basic idea to measure the spatial coherence of the beam [1, 4] is to rotate the grating until the apparent periodicity becomes larger than the coherence length of the X-ray beam so that the periodic structure is washed out. A condition for carrying out experiments is that the resolution must be coarse enough out of the scattering plane and extremely good in the scattering plane. Such a condition is easily obtained at small angle of scattering. However a complete understanding of coherence effects is only possible if one can discriminate between resolution and coherence effects.

The aim of this paper is to derive in a very simple way a general expression of the reflected intensity in the q_x, q_z scattering plane of a laterally modulated structure which presents a *quantized distribution of thickness* in the z direction. We present a complete calculation of scans performed parallel to the surface of the grating. We use the two different approaches of infinite spatial coherence corrected from instrumental resolution and of finite spatial coherence by studying the linewidth of the different diffracted orders and by comparing the observed results with those calculated from resolution and coherence effects. We do not consider the effect of the time coherence length which has the same effect as a wavelength spread and we therefore assume that this quantity is infinite.

We consider the stripes of the grating are parallel to the y direction and that the scattering plane will be define by the q_x, q_z directions with q_z normal to the face of the grating. We also assume for sake of simplicity that the grating presents a uniform electron density inside the material which makes its structure. The structure is supposed to be characterized by two specific thicknesses called L_1 and L_2 which are the thicknesses of the bottom B and top T parts of the material with respect to the reference level of the flat substrate on which it has been deposited (see Fig. 1). We neglect in this calculation the roughness of the two quantized surfaces and we also consider that one moves from B to T by a step function.

The passage from the altitude L_1 to the altitude L_2 is defined by a two-dimensional probability function $P(x, y)$ which is zero or 1 when one is respectively at the level L_1 or L_2 in the structure. This is a way of seeing the structure of the surface as a binary object. We then use the kinematical approximation in which the scattered amplitude is defined as the Fourier transform of the electron density:

$$A(Q) = \int_{-\infty}^{\infty} \rho(r) \mathrm{e}^{-iQ\cdot r} \mathrm{d}r \tag{1}$$

The integration is carried out in the 3 directions x, y and z and separated into two terms according to whether the scattering point belongs to the bottom or top part of the material.

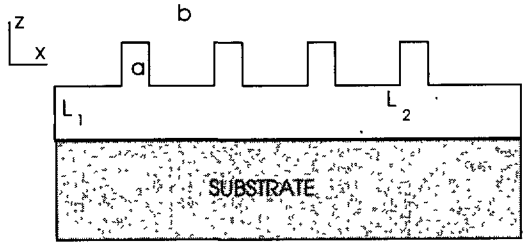


Fig. 1. — A schematic representation of the grating structure.

This yields to the following scattering amplitude:

$$\begin{aligned} A(Q) &= \rho_s \int_{\infty}^{0} \mathrm{d}z \mathrm{e}^{-iq_z z} \int\int_S \mathrm{d}x\mathrm{d}y \mathrm{e}^{-i(q_x+q_y y)} + \rho \int_0^{L_1} \mathrm{d}z \mathrm{e}^{-iq_z z} \int\int_S \mathrm{d}x\mathrm{d}y \mathrm{e}^{-i(q_x x+q_y y)} \\ &+ \rho \int_{L_1}^{L_2} \mathrm{d}z \mathrm{e}^{-iq_z z} \int\int_S \mathrm{d}x\mathrm{d}y P(x,y) \mathrm{e}^{-i(q_x x+q_y y)} \end{aligned} \quad (2)$$

where S is the surface of the sample which is illuminated coherently by the X-ray beam. In the absence of a general formalism to describe the scattering in terms of partial coherence of the X-ray source, there are two ways to perform the above integration. One can consider either a perfectly coherent beam of infinite coherence length and take into account the resolution function, or a beam of finite coherence lengths and integrate over the limiting values ξ_x, ξ_y which are the coherence lengths of the beam along the x and y directions. In the first case (called the R case in the following) the surface S is infinite and integration of (2) leads to:

$$\begin{aligned} A_{\infty}(Q) &= \left(-\frac{\rho_s}{iq_z} + \rho L_1 \mathrm{e}^{-\frac{iq_z L_1}{2}} \operatorname{sinc}\left(\frac{q_z L_1}{2}\right)\right) \delta q_x \delta q_y \quad (3a) \\ &+ \rho(L_2 - L_1) \operatorname{sinc}\left(\frac{q_z(L_2-L_1)}{2}\right) \mathrm{e}^{\frac{iq_z(L_1+L_2)}{2}} \int\int_{S=\infty} \mathrm{d}x\mathrm{d}y P(x,y) \mathrm{e}^{-i(q_x x+q_y y)} \end{aligned}$$

The scattering cross-section is then given by:

$$S_{\infty}(Q) = A_{\infty}(Q) A^*_{\infty}(Q) \quad (3b)$$

and the measured intensity is the convolution of the scattering cross-section with the instrumental resolution function. In reflection geometry and at small angle of incidence this yields:

$$I_{\mathrm{R}}(Q) = \frac{1}{q_z^2} \int S(Q') R(Q-Q') \mathrm{d}Q' \quad (4)$$

In the second case (called the C case in the following), the integration along the x and y directions is limited to the coherence lengths of the beam in these directions and produces a similar effect as to truncating the electron density of the material by a two-dimension step function $\prod$ of FWHM ξ_x, ξ_y:

$$\rho_{\mathrm{c}}(r_{/\!/}) = \rho_{\infty}(r_{/\!/}) \cdot \prod(r_{/\!/}) \quad (5)$$

The scattered amplitude is then the convolution of the infinite scattered amplitude with the Fourier transform of the step function and leads to:

$$\begin{aligned} A_c(Q) &= \left(-\frac{\rho_s}{iq_z} + \rho L_1 \mathrm{e}^{-\frac{iq_z L_1}{2}} \sin\mathrm{c}\left(\frac{q_z L_1}{2}\right)\right) \xi_x \xi_y \sin\mathrm{c}\frac{q_x \xi_x}{2} \sin\mathrm{c}\frac{q_y \xi_y}{2} \\ &\quad + \rho(L_2 - L_1)\sin\mathrm{c}\left(\frac{q_z(L_2 - L_1)}{2}\right) \mathrm{e}^{\frac{iq_z(L_1+L_2)}{2}} \int\int_{S=\xi_x\xi_y} \mathrm{d}x\mathrm{d}y P(x,y) \mathrm{e}^{-i(q_x x + q_y y)} \end{aligned} \tag{6}$$

Taking into account the geometry of the scattering experiment, one gets the expression of the measured intensity for finite coherent scattering:

$$I_c(Q) = \frac{1}{q_z^2} \left| \begin{array}{c} \left(-\frac{\rho_s}{iq_z} + \rho L_1 \mathrm{e}^{-\frac{iq_z L_1}{2}} \sin\mathrm{c}\left(\frac{q_z L_1}{2}\right)\right) \xi_x \xi_y \sin\mathrm{c}\frac{q_x \xi_x}{2} \sin\mathrm{c}\frac{q_y \xi_y}{2} \\ +\rho(L_2 - L_1)\sin\mathrm{c}\left(\frac{q_z(L_2-L_1)}{2}\right) \mathrm{e}^{i\frac{q_z(L_2+L_1)}{2}} \int\int_{S=\xi_x\xi_y} \mathrm{d}x\mathrm{d}y P(x,y)\mathrm{e}^{-i(q_x x+q_y y)} \end{array} \right|^2 \tag{7}$$

In the following, we restrict ourselves to the case of the grating that is to say to the case of a laterally modulated structure which is periodic. We assume that the grating has a uniform electron density so that $L_1 = 0$, $L_2 = h$ and $\rho_s = \rho$. Expressions (3a) and (7) then transform into:

$$S_\infty(Q) = \left| -\frac{\rho_s}{iq_z}\delta q_x \delta q_y + \rho h \sin\mathrm{c}\left(\frac{q_z h}{2}\right) \mathrm{e}^{\frac{i(q_z h)}{2}} \int\int_{S=\infty} \mathrm{d}x\mathrm{d}y P(x,y)\mathrm{e}^{-i(q_x x + q_y y)} \right|^2 \tag{8a}$$

$$\begin{aligned} &S_c(Q) = \\ &= \left| -\frac{\rho_s}{iq_z}\xi_x\xi_y \sin\mathrm{c}\frac{q_x\xi_x}{2} \sin\mathrm{c}\frac{q_y\xi_y}{2} + \rho h \sin\mathrm{c}\left(\frac{q_z h}{2}\right) \mathrm{e}^{i\frac{q_z h}{2}} \int\int_{S=\xi_x\xi_y} \mathrm{d}x\mathrm{d}y P(x,y) \mathrm{e}^{-i(q_x x+q_y y)} \right|^2 \end{aligned} \tag{8b}$$

Before completing these calculations, we point out that the first term of the modulus which is observed only in the specular part of the scattering, produces an obvious phase shift between the specular and the off-specular contributions. This behavior has been already evidenced by Tolan *et al.* [1, 6] and is clearly illustrated in this calculation. It is thus clear that the phase shift comes from the substrate and it can even be inferred that if it was possible to maintain the grating without its substrate in the reflection geometry, this feature would disappear. We also note that this phase shift can be more subtle in the case of asymmetric non-square structures [6].

We now consider the second term of (8) and we derive $P(x, y)$. The width of the stripes is called a, and their periodicity, b, with a profile which is a step function. The two-dimensional probability function reduces in such a case to:

$$P(x,y) = \prod_{\mathrm{a}}(x) * \sum_{p=0}^{N-1} \delta(x - pb) \tag{9}$$

in which $\prod$ is the symbol of the step function of FWHM $= a$, δ is the Dirac distribution and $*$ stands for the convolution operation. The summation is carried out over a number N of periods which really depends on the coherence length of the X-ray beam. In the R case N is infinite and in the C case N depends on the projected value of the coherence length along the x direction. For a beam presenting an incident angle α and an exit angle β with respect to the surface of the sample, it is important to compare the following parameters:

- the footprint F of the beam on the sample surface, *i.e*, $F = t/\sin\alpha$ where t is the thickness of the beam defined by the front collimation;
- the coherently illuminated part of the sample, *i.e.* the incoming coherence cut-off length of the beam projected on the surface $\xi_x^{\rm i} = \xi^{\rm i}/\sin\alpha$;
- the coherently "seen" part of the sample , *i.e.* the outgoing coherence cut-off length of the beam projected on the surface $\xi_x^{\rm e} = \xi^{\rm e}/\sin\beta$;
- the characteristic length $L_{/\!/}$ associated to the resolution of the instrument in the transverse direction, *i.e.*, $L_{/\!/} = 2\pi/\Delta q_x$;
- the period b of the grating.

To properly measure a diffraction pattern from a grating, the following condition must be fulfilled:

$$F > \xi_x, L_{/\!/} > b \tag{10}$$

It is then interesting to understand what we can expect from the two calculations. In the C case the incident number of periods which is illuminated coherently is $\tilde{N}^{\rm i} = \frac{\xi^{\rm i}}{b\alpha}$. This number is not constant for a q_x scan at a constant q_z wavevector transfer. Its dependence with the angle of incidence α is given by:

$$\tilde{N}^{\rm i}(q_x, q_z) = \frac{\xi^{\rm i}}{b\alpha} = \frac{4\pi\xi^{\rm i}}{b\lambda q_z\left(1 + \dfrac{q_x 4\pi}{\lambda q_z^2}\right)} = \frac{N^{\rm i}(0, q_z)}{1 + \dfrac{q_x 4\pi}{\lambda q_z^2}} \tag{11}$$

where λ is the wavelength of the X-ray beam. An alternative approach to maintain $\tilde{N}^{\rm i}$ constant has been recently proposed by Salditt *et al.* who use a non-classical set-up to perform the measurements. It must be stressed at this stage that $\tilde{N}^{\rm i}$ can be very different from one source to another one and that typically for a normal tube a coherence length of several tenth of microns can be expected and that for a synchrotron source the coherence length can be a hundred time greater as reported in [2,3]. Further progress can be made in (11) by introducing the expression of the coherence length of the beam. Using the criterion of Rayleigh and following the Van Citert-Zernike theorem [6] we have:

$$\xi^{\rm i} = \frac{\lambda}{\delta\alpha} \tag{12}$$

where $\delta\alpha$ is the divergence of the incident beam. The number of stripes which is coherently illuminated by the incident beam is then:

$$\tilde{N}^{\rm i}(q_x, q_z) = \frac{4\pi}{b q_z \delta\alpha\left(1 + \dfrac{q_x 4\pi}{\lambda q_z^2}\right)} \tag{13}$$

We now express a similar quantity which can be defined from the point of view of an observer located at the detector position:

$$\tilde{N}^{\rm e}(q_x, q_z) = \frac{4\pi}{b q_z \delta\beta\left(1 - \dfrac{q_x 4\pi}{\lambda q_z^2}\right)} \tag{14}$$

and we finally assume that these incident and exit quantities are mixed together so as to define an average number $\tilde{N}$ of stripes coherently illuminated.

The Fourier transform of equation (9) is respectively for the R and C cases:

$$\begin{aligned}\Phi_\infty(q_x,q_z) &= \frac{a}{b}\delta q_y \operatorname{sin\,c}\frac{q_x a}{2}\sum_n \delta\left(q_x - \frac{2\pi n}{b}\right)\\ \Phi_{\mathrm{c}}(q_x,q_y) &= \xi_y a \operatorname{sin\,c}\frac{q_y\xi_y}{2}\operatorname{sin\,c}\frac{q_x a}{2}\frac{\sin\dfrac{q_x\tilde{N}b}{2}}{\sin\dfrac{q_x b}{2}}\mathrm{e}^{-i\frac{q_x(\tilde{N}-1)b}{2}}\end{aligned} \tag{15}$$

and the off-specular measured intensity is given at $q_y = 0$ by:

$$\begin{aligned}I_{\mathrm{R}}(q_x,q_z) &= \frac{h^2a^2}{q_z^2b^2}\operatorname{sin\,c}^2\frac{q_z h}{2}\operatorname{sin\,c}^2\frac{q_x a}{2}\sum_n \delta\left(q_x - \frac{2\pi n}{b}\right) * R(q_x)\\ I_{\mathrm{c}}(q_x,q_y) &= \xi_y^2\frac{h^2a^2}{q_z^2}\operatorname{sin\,c}^2\frac{q_z h}{2}\operatorname{sin\,c}^2\frac{q_x a}{2}\frac{\sin^2\dfrac{q_x\tilde{N}b}{2}}{\sin^2\dfrac{q_x b}{2}}\end{aligned} \tag{16}$$

These two expressions are extremely similar and only differ by the fact that in one case we measure diffracted peaks having a width given by the instrumental resolution and in the other case a width given by the coherent limited term in the q_x direction. We now present a comparison of these two quantities. We assume that the resolution function is Gaussian and has a width Δq_x. This quantity is obtained by differentiation of the x component of the wavevector transfer [8,9] and by assuming that the different terms are random Gaussian variables. It can be then shown that:

$$\Delta^{\mathrm{R}}q_x = \frac{q_z w}{2}\sqrt{1+\frac{4\pi q_x}{\lambda q_z^2}\left(2\frac{\delta\alpha^2-\delta\beta^2}{w^2}+\frac{4\pi q_x}{\lambda q_z^2}\right)} \tag{17}$$

where $w = \sqrt{\delta\alpha^2+\delta\beta^2}$ is the HWHM of the direct beam and $\delta\beta$ the divergence of the outgoing beam.

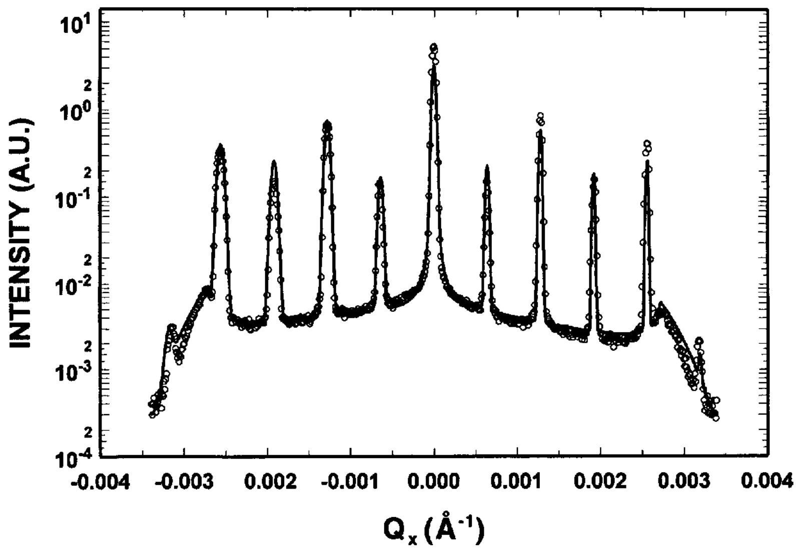
To estimate how this quantity differs from what one can expect from the coherent model, we define the coherent widths as:

$$\begin{aligned}\Delta_{\mathrm{c}}^{\mathrm{i}}q_x &= \frac{2\pi}{\tilde{N}^{\mathrm{i}}b} = \frac{q_z\delta\alpha}{2}\left(1+\frac{q_x4\pi}{\lambda q_z^2}\right)\\ \Delta_{\mathrm{c}}^{\mathrm{e}}q_x &= \frac{2\pi}{\tilde{N}^{\mathrm{e}}b} = \frac{q_z\delta\beta}{2}\left(1-\frac{q_x4\pi}{\lambda q_z^2}\right)\end{aligned} \tag{18}$$

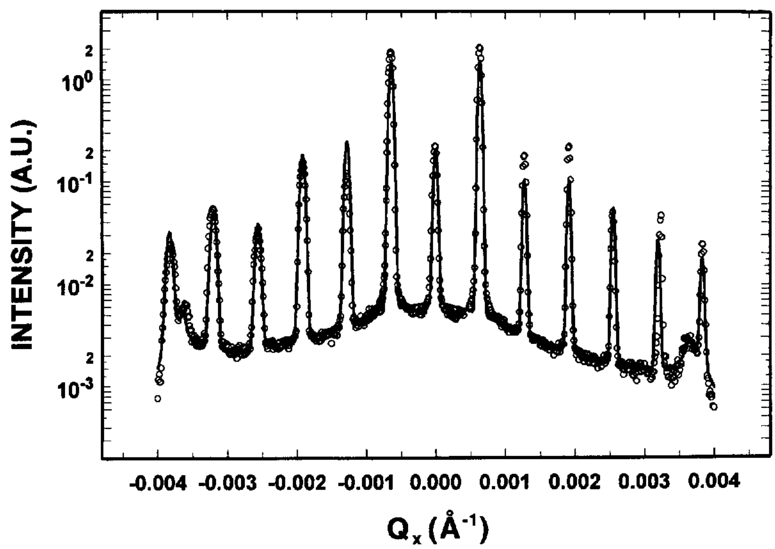
and we consider that these quantities are random Gaussian variables so that we define the real width for coherence as:

$$\Delta^{\mathrm{c}}q_x = \frac{2\pi}{\tilde{N}b} = \sqrt{\Delta_{\mathrm{c}}^{\mathrm{i}}q_x^2+\Delta_{\mathrm{c}}^{\mathrm{e}}q_x^2} \tag{19}$$

It then turns out that under these assumptions of random Gaussian variables, the coherent and the resolution expressions (17) and (19) are rigorously the same and in the case where $\delta\beta > \delta\alpha$ they lead to the narrowing of the peaks when one goes from q_x negative to q_x



a)



b)

Fig. 2. — Observed and calculated scattering intensities in transverse scans at $q_z = 0.19$ Å^{-1} (a) and at $q_z = 0.216$ Å^{-1} (b). The calculation is made according to the model proposed by Tolan *et al.* [6].

positive. The experimental verification of this statement is made possible by the analysis of the width of the different diffracted orders of the grating. Experiments were performed at the NSLS (Brookhaven National Lab.) on the X10B Exxon beam line. We used a 9832 Å periodic silicon grating and a wavelength $\lambda = 1.185$ Å. The incident beam and outgoing beams were collimated to yield a direct beam of FWHM $= 0.032°$. The rear collimation slit was more opened than the front one so that $\delta\beta > \delta\alpha$. We present in Figure 2 typical transverse scans at a maximum and a minimum value of the reflectivity curve. The well defined peaks allow the determination of the width which is in turn compared to (17) and (19). The results

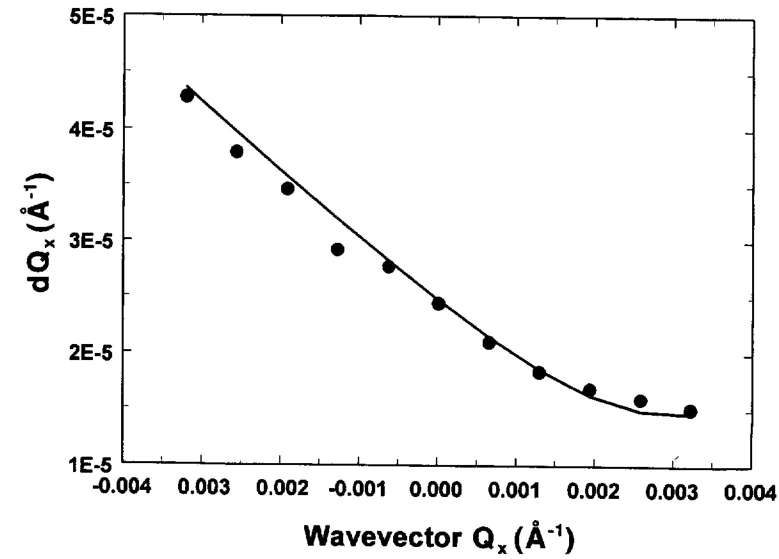


Fig. 3. — Evolution of the observed and calculated (full line) widths of the different diffracted orders as a function of q_x at $q_z = 0.19$ Å^{-1} (the calculation is made according to Eqs. (17) and (19)).

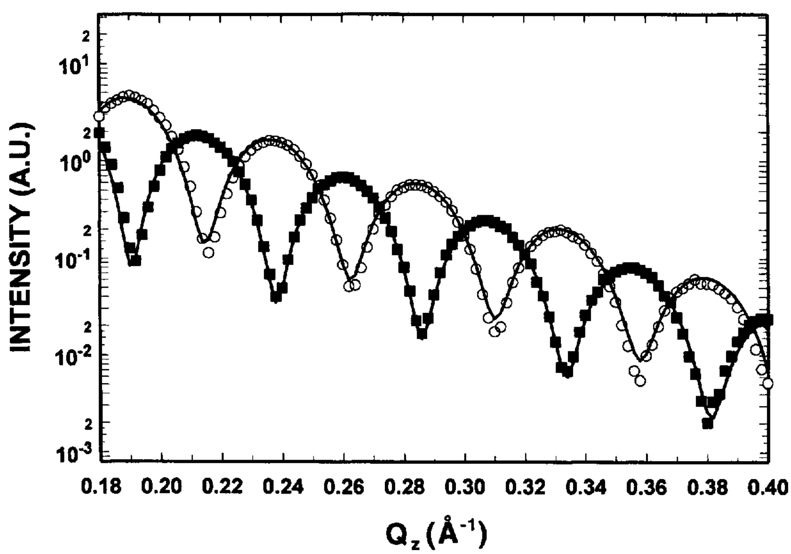


Fig. 4. — The calculation of the reflected intensity at $q_x = 0$ Å^{-1} specular (open circles) and at the position of the first order of diffraction (squares) giving the height $h = 132$ Å of the grating (the two curves are calculated simultaneously).

are presented in Figure 3 and show the beautiful agreement between the experimental and calculated values for $q_z = 0.19$ Å^{-1} The full line in Figure 3 is a fit to the data according to equation (17) for this latter wavevector transfer and leads to $w = 0.016°$ and $\delta\beta = 0.014°$ in full agreement with the experimental set-up.

In addition a complete calculation of the transverse scans is also performed. However it was found that equations (16) do not completely agree with the observed data because the assumption of a step profile is too severe. The complete calculation is then carried out by convoluting the transverse resolution function with the expression given by Tolan *et al.* [6] which is equivalent to expression (16) in the case of a step profile but which allows a more general trapezoidal shape. Tolan *et al.*'s expression is useful to describe the form factor of a non-square grating and beautifully provides the analytical expression of the peak intensity of the different diffracted orders. The calculation is carried out after having determined the height h of the grating from the specular reflectivity as shown in Figure 4. From the calculation of the specular reflectivity we obtain $h = 132$ Å, a value which is then held fixed in the calculation of the transverse scans. It is found necessary to introduce an asymmetry in the trapezoidal lineshape to describe the intensity of the different orders observed in transverse scans (Figs. 2a and 2b). This yields a profile defined by $b_1 = 1500 \pm 50$ Å, $b_2 = 3200 \pm 100$ Å and $g = 3500 \pm 100$ Å with the notation of [6].

In conclusion, we have shown in this paper that the narrowing of the width of the diffracted peaks observed in the transverse scans when q_x goes from negative to positive values is consistent with the evolution of the resolution function in this direction. In addition, by calculating the evolution of the linewidth of the various orders in two different manners, *i.e.* the coherent approach (in which we assume perfect incoming and outgoing coherence cut-off lengths) and the resolution approach, we show that the evolution of the width in terms of coherence effects is identical to what one can expect from a resolution effect at least under certain circumstances which are a coherence length less than the size of the beam and non-overlapping reflections. This agreement is caused by the fact that the coherence length is defined by equation (12) as stated in the Van-Citert-Zernike theorem [7] and that we incoherently add up the contributions of coherent areas over the size of the beam. However the agreement is only valid if we take in account both the incident *and* scattered beams to define the coherence length and as long as the diffracted orders do not overlap. This latter point is implicitly expressed in equation (16) and if this condition is not fulfilled we expect the resolution-folding process to give a wrong description of the scattering. In this paper, the experimental verification of this statement which is made clear in the two parallel calculations of (16), has been established in the simple case of non- overlapping reflections. A more general formalism in which it will be shown that coherence and resolution effects can be separated when the scattering arises from overlapping reflections analyzed with a partial coherent beam will be soon presented [10].

Acknowledgments

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