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Lattice Fringe Imaging of Modulated Structures

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ABSTRACT

A study is made of the accuracy with which two-beam lattice fringe images of modulated structures can be interpreted, with particular reference to spinodally decomposed alloys. The dependence of the image upon foil thickness, lens parameters and atomic segregation is discussed and the importance these parameters have upon similar images from any region of slowly varying interplanar spacings is outlined.

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I. INTRODUCTION

The method of two-beam lattice fringe imaging with tilted illumination has been used in many studies to investigate slowly varying changes in interplanar spacings such as occur in regions close to a grain boundary (Gronsky and Thomas 1977) and in spinodally decomposed alloys (Sinclair, Gronsky and Thomas 1976). (For a review of applications of the method see Sinclair (1979)). On the assumption of a one-to-one relationship between fringe spacings and interplanar spacings, many of these studies have given results which are in good agreement with other structural data about the relationship between composition and lattice parameter. However it has been established that in regions of rapidly varying interplanar spacing, such as occur close to dislocation cores, the method has serious shortcomings (Cockayne, Parsons and Hoelke 1971). It is therefore of some importance to establish the accuracy with which such images can be interpreted in the case of slowly varying interplanar spacings. Unlike the method of structural imaging where the experimental conditions necessary for reliable image interpretation have been defined (e.g Cowley and Iijima 1972, Cowley 1975) only approximate solutions to the problem have been offered in the case of two-beam imaging (e.g. Sinclair 1979, Sinclair and Thomas 1978), although the particular case of spinodal decomposition has been analyzed to varying degrees of approximation (Rez 1978, Spence, Cowley and Gronsky, 1979). In this paper, the accuracy with which interplanar spacings can be determined from interfringe spacings is studied with particular reference to experimental studies of spinodally decomposed alloys.

II. DISTRIBUTION OF ELASTIC DIFFUSE SCATTERING

For a foil of thickness t , we denote the two beams contributing to the image as $\phi_h(x) = \phi_h(x, t)$ ($h = 0, g$), where x is a coordinate in the object

parallel to the vector $\underline{g}$. In the diffraction pattern we assume that the scattered amplitude distribution $\Psi_{\underline{u}} \equiv \Psi_{\underline{u}}(t)$ is concentrated around the reciprocal lattice points $\underline{h}$, and we then partition this distribution in the form (shown in Fig. 1)

$$\Psi_{\underline{u}} = \sum_{\underline{h}} \phi_{\underline{h}}^{(\underline{h})} \quad \text{where } \underline{w} \equiv \underline{u}^1 = \underline{u} - \underline{h} \quad (1)$$

Then $\phi_{\underline{h}}(\underline{x}) = \int d\underline{w} \phi_{\underline{w}}^{(\underline{h})} \exp(2\pi i \underline{w} \cdot \underline{x})$

[For a detailed discussion see Anstis and Cockayne (1979)].

If we write $\phi_{\underline{h}}(\underline{x}) \equiv |\phi_{\underline{h}}(\underline{x})| \exp\{2\pi i \theta_{\underline{h}}(\underline{x})\}$

then the image intensity at $\underline{x}$ is

$$I(\underline{x}) = |\phi_{\underline{o}}(\underline{x})|^2 + |\phi_{\underline{g}}(\underline{x})|^2 + 2|\phi_{\underline{o}}(\underline{x})| |\phi_{\underline{g}}(\underline{x})| \cos\{2\pi(\underline{g} \cdot \underline{x} + \langle \theta_{\underline{g}} - \theta_{\underline{o}} \rangle(\underline{x}))\} \quad (2)$$

The local period is then defined as

$$d^1(\underline{x}) \equiv 1/g^1(\underline{x})$$

where $g^1(\underline{x}) = g + d(\theta_{\underline{g}} - \theta_{\underline{o}})(\underline{x})/dx$

i.e. $I(\underline{x}) \propto \cos(2\pi g^1 \underline{x})$

If we now introduce the lens transfer function

$$\chi(u) = 2\pi(\Delta f \lambda \frac{u^2}{2} + C_s \lambda^3 \frac{u^4}{4})$$

where Δf is the defocus, C_s the spherical aberration coefficient, and λ the electron wavelength, then the image is formed from

$$\begin{aligned} \phi_{\underline{h}}^1(\underline{x}) &= \int d\underline{w} \phi_{\underline{w}}^{(\underline{h})} \exp\{2\pi i \underline{w} \cdot \underline{x}\} \exp\{i\chi(\underline{w})\} \\ &= |\phi_{\underline{h}}^1(\underline{x})| \exp\{2\pi i \theta_{\underline{h}}^1(\underline{x})\} \\ &= |\phi_{\underline{h}}^1(\underline{x})| \exp\{2\pi i \theta_{\underline{h}}(\underline{x})\} \exp\{i\Gamma_{\underline{h}}(\underline{x})\} \end{aligned} \quad (4)$$

where $\Gamma_{\underline{h}}(\underline{x}) = 2\pi(\theta_{\underline{h}}^1 - \theta_{\underline{h}})(\underline{x})$

and the aberrated image is

$$I^1(x) = |\phi_o^1(x)|^2 + |\phi_g^1(x)|^2 + 2|\phi_o^1(x)||\phi_g^1(x)| \cos \{2\pi(gx + \langle \theta_g - \theta_o \rangle(x)) + \langle \Gamma_g - \Gamma_o \rangle(x)\}.$$

The local period is $d^{11} = 1/g^{11}$

where $g^{11} = g^1 + \frac{1}{2\pi} d \left[\langle \Gamma_g - \Gamma_o \rangle(x) \right] / dx$.

Thus $I^1(x) \propto \cos \{2\pi(g^1 x + (\Gamma_g - \Gamma_o)(x))\}$ (4)

It is sometimes assumed that, in eqn (4), $\Gamma_g(x) = \chi(g^1)$ i.e. that if the local periodicity at x in the unaberrated image is $1/g^1$, then the aberrated image at x can be obtained from the unaberrated image of eqn (2) by introducing into eqn (2) the phase shift $\chi(u)$ at $u = g^1$. But there is no a priori reason to make this assumption because the derivation of eqn (2) from eqn (1) illustrates that the local image periodicity $d^1 \equiv 1/g^1$ arises from the distribution of $\phi_u^{(h)}$ about $u = g^1$, and not just from $\phi_{g^1}^{(h)}$. To stress this point we can note that the observations of a localized periodicity $1/g^{11}$ in the image does not imply that $\chi(u)$ is sampled at $u = g^{11}$ since $\phi_u^{(h)}$ may be identically zero at $u = g^{11}$.

The severity of making the approximation $\Gamma_g(x) = \chi(g^1)$ depends upon both the rate of change of d^1 over x (i.e. how rapidly the object spacing varies (where the object may be the object wave function)), and of the rate of change of $\chi(u)$ about $u = g^1$. The more localized is the region over which $d^1 \approx \text{constant}$, the more delocalized is $\phi_u^{(h)}$. This can easily be understood by analogy with diffraction of light from a finite number N of slits each d apart (e.g. see Jenkins and White (1957)). The diffraction pattern of this system is a set of 'satellite' reflections distance $\Delta u = 1/d$ apart, modulated by the function $\sin(\pi N u d)/(u d)$. The diffraction pattern has 'width' $\Delta u^1 = 2/N$, so that the larger is N , the more localized is the

diffraction pattern. In pictorial terms, if the object of spacing d is localized to a region ΔX , $\chi(u)$ may vary across $\phi_u^{(h)}$ to such an extent that it influences $\phi_u^{(h)}$ sufficiently to extend the image well beyond the region ΔX .

III. DIFFRACTION FROM MODULATED STRUCTURES

We consider diffraction by crystals having interplanar spacings $d(x)$ which vary periodically with x . The two cases considered are shown in Fig. 2, one having an interplanar spacing which varies linearly according to the expression

$$\begin{aligned} d(x) &= d_0 (1 + \epsilon (\frac{4|x|}{d_L} - 1)) \text{ for } |x| \leq \frac{d_L}{2} \\ &= d(x-d)_L \text{ for } |x| > \frac{d_L}{2} \end{aligned} \quad (5)$$

and the other having an interplanar spacing which varies sinusoidally according to the expression

$$d(x) = d_0 (1 + \epsilon \cos (\frac{2\pi x}{d_L})) \quad (6)$$

In both cases the interplanar spacing varies only in the foil plane, and not normal to it, and ϵ defines the maximum variation in interplanar spacing, d_0 the average interplanar spacing and d_L the modulation period. For both systems, the diffraction pattern has satellite reflections at $u^1 = n/d_L$ (n integral) on either side of the fundamental reflections which are themselves at $u = m/d_0$ (m integral) (Fig. 2b).

1. Sinusoidal Modulation

If $R(x)$ is the displacement of the atom normally at x in the perfect crystal, then the interplanar spacing given in eqn. 6 is obtained for

$$R(x) = \frac{d_L \epsilon}{(\frac{2\pi}{d_L})} \sin (\frac{2\pi x}{d_L}) = \frac{d_L \epsilon}{2\pi} \sin \frac{2\pi x}{d_L}$$

Using this expression for $R(x)$, the structure factor $F(n)$ for the n th satellite reflection about the fundamental reflection $m = 1$ has the form (de Fontaine 1966).

$$F(n) \propto f_{el}(\theta_n) A(n)$$

where $A(n) = J_n\left(\epsilon \left| \frac{d_L}{d_0} + n \right| \right) = J_n\left(\epsilon \left[\frac{d}{d_0} L + N \right] \right)$ (7)

(A more general expression may be derived for other values of m .) It is to be noted that eqn 7 reduces to $A(n) = J_n\left(\epsilon \frac{d_L}{d_0}\right)$ for large d_L/d_0 , and this is the result obtained using the deformable ion approximation, and given by Spence, Cowley and Gronsby (1979).

We note from tables of Bessel functions that, for $\epsilon \left(\frac{d_L}{d_0} + n \right) > 1.4$, $|A(0)|$ is not necessarily greater than $|A(1)|$ in eqn. 7 i.e. if $f_{el}(\theta_n)$ is assumed constant, the "Bragg" reflection is not necessarily stronger than the side band reflections. For example for $\epsilon = 0.1$ and $d_L/d_0 = 18$, which approximates the case for spinodal decomposition in Au + 77 at % Ni with $d_0 = d_{200}$ as investigated by Sinclair et al (1976), eqn. 7 gives $|A(1)| > |A(2)| > |A(0)|$.

The method of periodic continuation (Grinton & Cowley 1971) was used to calculate diffraction patterns and images for the parameters given above (Au + 77 at % Ni) with 100keV electrons. A study was first made for pure lattice distortion (assuming all atoms to be Ni). The variation of the scattered amplitudes $\phi_n^{(200)}$ (the n th satellite reflection of the 200 fundamental reflection) for $s_{200} = 0$ is shown in Fig. 3. Despite the thickness dependence of the diffracted amplitudes observed in Fig. 3b, the relative intensities of the fundamental and satellite reflections remain constant and within 4% of the ratios given in eqn. 7 throughout

the thickness range shown. At the same time Fig. 3 shows that the phases of these reflections relative to that of the incident beam remain almost constant, and close to $\pi/2$, to a thickness approaching 12nm. At $t \sim 12$ nm the phases deviate from this value as the magnitude of the incident beam becomes small.

2. Linear Variation in Interplanar Spacing

A similar analysis to that carried out above can be made for the interplanar spacing of eqn 5, shown pictorially in Fig. 2a. In carrying out the calculations using the method of periodic continuation, a displacement field was constructed to satisfy eqn 5, everywhere i.e. with no discontinuities either at the centre, or at the boundaries, of the extended cell. An analytical expression equivalent to eqn 7 can be obtained for the structure factors of the satellite reflections (to be published) but, with the analysis of 3.1 for comparison, it will suffice to summarize the results obtained.

Diffraction patterns and images were calculated for nickel using eqn 5 with $d_0 = d_{200}$, $d_L = 40d_0$, $s_{200} = 0$, 100 keV electrons and $\epsilon = 0.125$. For this value of ϵ the maximum variation in interplanar spacing is 12.5%. Consequently if we define the maximum interplanar spacing to be $d(\max)$, then the satellite reflection of $\phi_n^{(200)}$ for a foil thickness 4 nm, where it is seen that the elastic diffuse scattering in the region of $n = \pm 5$ is appreciable.

The variation of $\phi_n^{(200)}$ with foil thickness is shown in Fig. 5 for $n = 0, \pm 2$ (curves for other values of n are similar in form). As was the case for sinusoidal lattice distortion, the relative amplitudes of the beams remain constant (to 5%) for $t < 10$ nm and, as well, Fig. 5 shows that their phases, relative to that of the central beam, stay close to $\pm\pi/2$.

IV. IMAGES OF MODULATED STRUCTURES (2-BEAM)

1. Perfect Lens

Fig. 6 shows the calculated fringe spacings across one period of the sinusoidally modulated structure discussed in 3.1 (single atomic species) for a foil of thickness 4 nm. The parameters are those of Fig. 3 with the two beams $\phi_u^{(000)}$ and $\phi_u^{(200)}$ contributing to the image. Results for two values of C_s are shown. For $C_s = 0$ and $\Delta f = 0$ (or similarly $\chi(u) = \text{constant}$, $u \neq 0$), it is seen that the image spacings conform to the interplanar spacings of the object to better than 10%.

In eqn 1, if

$$\phi_u^{(h)}(t = t_2) = C^h \cdot \phi_u^{(h)}(t = t_1) \text{ for all } u \quad (8)$$

where C^h is a constant, then

$$\phi_h(x, t = t_2) = C^h \phi_h(x, t = t_1) \text{ and consequently (from eqn 2),}$$

$$g^1(x, t = t_2) = g^1(x, t = t_1)$$

i.e. the local fringe spacings for $t = t_1$ and $t = t_2$ are equal. Figs. 3 and 5 show that for both the sinusoidal and linear modulations considered, equation 8 is approximately satisfied for $t \lesssim 12$ nm. Consequently it is expected that since, as seen above for the sinusoidal modulation, the local fringe spacings closely reflect the object for $t = 4$ nm and $C_s = 0$, then they will do so for all $t \lesssim 12$ nm. Calculated images confirm this conclusion both for the sinusoidal and linear interplanar modulations.

2. Imperfect Lens

The influence of spherical aberration and defocus upon the image depends upon how $\chi(u)$ in eqn 2 varies across the distribution $\phi_u^{(g)}$.

Fig. 7 shows $\phi_u^{(200)}$ plotted for $t = 4$ nm for the sinusoidally modulated structure together with $\chi(u)$ for the 'optimum defocus' condition for tilted

illumination given by

$$\Delta f = -C_s \lambda^4 / 4d_o^2 \quad (9)$$

for $C_s = 3$ mm and 100keV electrons. Because both the first and second order satellite reflections are relatively strong compared with the 'Bragg' reflection ($n = 0$), it is clear from Fig. 7 that $\chi(u)$ is far from constant for the part of $\phi_u^{(200)}$ which contributes significantly to the image structure. For the n th satellite reflection, the difference in the phase angle $\chi(u)$ from its value at the 'Bragg position' is, for example, ($n = -2$) 126° , ($n = -1$) 38° , ($n = +1$) 40° . Consequently the image calculated with $C_s = 3$ mm and $\Delta f = -330$ nm, which is shown in Fig. 6, has spacings which differ greatly (by up to 50% for $t = 4$ nm) from the object spacings. With decreasing C_s (and with the corresponding optimum defocus values given by eqn 9) the image spacings more closely approximate those of the object (Fig. 8), because of the smaller variation in $\chi(u)$ across the distribution $\phi_u^{(h)}$. In particular for $C_s = 1$ mm, the deviation of fringe spacings from object spacings is less than 10% at optimum defocus.

At 'optimum defocus' the phase angle $\chi(u)$ is relatively constant about the 'Bragg position', compared with its form for other defocus values. Consequently it is to be expected that for defocus values other than 'optimum' the disagreement between object and image spacings will be at least as great as that for 'optimum defocus' and, in general, this is borne out by calculated images.

V. SINUSOIDAL VARIATION WITH ATOMIC SEGREGATION

We now consider the situation when there is a periodic segregation of atomic species (which, in practice, generally is the cause of the interplanar spacing variations). As a model, we take the same parameters as were used for the sinusoidal variation in interplanar spacings (3.1),

but we assume that in Au + 77 at % Ni, the Au atoms occupy that 23% of the modulated structure having the largest interplanar spacings in eqn 6. The diffraction pattern has the same form of fundamental and satellite reflections as for the pure Ni case, but the distribution of amplitude between satellite reflections differs markedly (Fig. 9b). For example the satellite reflection $n = -1$, which corresponds to larger interplanar spacings (where Au resides) increases in magnitude more rapidly with thickness, and has a shorter 'extinction distance', than the $n = +1$ satellite reflection. This is in accord with the larger scattering factor of Au compared with that for Ni. Consequently the relative amplitudes of the fundamental and satellite reflections are thickness dependent (c.f. Fig. 3 for pure Ni) as are their phase angles (Fig. 9a). It is therefore not surprising, following the analysis given in 4.1, that even for a perfect lens the images for $t = 4$ nm show fringe spacings very different from the interplanar spacings of the object (Figs. 10, 11). When imperfect lenses and optimum defocus are used, this disagreement between image and object increases. For example at optimum defocus (eqn 9) with $C_s = 3$ mm, some regions of the image have spacings which differ from the object by up to 50% for $t = 4$ nm (Fig. 10).

VI. MEAN FRINGE SPACING

In Fig. 11 calculated results are shown for a structure with sinusoidally varying interplanar spacing and atomic segregation for a foil of thickness 14 nm. Careful inspection shows that while for both examples of C_s given (0 and 3 mm) the period d_L of the object is reproduced in the image, the number of fringes within the period d_L is not equal to a $d_L/d_o (=18)$, but is 19 in one case and 22 in the other. This means that, in the image, the average spacing of the fringes across one period of the modulation may differ from d_o . In the diffraction pattern (Fig. 12, $t = 14$ nm)

corresponding to Fig. 11 this effect is observed as a shift of intensity to higher scattering angles.

VII. COMPARISON WITH EXPERIMENT

The preceding analysis shows that for the case of a spinodally decomposed Au + 77 at % Ni and conditions generally met experimentally (imperfect lens, atomic segregation), there is an appreciable difference between image and object spacings, but that the period of modulation is retained in the image. While the thickness used in the theoretical analysis is a lower limit to that used experimentally, selected calculations for thicker specimens indicate that the same difficulties occur for greater thicknesses of specimen. Fig. 13 reproduces an experimental example of an image from a Au + 77 at % Ni taken under the conditions considered in this paper. The measured fringes spacings are shown in Fig. 14. For this study $C_s = 1.6$ mm. It is apparent that the fringe spacings have a variation which extends beyond the bounds of pure Ni ($d_{200} = 0.176$ nm) and pure Au ($d_{200} = 0.204$ nm) in a way which can be explained by the above analysis. At the same time a detailed scrutiny of Fig. 13 shows terminating fringes at many points in the image, an example of which is shown in Fig. 15. Although the presence of numerous dislocations cannot be ruled out as an explanation, the analysis of 6 suggests that such terminating fringes arise from slight differences in thickness or focus across the object.

While the analysis in this paper explains a number of anomalies apparent in earlier studies of spinodally decomposed alloys, viz. fringe separations exceeding the lattice parameters of the constituent elements and terminating fringes, its prediction that in certain cases, Au + 77% Ni being one, the satellite reflections might be stronger than the 'Bragg' reflection does not appear to be observed in practice. This discrepancy could be due to the sinusoidal model of interplanar variation being inaccurate

or to the fact that the modulation d_L is not strictly preserved. The possibility that there is a variation in interplanar spacings in a direction normal to the foil plane also cannot be ruled out as an explanation, although this seems unlikely given the observed sharpness of the satellite reflections (Gronsky, Okada, Sinclair and Thomas, 1975).

VIII. CONCLUSIONS

The following conclusions can be drawn for two-beam lattice fringe images using tilted illumination and 100keV electrons:-

1. For slowly varying interplanar spacings, the variation of the lens function $\chi(u)$ across the distribution of elastic diffuse scattering must be considered when determining how closely interfringe and interplanar spacings match.

2. For systems approximating that of Au + 77 at % Ni ($d_L = 3.2$ nm, $d_L/d_o = 18$, $\epsilon \approx 0.1\%$), but where variations in scattering factor between the segregated atomic species can be ignored, two-beam images from thin crystals have agreement between image and object spacings to better than 10% for $C_s < 1$ mm, and to $\approx 50\%$ for $C_s \approx 3$

3. For systems as in (2) above, but where differences between scattering factors of segregated atoms are significant, severe distortion between interplanar and interfringe spacings may occur even for negligible C_s .

4. In two-beam images of systems with periodically varying interplanar spacings, the period spacing is reproduced in the image but the average fringe spacing within one period may not equal the average interplanar spacing. This may produce terminating fringes which do not correspond to dislocations, and may give an optical diffraction pattern with average spacings different from those of the electron diffraction pattern.

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FIGURE CAPTIONS

- Fig. 1 Nomenclature for elastic diffuse scattering.
- Fig. 2a The two forms of modulated interplanar spacing $d(x)$ considered. Mean spacing = d_o , modulation period = d_L .
- 2b The form of the elastic diffuse scattering for the modulated structures of (2a), with 'satellites' denoted by n .
- Fig. 3 The variation of phase angle $\theta_n^{(200)}$ and amplitude $\phi_n^{(200)}$ as a function of foil thickness for the n th satellite reflection in a Ni foil with sinusoidally varying interplanar spacing. 100keV electrons, $s_{200} = 0$, $d_L = 18d_o$, $d_o = d_{200}(\text{Ni})$, $\epsilon = 0.1$.
- Fig. 4 The distribution of elastic diffuse scattering $\phi_n^{(h)}$ for a Ni foil of thickness 4 nm for a modulated structure of the form given by eqn 5. 100keV electrons, $s_{200} = 0$, $d_L = 40d_o$, $\epsilon = 0.125$. The lens phase shift $\chi(u)$ is shown for $C_s = 3\text{mm}$ and optimum defocus, for tilted illumination.
- Fig. 5 The variation of phase angle $\theta_n^{(200)}$ and amplitude $\phi_n^{(200)}$ as a function of foil thickness for the n th satellite reflection in a Ni foil with linearly varying interplanar spacing. 100keV electrons, $s_{200} = 0$, $d_L = 40d_o$, $\epsilon = 0.125$.
- Fig 6 Calculated fringe spacings $d^1(x)$ across one period of a Ni foil with sinusoidally modulated interplanar spacing. 100keV electrons, $t = 4\text{nm}$, $s_{200} = 0$, $d_L = 18d_o$,

$d_o = d_{200}(\text{Ni})$, $\epsilon = 0.1$, tilted illumination. Results are shown for optimum defocus for $C_s = 3\text{mm}$ and 0mm .

Fig. 7 The distribution of elastic diffuse scattering $\phi_n^{(h)}$ for a Ni foil of thickness 4nm with a sinusoidally modulated interplanar spacing. 100keV electrons, $s_{200} = 0$, $d_L = 18d_o$, $\epsilon = 0.1$. The lens phase shift $\chi(u)$ is shown for optimum defocus for tilted illumination with $C_s = 3\text{mm}$.

Fig. 8 Calculated fringe spacings $d^1(x)$ across one period of a Ni foil with sinusoidally modulated interplanar spacing. Parameters as for fig. 6. Results are shown for optimum defocus for $C_s = 3\text{mm}$ and 1mm .

Fig. 9 The variation of phase angle $\theta_n^{(200)}$ and amplitude $\phi_n^{(200)}$ as a function of foil thickness for the n th satellite reflection for a Au + 77 at % Ni foil with sinusoidally varying interplanar spacing and atomic segregation. 100keV electrons, $s_{200} = 0$, $d_L = 18d_o$, $d_o = d_{200}(\text{Ni})$, $\epsilon = 0.1$.

Fig. 10 Calculated fringe spacings $d^1(x)$ across one period of a Au + 77 at % Ni foil of thickness 4nm , with sinusoidally modulated interplanar spacing and atomic segregation. 100keV electrons, $s_{200} = 0$, $d_L = 18d$, $d_o = d_{200}(\text{Ni})$, $\epsilon = 0.1$, tilted illumination. Results are shown for optimum defocus for $C_s = 0$ and 3mm .

Fig. 11 As for fig. 10, but with foil thickness 14nm .

- Fig. 12 Relative magnitudes of elastic diffuse reflections for the model of fig. 10, for foils of thickness 4, 10, and 14nm. The shift of the scattering to higher scattering angles ($n = 1$) for $t = 14\text{nm}$ is observed.
- Fig. 13 Lattice fringe image of a Au + 77 at % Ni alloy aged for 21 hrs. at 150°C , with inset diffraction pattern showing the imaging condition. Two beams were used, centered about the optic axis (located at x). The fine scale fringes are modulated in spacing with a wavelength of 3nm.
- Fig. 14 Plot of individual fringe spacings measured from a micro-densitometer trace normal to the fringes in fig. 12.
- Fig. 15 Enlargement of fig. 12 showing that the fringes produced by the 200 lattice planes end at discrete positions (marked $\perp$ here). A typical "Burgers circuit" is indicated, showing closure failure.

REFERENCES

- Anstis, G. R. and Cockayne, D. J. H., 1979, *Acta Cryst.*, A35, 511.
- Cockayne, D. J. H., Parsons, J. R., and Hoelke, C. W., 1971, *Phil. Mag.*, 24, 139.
- Cowley, J. M., 1975, *Diffraction Physics* (North Holland).
- Cowley, J. M., and Iijima, S., 1972, *Z. Naturforsch.*, 27a, 445.
- de Fontaine, D., 1966, *Local Atomic Arrangements Studied by X-Ray Diffraction*, A.I.M.E. Conference Series 36, (Gordon and Breach), p. 51.
- Grinton, G. R., and Cowley, J. M., 1971, *Optik*, 34, 221.
- Gronsky, R., Okada, M., Sinclair, R., and Thomas, G., 1975, 33rd Ann.Proc. Electron Microsc. Soc. Amer., (Baton Rouge, Claiborne's), p. 22.
- Gronsky, R. and Thomas, G., 1977, 35th Ann. Proc. Electron Microsc. Soc. Amer., (Baton Rouge, Claiborne's), p. 116.
- Jenkins, F. A., and White, H. E., 1957, *Fundamentals of Optics* (McGraw Hill).
- Rez, P., 1978, Ninth Int. Congr. on Electron Microscopy, 1, (The Imperial Press), p. 280.
- Sinclair, R., 1979, *Introduction to Analytical Electron Microscopy*, (Plenum Press), p. 507.
- Sinclair, R., Gronsky, R., and Thomas, G., 1976, *Acta Met.*, 24, 789.
- Sinclair, R., and Thomas, G., 1978, *Met. Trans.* 9a, 373.
- Spence, J. C. H., Cowley, J. M., and Gronsky, R., 1979, *J. Ultramicroscopy*, 4, 429.

Fig. 1

Fig. 2a

XBL 798-6736

Fig. 2b

XBL798-6732

Fig. 3

Fig. 4

XBL 798-6735

XBL 797-6611

Fig. 5

Fig. 6

Fig. 7

XBL 797-6613

XBL 798-6734

Fig. 8

XBL 797-6612

Fig. 9

Fig. 10

XBL 797-6610

XBL 797-6609

Fig. 11

Fig. 12

XBB 790 14918

Fig. 13

XBL 757-6648

Fig. 14

Fig. 15

XBB 798 10322