Elastic model for the partially coherent growth of metallic superlattices. I. Interdiffusion, strain, and misfit dislocations

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We have performed a calculation of the elastic energy density of an epitaxial superlattice of known composition profile. We assume an interplay between coherency strains and misfit dislocations as complementary matching mechanisms between consecutive layers of different composition. This model allows us to extract the strain profile and the average distance δ between misfit dislocations at the bilayer interfaces from high-angle x-ray data. An example of the structural characterization of an Mo/V superlattice using this model is given.

I. INTRODUCTION

One of the principal aims in growing metallic superlattices is to create new materials with properties different from those of their constituents or alloys. It is then of crucial importance to characterize the structure of these new materials.[1] One of the best nondestructive tools is x-ray diffraction. Low-angle x rays can provide information on the composition profile of the multilayer while high-angle x rays can give information about strain in the crystal lattice. The strain profile is less easy to extract from x-ray data than the composition profile because at high angles both are interrelated. Thus the primary motivation of this work is to provide a model connecting the known composition profile to the strain in order to extract structural information by fitting the experimental high-angle x-ray diffraction data.

In dealing with sinusoidal composition fluctuations in binary solid solutions, Cahn[2] considered a perfectly coherent deformation of the lattice. Since in a perfectly coherent lattice there are no dislocations, the interplaner spacing normal to the growth direction must take a constant value d, and the total strain is computed using isotropic continuum elasticity theory. This approach yields an elastic energy density (EED) which depends only on the deviation from the average composition, and is independent of the modulation wavelength Λ. This model holds for weak amplitude composition modulation and was successfully used by Philofsky and Hilliard[3] to study the effect of coherency strains on diffusion in modulated binary alloys.

For artificial metallic superlattices with steep composition variations at the bilayer interfaces, the perfectly coherent image is no longer justified. In this work we propose an alternative image of partial coherence in which the chemical modulation produces both coherency strains and misfit dislocations. This is because now there is a large lattice mismatch between the layers, and so it is energetically more favorable for the system to partially relax the strains by forming misfit dislocations in the region of the interfaces.[4] If we consider the superlattice to be an equilibrium state we can determine the strain profile by minimizing the elastic energy density for a given composition profile and density of dislocations. In practice the composition profile is known from low-angle x-ray data and the extremalization procedure provides us with a relationship between strain and dislocations. This relationship allows us to describe the strain-dislocation structure with a unique parameter δ, to be defined below, which can be determined by fitting the high-angle x-ray diffractograms.

The paper is organized as follows. In Sec. II we first describe our model. In Sec. III we compute the local elastic energy density (EED) associated with a planar deformation. In Sec. IV we derive the period average of the EED for a single harmonic composition modulation and determine the equilibrium values of relevant structural parameters. We also discuss two limiting cases: the strongly coherent and the weakly coherent limits. In Sec. V we give an example of the structural characterization of an Mo/V superlattice, and in Sec. VI we conclude this work.

II. THE MODEL

For the completely coherent growth situation considered by Cahn, the film is coherent throughout the plane of the film, i.e., in the x and y directions. As a result the spacing of atoms in the xy plane (in-plane spacing) is a constant in the z direction (growth direction). We assume now that for partially coherent growth the film will be coherent only over a distance δ in the x and y directions. A misfit dislocation will then relax the strains. We therefore have such misfit dislocations at an average distance δ in the xy plane. The degree of coherence is thus described in this model by δ. For $\delta=\infty$ we have completely coherent growth and for $\delta=0$ completely incoherent growth. For partially coherent growth (i.e., δ finite) the inplane lattice spacing will vary in the growth direction and we must consider not only the compressional strain, as in Cahn's model, but also shear. Thus the total elastic energy of such a film is made up of three terms: the compressional energy, the shear energy,

and the core energy of the dislocations. The last term is proportional to the number of dislocations in the film. In this paper we shall consider only the balance between the compressional and the shear energy and how this is related to the composition profile of the multilayer film for a given number of misfit dislocations. For this study we therefore consider δ as an external parameter and we neglect the core energy of the dislocations. The subject of the following paper will be the inclusion of the dislocation core energy to study the equilibrium between the elastic strains and the number of misfit dislocations and thus the transition from a coherent to a partially coherent state.

Specifically, we consider here the total film to be the sum of many films of finite size δ in the x and y directions, each of which grow coherently. Our problem is therefore reduced to calculating the elastic energy of a completely coherent film with finite size δ in the x and y directions.

III. THE LOCAL ELASTIC ENERGY DENSITY

In order to compute the total elastic energy of a strained, chemically modulated structure we must integrate the local contribution over an entire superlattice period. We begin by dividing our film (of finite width δ) into thin slabs of thickness $2l$ in the growth direction, which we take as the z direction. Consider one such slab. When it is connected to the other slabs in the film it will be distorted (see Fig. 1). Let $d_{sf}(z)$ be the stress-free lattice spacing in the undistorted slab and $d_{\perp}(z)$ the inplane lattice spacing in the distorted slab. The inplane strain $\epsilon_{\perp}$ is then

$$\epsilon_{\perp}(z)=\frac{d_{\perp}(z)-d_{sf}(z)}{d_{sf}(z)} . \tag{1}$$

Here we assume that the z dependence within the slabs can be neglected and $d_{\perp}(z)$ and $d_{sf}(z)$ are the values in the middle of the slab. We will explicitly introduce the form of the z dependence for a multilayer film in Sec. IV.

As discussed above the elastic energy will consist of contributions from both normal stresses and shearing stresses. We will compute these two contributions independently and then minimize their sum in order to find the equilibrium value of the elastic energy density. Let u_1 be the contribution of the normal stresses and u_2 that of the distortion. We will compute u_1 first. As in Ref. 2 we assume no tensions in the growth direction z, but isotropic inplane tensions which we write as

$$\sigma_{\parallel}=\sigma_{zz}=0 , \tag{2}$$

$$\sigma_{\perp}=\sigma_{xx}=\sigma_{yy}\neq 0 . \tag{3}$$

Using standard elasticity theory, the strains can be expressed in terms of the normal stresses with the aid of the superposition principle as

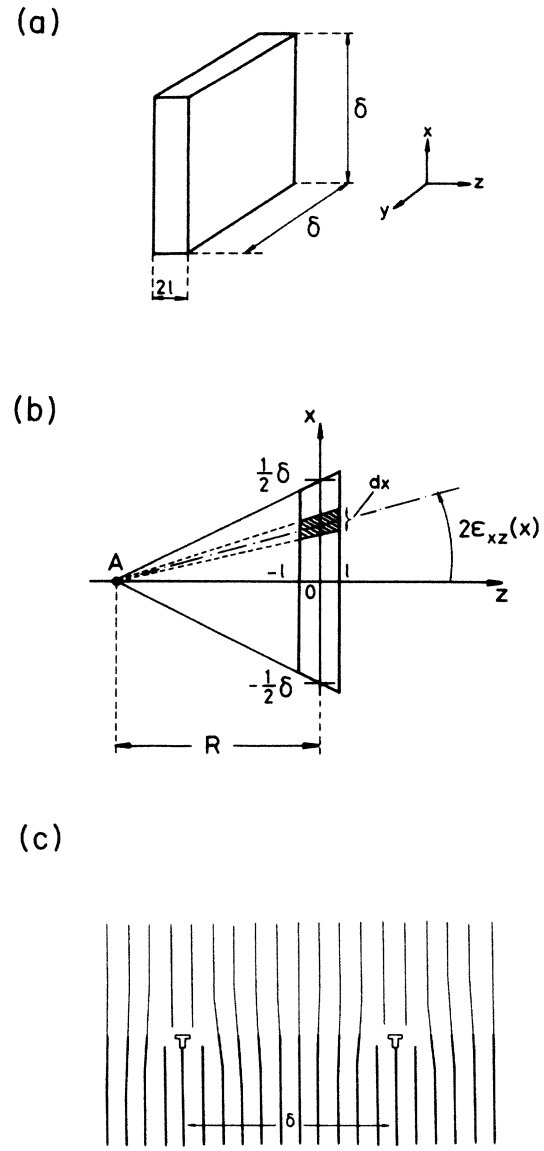

FIG. 1. (a) is an unstrained sheet of elastic material while (b) gives the dimensions of a strained sheet of material of uniform composition used in the calculation of the local elastic energy density. (c) is a schematic representation of misfit dislocations appearing at the interface of two lattice-mismatched materials.

$$\epsilon_{xx}=\frac{1}{E}(\sigma_{xx}-\nu\sigma_{yy}-\nu\sigma_{zz}) ,$$

$$\epsilon_{yy}=\frac{1}{E}(-\nu\sigma_{xx}+\sigma_{yy}-\nu\sigma_{zz}) ,$$

$$\epsilon_{zz}=\frac{1}{E}(-\nu\sigma_{xx}-\nu\sigma_{yy}+\sigma_{zz}) .$$

Then from Eq. (3) we get

$$\epsilon_{\perp}=\frac{1-\nu}{E}\sigma_{\perp} ,$$

$$\epsilon_{\parallel}=\frac{-2\nu}{E}\sigma_{\perp} , \tag{4}$$

where $\epsilon_\perp=\epsilon_{xx}=\epsilon_{yy}$, $\epsilon_\parallel=\epsilon_{zz}$, E is Young's modulus, and ν the Poisson ratio.

The contribution of the normal stresses to the elastic energy density is then

$$u_1(z)=\frac{dU_1(z)}{dV}=\tfrac{1}{2}(\sigma_{xx}\epsilon_{xx}+\sigma_{yy}\epsilon_{yy}+\sigma_{zz}\epsilon_{zz})$$

$$=\frac{E}{1-\nu}\epsilon_\perp^2(z)\ . \tag{5}$$

Since we neglected the z dependence within the plane this contribution is independent of l. A more exact calculation would give a term proportional l^2. In the limit $l\to 0$, the limit we are considering here, this term disappears.

We now consider the angular distortion of the sheet. The shear contribution U_2 can be evaluated by

$$dU_2=(\sigma_{xy}\epsilon_{xy}+\sigma_{yz}\epsilon_{yz}+\sigma_{zx}\epsilon_{zx})dV\ , \tag{6}$$

with

$$\sigma_{ij}=2G\epsilon_{ij}\quad \text{when } i\neq j\ ,$$

where G is the shear modulus of the material and the σ_{ij}'s and the ϵ_{ij}'s are, respectively, the shearing stresses and the shearing components of the strain. For thin slab deformations as in Fig. 1(b), the distortion in the xz plane around a point placed at a distance x from the z axis can be expressed by

$$\epsilon_{xz}(x)=\frac{1}{2}\frac{x}{R}\ .$$

Analogously, in the yz plane

$$\epsilon_{yz}(y)=\frac{1}{2}\frac{y}{R}$$

and $\epsilon_{xy}=0$ since no distortion appears in the xy plane. Noting that $R(z)=d_{\rm sf}(z)/\nabla_z d_\perp(z)$, we can write

$$\epsilon_{xz}(x,z)=\frac{1}{2}\frac{x}{d_{\rm sf}(z)}\nabla_z d_\perp(z)\ ,$$

$$\epsilon_{yz}(y,z)=\tfrac{1}{2}\frac{y}{d_{\rm sf}(z)}\nabla_z d_\perp(z)\ .$$

Thus we can write the shear contribution as

$$dU_2=\tfrac{1}{2}G\frac{(\nabla_z d_\perp)^2}{d_{\rm sf}^2}(x^2+y^2)dV\ ;$$

and integrating over the entire volume of the slab,

$$\frac{U_2}{V}=u_2=\frac{G}{12}\frac{\delta^2}{d_{\rm sf}(z)^2}(\nabla_z d_\perp)^2\ . \tag{7}$$

Thus we can write the total local elastic energy density as follows:

$$u(z)=u_1(z)+u_2(z)=\frac{E}{1-\nu}\epsilon_\perp^2(z)+\tilde{G}[\nabla_z d_\perp(z)]^2\ , \tag{8}$$

where

$$\tilde{G}=\frac{1}{12}\frac{\delta^2}{d_{\rm sf}^2}G\simeq\frac{1}{12}\frac{\delta^2}{d^2}G\ . \tag{9}$$

Here we take $d_{\rm sf}$ in the denominator to be a constant d since it is only a small modulation about an average lattice spacing. Note that the important z dependencies are those of $\epsilon_\perp^2(z)$ and $\nabla_z d_\perp(z)$. The other z dependencies in this expression can be neglected.

IV. THE PERIOD AVERAGE OF THE EED

Any periodic composition modulation can be Fourier analyzed. The orthogonality of Fourier components allows us to find the total elastic energy density by adding the contribution of each independent mode. Thus in this section we will consider a single harmonic composition modulation of period Λ defined by

$$C(z)=\bar{C}[1+\eta\sin(\beta z)] \tag{10}$$

with

$$\beta=2\pi/\Lambda$$

and

$$\eta=\frac{C_{\max}-C_{\min}}{2\bar{C}}\ ,$$

where $C(z)$ is the concentration of one of the constituents and $\bar{C}$ is its average.

The stress-free interplanar spacing is, assuming Vegard's law,[5]

$$d_{\rm sf}(z)=\bar{d}[1+A\sin(\beta z)] \tag{11}$$

where $\bar{d}$ is the average interplanar spacing, $A=2\bar{C}\eta\epsilon_0$, and $\epsilon_0=(d_{\rm I}-d_{\rm II})/(d_{\rm I}+d_{\rm II})$. ϵ_0 is the differential strain between constituents I and II.

The stressed in-plane profile corresponding to Eq. (10) will be

$$d_\perp(z)=\bar{d}[1+\tau_\perp A\sin(\beta z)] \tag{12}$$

where $\tau_\perp$ is called the in-plane deformation parameter. In fact, within this linear elasticity model the elastic deformation affects the modulation amplitude but not the sinusoidal shape of the spacing profile. It can easily be shown that further sinusoidal components in $d_\perp$ would enhance the average elastic energy. Analogously for the growth direction we can write the interplanar spacing profile as

$$d_\parallel(z)=\bar{d}[1+\tau_\parallel A\sin(\beta z)]\ , \tag{13}$$

where $\tau_\parallel$ is related to $\tau_\perp$ according to Eqs. (1), (4), (12), and (13) by

$$\tau_\parallel=1-\frac{2\nu}{1-\nu}(\tau_\perp-1)\ . \tag{14}$$

The quantity $\epsilon_\perp(z)$ appearing in Eq. (8) can be approximately evaluated from Eqs. (11) and (12) as follows:

$$\epsilon_\perp(z)\simeq\frac{d_\perp(z)-d_{\rm sf}(z)}{\bar{d}}=(\tau_\perp-1)A\sin(\beta z)\ . \tag{15}$$

Here again we took $d_{sf} \simeq \bar{d}$ in the denominator since it provides only a small modulation about this average interplanar spacing. Then taking E, ν, and G as constant, the EED period average defined by

$$\bar{u} = \frac{1}{\Lambda} \int_0^{\Lambda} u(z) dz$$

takes the value

$$\bar{u} = \tfrac{1}{2} A^2 \frac{E}{1-\nu} \left[(\tau_\perp - 1)^2 + \frac{1}{24} \frac{1-\nu}{1+\nu} \delta^2 \beta^2 \tau_\perp^2 \right] . \quad (16)$$

At this point it is useful to analyze how the two terms in Eq. (16) will influence the strain in the superlattice. The compressional part is proportional to $(\tau_\perp - 1)^2$ and will be minimal when $\tau_\perp = 1$, i.e., it will want the film to have $d_\perp = d_{sf}(z)$. This is possible in a film of finite size in the x and y directions and means that the width of the film will be modulated. This then means that shear is introduced and indeed the second term is large for $\tau_\perp = 1$. To see how shear comes into the problem it may be useful to consider the coherent growth of one slab of one material on top of a similar slab of another material. If these slabs have finite sizes in the x and y directions, like the ones in Fig. 1, coherent growth must lead to shear. The slab with the larger lattice parameter will be compressed at the interface and the other slab extended at the interface. This is characteristic of a slab of finite size. If the size in the x and y directions is extended to infinity, coherency is only possible if $d_\perp$ becomes constant in the z direction and there will be no shear. The equilibrium state in our problem will therefore be a compromise between the compressional energy and the shear energy.

The equilibrium inplane deformation τ_{eq} is the value of $\tau_\perp$ that minimizes u, i.e.,

$$\tau_{eq} = \left[1 + \frac{1}{24} \frac{1-\nu}{1+\nu} \delta^2 \beta^2 \right]^{-1} . \quad (17)$$

τ_{eq} depends, as expected, on δ and goes to zero as $\delta \to \infty$. In a completely coherent film there is no shear.

We are now able to compute the equilibrium elastic energy density just by replacing $\tau_\perp$ by τ_{eq} in Eq. (16):

$$\bar{u}_{eq} = \tfrac{1}{2} A^2 \frac{E}{1-\nu} \frac{[(1-\nu)/(1+\nu)]\delta^2 \beta^2}{24 + [(1-\nu)/(1+\nu)]\delta^2 \beta^2} . \quad (18)$$

We note that in this model strain is defined by an optimization procedure. Also, in contrast to Ref. 1, the elastic energy is wavelength dependent. This is due to the gradient term in the local energy of Eq. (8). In the limit where the shearlike modulus $\tilde{G}$ is infinite (that is, δ is infinite), we recover, as discussed above, Cahn's result,

$$u_0 = \tfrac{1}{2} A^2 \frac{E}{1-\nu} ,$$

which is consistent with a strained but undistorted lattice [$\tau_\perp = 0$, and thus $d_\perp(z)$ in Eq. (12) takes the constant value $\bar{d}$].

In deriving Eq. (16), we have made two major approximations. The first was to ignore the spatial dependence of the elastic moduli. In principle, chemical modulation must be accompanied by a variation of the elastic properties. By assuming that the elastic moduli which appear in the local energy expression [Eq. (8)] have a sinusoidal variation about a certain average, one can show by parity arguments that the sole nonzero contribution to the integral leading to Eq. (16) comes from this average part. For a nonsinusoidal composition we expect corrections to appear.

The second approximation has to do with the spatial dependence of δ. We expect a high density of dislocations where the composition gradient is large, i.e., at the interfaces. Misfit dislocations at the interfaces ease the strain towards the center of the layers where it then becomes less necessary to introduce them. Nevertheless, there is no contradiction that in our model the basic cell for the elastic energy density calculation is limited transversely by interface dislocations. This is because the δ^2 term in Eq. (8) is modified by the square of the gradient and this becomes important only close to the interfaces. Thus we can define δ^2 in Eq. (16) as the weighted average of a quantity $\delta^2(z)$, given by

$$\delta^2 = \frac{\int dz\, \delta^2(z) [\nabla_z d_\perp(z)]^2}{\int dz [\nabla_z d_\perp(z)]^2} .$$

δ is then an average value of the distance between dislocation lines close to the bilayer interfaces.

We consider two limiting cases: one for $\delta \gg \Lambda$ (a highly coherent structure) and another for $\delta < \Lambda$ (a weakly coherent structure). In the high-coherence limit, the value of the in-plane deformation parameter $\tau_\perp$ tends to zero or equivalently the distance between the misfit dislocations goes to infinity. Consequently, along the growth direction [see Eq. (14)] we have $\tau_\parallel = (1+\nu)/(1-\nu)$. This result is the starting point of Cahn's[2] evaluation of the elastic energy associated with a sinusoidal composition fluctuation which we discussed in the Introduction.

At the other extreme, when the distance between dislocations goes to zero, $\tau_\perp$ goes to 1 and $d_\perp(z)$ just takes on the stress-free interplanar spacing profile of Eq. (11). In the weakly coherent limit, when δ takes on a finite value, the value of $d_\perp(z)$ can assume values between the constant d of perfect coherence and the stress-free value of no coherence. To try to get a better intuitive feel for what is going on we can write for the deformation parameters, by expanding Eq. (17),

$$\tau_\perp = 1 - \frac{1}{24} \frac{1-\nu}{1+\nu} \delta^2 \beta^2 + O(\delta^4 \beta^4) \quad \text{(in plane)} , \quad (19)$$

$$\tau_\parallel = 1 + \frac{1}{12} \frac{\nu}{\nu+1} \delta^2 \beta^2 + O(\delta^4 \beta^4) \quad \text{(growth axis)} . \quad (20)$$

We can use Eq. (20) for the value of $\tau_\parallel$ which appears in Eq. (13). Thus the spacing modulation between planes perpendicular to the growth axis is now described by

$$d_{\parallel}(z) \simeq d_{sf}(z) + \bar{d}\frac{A}{12}\frac{\nu}{1+\nu}\delta^2\beta^2\sin(\beta z) .$$

Recognizing the second derivative of $d_{sf}(z)$ from Eq. (11), we get

$$d_{\parallel}(z) \simeq d_{sf}(z) - \alpha d''_{sf}(z) , \tag{21}$$

where

$$\alpha = \nu/(1+\nu)\delta^2/12 .$$

Thus, in the weakly coherent limit, the actual spacing profile is obtained from the stress-free one by a correction proportional to its local curvature. Equation (21) was derived for a single harmonic modulation, but it obviously holds for any periodic modulation in the weakly coherent limit. The signs in Eqs. (19) and (20) show that the elastic response of the structure to the composition wave leads to a reduction of the local curvature in the profile of interplanar spacings perpendicular to the growth direction. Its corresponding enhancement along the growth axis z costs no energy since z is the stress-free axis of the structure. This fact is highly satisfactory because it fits well with the following intuitive image of epitaxial strain. Consider a region where the composition gradient is uniform. In this region, bonds on a given transversal plane will be equally requested by the two adjacent atomic planes since the mismatch between consecutive stress-free atomic planes follows the composition. A uniform composition gradient does not therefore lead to strain. If the gradient varies locally, the spacing in such a plane will move from its stress-free value in order to reach mechanical equilibrium. In other words, strain comes from curvature in the composition profile.

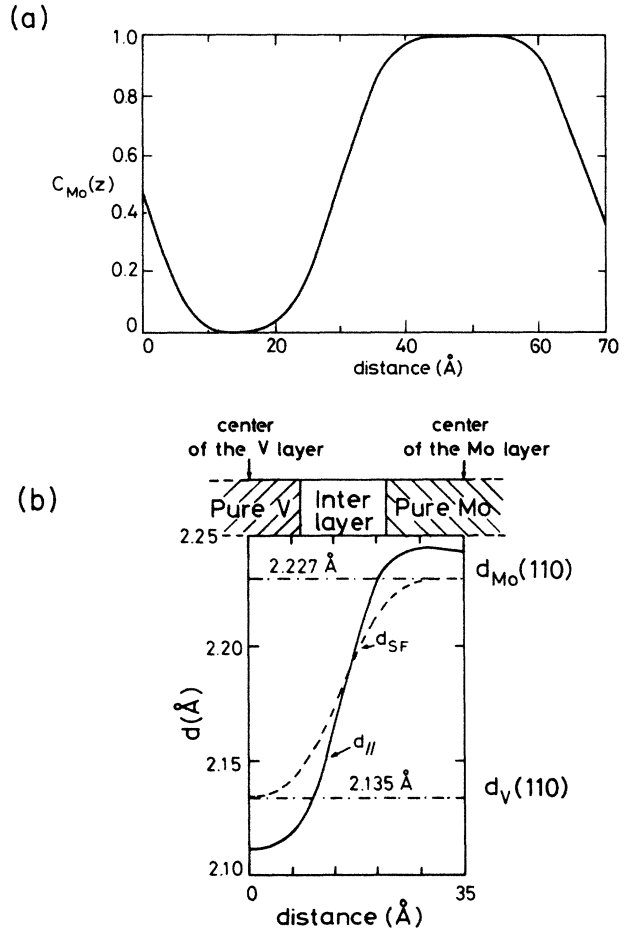

FIG. 2. (a) is the composition profile, reconstructed from low-angle x-ray data, of a 65-Å-wavelength Mo/V superlattice. (b) is the interplanar spacing profile in the region of the interface for the 65-Å Mo/V superlattice. The solid line is the actual spacing profile determined self-consistently from the high-angle x rays while the curved dashed line is the stress-free profile discussed in the text. The bulk interplanar spacing values for Mo (2.227 Å) and V (2.135 Å) are also indicated.

V. CHARACTERIZATION OF A Mo/V SUPERLATTICE

The diffraction of x rays is influenced by the interplanar spacing modulation $d_{\parallel}(z)$ between planes perpendicular to the growth axis. The corresponding deformation parameter in the general case is, from (14) and (17),

$$\tau_{\parallel} = \frac{1+\frac{1}{24}\delta^2\beta^2}{1+\frac{1-\nu}{1+\nu}\frac{1}{24}\delta^2\beta^2} . \tag{22}$$

With (22), we can get the actual amplitude of the spacing modulation for each mode of the stress-free spacing profile of inverse wavelength β. As an example, let us

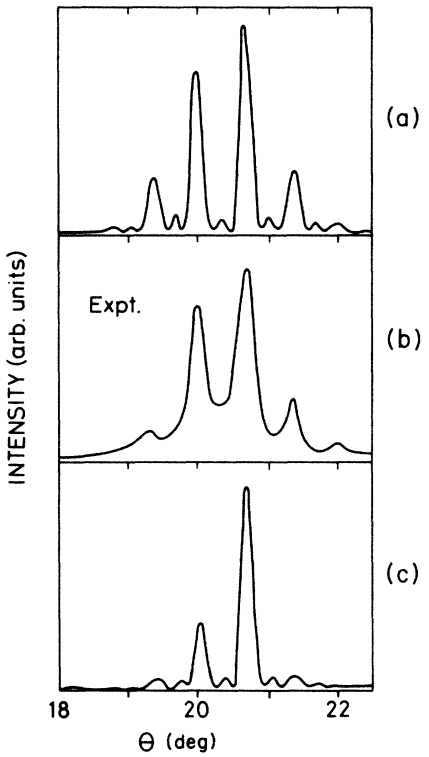

FIG. 3. The high-angle x-ray diffractogram (b) for the 65-Å-wavelength Mo/V superlattice. The best fit to (b), using the information in Fig. 2, is shown in (a). (c) is the fit that results from a stress-free profile.

use this model to characterize a 65-Å period Mo/V superlattice grown on an $Al_2O_3(1120)$ substrate.[6]

The composition profile determined from low angle x rays is shown in Fig. 2(a). The Fourier amplitudes of the composition profile were obtained from low-angle x-ray peak intensities corrected by the Lorentz-polarization and geometric factors. The relative phases were assigned by comparison with a rectangular wave, and the resulting composition wave was obtained from a cosine Fourier series. Coefficients were normalized to give 100% Mo at the origin. The stress-free spacing profile $d_{sf}(z)$ [dotted line in Fig. 2(b)] was obtained from the composition profile using Eq. (11) for each mode. The actual spacing profile was determined self-consistently by reproducing the experimentally determined high-angle x-ray diffractograms (Fig. 3) using the composition profile and the interplaner spacing profile $d_{\parallel}$ which, as we have seen from the minimization of the elastic energy density, depends on δ. Thus the best fit [Fig. 3(a)] to the x rays, obtained by adjusting the density of dislocations δ, gives us $d_{\parallel}$, which in turn enables us to determine the strain profile of the superlattice. The resulting spacing profile is given by the solid line in Fig. 2(b). For this example, the fitting parameter δ was found to be 80 Å and the resulting average stress in the xy plane was $\langle \epsilon_{\perp} \rangle = 7.98 \times 10^{-3}$, while in the center of the V layer it was $\epsilon_{\perp} = 14.5 \times 10^{-3}$. In Fig. 2(c) we also give the x-ray diffractogram calculated using the stress-free spacing profile d_{sf} in order to illustrate the necessity of introducing strain to explain the high-angle x-ray results.

VI. CONCLUSIONS

In this work we have calculated the elastic energy density for a partially coherent superlattice and have obtained a relation between the strain and the density of misfit dislocations. Using this relation and the composition profile obtained from low-angle x-ray diffraction, we have determined the strain in a partially coherent Mo/V superlattice from high-angle x-ray diffraction.

Our model thus allows us to quantify the coherence in the growth of superlattices. The necessity to obtain such information arose from a previous study of Mo/V superlattices, where we found striking anomalies in the transport and superconducting properties as a function of the modulation period. In separate papers[7,8] we report on a detailed study of the Mo/V system where we successfully used the results of this paper to show that the anomalies are related to a transition from incoherent growth to partially coherent growth as the modulation falls below 70 Å.

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