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Dynamical effects in strain measurements by dark-field electron holography

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ABSTRACT

Here, we study the effect of dynamic scattering on the projected geometric phase and strain maps reconstructed using dark-field electron holography (DFEH) for non-uniformly strained crystals. The investigated structure consists of a {SiGe/Si} superlattice grown on a (001)-Si substrate. The three-dimensional strain field within the thin TEM lamella is modelled by the finite element method. The observed projected strain is simulated in two ways by multiplying the strain at each depth in the crystal by a weighting function determined from a recently developed analytical two-beam dynamical theory, and by simply taking the average value. We demonstrate that the experimental results need to be understood in terms of the dynamical theory and good agreement is found between the experimental and simulated results. Discrepancies do remain for certain cases and are likely to be from an imprecision in the actual two-beam diffraction conditions, notably the deviation parameter, and points to limitations in the 2-beam approximation. Finally, a route towards a 3D reconstruction of strain fields is proposed.

1. Introduction

Dark-field electron holography (DFEH) has emerged as a powerful tool for mapping strain at the nanoscale by measuring the geometric phase of diffracted beams [1,2]. The technique has been applied to the study of a variety of semiconductor nanostructures, ranging from strained-silicon devices to multilayers and quantum dots [3–11]. One of the underlying assumptions in these studies was that the strain field observed in projection is a linear average of the strain over the thickness of the foil that the electron beam traverses. However, the 2-beam diffraction conditions predominantly used for obtaining dark-field holograms are inherently dynamical. In a previous paper, we explored how, within dynamical 2-beam theory, the geometric phase propagates through a varying strain field [12]. The result of the dynamical theory is surprisingly simple in this case: the projected in-plane strain is an integral of the strain at different depths in the foil multiplied by a weighting function. This function takes a simple analytical form depending on the diffraction conditions and crystal parameters (extinction distance, deviation parameter and foil thickness).

In this paper, using DFEH experiments and finite element method modelling, we provide evidence for the validity of the "weighting function" theory applied to a strained sample with a well-characterized 3D deformation field. The studied structure consists of a $\{Si_{0.8}Ge_{0.2}/Si\}$ superlattice epitaxially grown on a (001)-Si substrate. For a $Si_{1-x}Ge_x$ alloy with germanium atoms incorporated substitutionally, the lattice parameter is larger than that in silicon. Thus, the substrate compressively strains the lattice in the SiGe layers. Consequently, the SiGe layers are pseudomorphic to the substrate (i.e. not subjected to a plastic relaxation) and have in-plane lattice parameter equal to that of Si and increased lattice parameter in the growth direction due to Poisson reaction. However, DFEH measurements are carried out at a lamella prepared from the bulk structure. There, the thin-film relaxation process taking place in a strained structure induces a two-dimensional strain distribution varying along the growth and perpendicular to the lamella surface directions [13]. Such thin-foil relaxation effects have been studied in detail in systems similar to the present work by Convergent Beam Electron Diffraction (CBED) [14,15] and Geometric Phase Analysis (GPA) of HRTEM images [16,17] combined with strain modelling by the Finite Element Method (FEM). Consequently, we also employ FEM modelling incorporating foil parameters, elastic and compositional properties of the SiGe and Si layers in order to obtain the strain distribution

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within the TEM lamella for comparison with the experimental results.

The organization of the paper is as follows. In Section 2, we will briefly introduce the 2-beam dynamical scattering theory predicting the geometric phase propagation through a sample [12] in a traditional notation [18]. Section 3 contains the description of the sample preparation, DFEH experiments and FEM modelling. Experimental results will be presented in Section 4. The comparison between the results obtained experimentally and the ones obtained by application of the "weighting function" theory to a FEM model will be presented in Section 5.

2. 2-Beam scattering theory

The phase of a diffracted beam (characterized by a diffraction vector $\mathbf{g}$) can be described as the sum of four components [2]

$$\phi_{\mathbf{g}}(\mathbf{r}) = \phi_{\mathbf{g}}^{G}(\mathbf{r}) + \phi_{\mathbf{g}}^{C}(\mathbf{r}) + \phi_{\mathbf{g}}^{M}(\mathbf{r}) + \phi_{\mathbf{g}}^{E}(\mathbf{r}) \tag{1}$$

where G refers to the geometric phase, C the crystalline lattice, M the magnetic contributions, and E the contributions from electric fields. The dynamic scattering theory that we have developed covers the geometric and crystalline phase components [12]. For materials of different compositions, the phase of a diffracted beam will contain an additional term of the electrostatic phase, which depends exclusively on the mean-inner potential (MIP) of the material the e-beam passes through

$$\phi_{MIP}(x) = C_E \int_0^t V_0(x,z)dz \tag{2}$$

To calculate the projected geometric phase from its z (sample depth)-dependent components we have combined the 2-beam dynamical scattering theory with a perturbation expansion [12]. For this, we have introduced an additional "z" dependent geometric phase factor $e^{2i\pi \mathbf{g}\cdot\mathbf{u}(z)}$ in the 2-beam Howie–Whelan (HW) equations of the Fourier components of the potential as shown in the following:

$$\begin{aligned} \frac{d\psi_0(z)}{dz} &= i\pi \frac{e^{i\theta_{-g}}}{\xi_{-g}} e^{2i\pi \mathbf{g}\cdot\mathbf{u}(z)} \psi_g(z) \\ \frac{d\psi_g(z)}{dz} - 2i\pi s_{\mathbf{g}} \psi_g(z) &= i\pi \frac{e^{i\theta_{\mathbf{g}}}}{\xi_g} e^{-2i\pi \mathbf{g}\cdot\mathbf{u}(z)} \psi_0(z) \end{aligned} \tag{3}$$

where ψ_0 and ψ_g respectively correspond to the transmitted and diffracted wave functions, z to the coordinate parallel to the optical axis, $u(z)$ to the displacement field, ξ_g to the dynamical extinction distance associated to the diffracted beam g, $s_{\mathbf{g}}$ to the excitation vector linked to the Bragg angle deviation and θ_g to the phase factor of the Fourier coefficient of the electrostatic potential [18].

Within the validity of the 2-beam approximation, the column approximation, small displacement variation $(d\mathbf{u}(z)/dz) \ll 1$ and neglecting the absorption effect, these coupled equations have been solved by terminating the von-Neumann expansion to the first order yielding a rather simple expression for the reconstructed geometric phase [12]

$$\phi_g = -2\pi \int_0^t \Re\{f_u^g(z)\} \mathbf{g}\cdot\mathbf{u}(z)dz \tag{4}$$

where "t" is the crystalline sample thickness, $\Re$ the real part and $f_u^g(z)$ a weighting function for the displacement projection

$$f_u^g(z,t) = \frac{i\pi(s_{\mathbf{g}} + (1/\xi_g^{eff}))e^{i\pi((t-2z)/\xi_g^{eff})} - i\pi(s_{\mathbf{g}} - (1/\xi_g^{eff}))e^{-i\pi((t-2z)/\xi_g^{eff})}}{e^{i\pi(t/\xi_g^{eff})} - e^{-i\pi(t/\xi_g^{eff})}} \tag{5}$$

with ξ_g^{eff} the effective extinction distance including the deviation from the exact Bragg position

$$\frac{1}{\xi_g^{eff}} = \sqrt{s_{\mathbf{g}}^2 + \frac{1}{\xi_g^{\,2}}} \tag{6}$$

Since absorption can be neglected in this case, we will only use the real part of the weighting function with the following analytical form:

$$\Re\{f_u^g(z)\} = \frac{\pi}{\xi_g^{eff}} \frac{\cos(\pi((t/\xi_g^{eff}) - 2(z/\xi_g^{eff})))}{\sin(\pi(t/\xi_g^{eff}))} \tag{7}$$

The weighting function depends notably on the effective extinction distance, which is directly linked to the choice of the diffracted beam g, deviation parameter and crystalline thickness of the sample. In fact, the weighting function depends on the scaled thickness, defined as a fraction of the effective extinction distance, t/ξ_g^{eff}.

The interpretation of the reconstructed geometric phase is then as follows: the weighted integral is a linear projection rule for the full geometric phase field along the electron beam. The phase of the diffracted beam at the exit surface corresponds to the sum of the geometric phases coming from the different depths in the foil, z, multiplied by the weighting factor. This projection can be applied to all values linearly depending on the reconstructed phase such as the in-plane strain field (described by the strain components ε_{xx}, ε_{yy}, ε_{xy}, and rigid-body displacement ω_{xy}).

A general feature of the weighting function is that

$$\int_0^t f_u^g(z)dz = 1 \tag{8}$$

Therefore, in case of a constant displacement field along z: $\mathbf{u}(z) = \mathbf{u}$, the normalization of the weighting function restores the currently used expression of the 2D reconstructed geometric phase: $\phi_g = -2\pi \mathbf{g}\cdot\mathbf{u}$.

Finally, the phase of the diffracted beam at the exit surface of the sample will be given by

$$\phi^g = \theta_g + \pi s_g t + C_E \int_0^t V_0(y)dz - 2\pi \int_0^t f_u^g(z) \mathbf{g}\cdot\mathbf{u}(z)dz \tag{9}$$

If both reference and strained areas of the sample have similar thickness, composition and crystalline orientation, then the phase difference between the reference and strained areas will be determined by the latter, geometric phase, term. In Section 5.1., we will explore the validity of this assumption for the case examined in this paper.

Table 1 gives the extinction distances and the scaled thickness of three different diffracted beams 111, 004 and 008 of Si which are present in the $[1\bar{1}0]$ zone axis and will be experimentally studied in this paper, employing 200 kV accelerating voltage and a 120 nm-thick lamella. It is worth noting that the most dynamic 111 diffracted beams have a short extinction distance related to sample thicknesses used in electron holography and typically deviate from true 2-beams conditions. This has to be considered

Table 1
Extinction distance ξ_g and ratio t/ξ_g for a 120 nm thick Si sample and an acceleration voltage 200 kV associated to 111, 004 and 008 diffracted beams.

Diffracted beam	111	004	008
ξ_g	79.4 nm	163.8 nm	540 nm
t/ξ_g	1.51	0.74	0.22