Faculty and Staff Publication:

Manasa Medikonda and Gangadhara R. Muthinti

College of Nanoscale Science and Engineering, SUNY

Jody Fronheiser and Vimal Kamineni

GLOBALFOUNDRIES, Albany, New York

Matthew Wormington and Kevin Matney

Jordan Valley Semiconductors Inc., 3913 Todd Lane, Suite 106, Austin, Texas

Thomas N. Adam

College of Nanoscale Science and Engineering, SUNY, New York

Evguenia Karapetrova

Advanced Photon Source, Argonne National Laboratory, 9700S Cass Ave., Argonne, Illinois

Alain C. Diebold

College of Nanoscale Science and Engineering, SUNY, New York

Citation for Article:

Medikonda, M., Muthinti, G. R., Fronheiser, J., Kamineni, V., Wormington, M., Matney, K. . . .
Diebold, A.C. (2014). Measurement of periodicity and strain in arrays of single crystal
silicon and pseudomorphic Si_{1-x}Ge_x/Si fin structures using x-ray reciprocal space maps.
*Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics:
Materials, Processing, Measurement, and Phenomena*, 32(2), 021804.
doi:10.1116/1.4863316

<http://avs.scitation.org/doi/full/10.1116/1.4863316>

Measurement of periodicity and strain in arrays of single crystal silicon and pseudomorphic $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ fin structures using x-ray reciprocal space maps

Manasa Medikonda and Gangadhara R. MuthintiJody Fronheiser and Vimal KamineniMatthew Wormington and Kevin MatneyThomas N. AdamEvguenia KarapetrovaAlain C. Diebold

Citation: *Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena* **32**, 021804 (2014); doi: 10.1116/1.4863316

View online: <http://dx.doi.org/10.1116/1.4863316>

View Table of Contents: <http://avs.scitation.org/toc/jvb/32/2>

Published by the [American Vacuum Society](#)

Instruments for Advanced Science

Contact Hiden Analytical for further details:

W www.HidenAnalytical.com
E info@hiden.co.uk

CLICK TO VIEW our product catalogue

Gas Analysis

- › dynamic measurement of reaction gas streams
- › catalysis and thermal analysis
- › molecular beam studies
- › dissolved species probes
- › fermentation, environmental and ecological studies

Surface Science

- › UHV TPD
- › SIMS
- › end point detection in ion beam etch
- › elemental imaging - surface mapping

Plasma Diagnostics

- › plasma source characterization
- › etch and deposition process reaction
- › kinetic studies
- › analysis of neutral and radical species

Vacuum Analysis

- › partial pressure measurement and control of process gases
- › reactive sputter process control
- › vacuum diagnostics
- › vacuum coating process monitoring

Measurement of periodicity and strain in arrays of single crystal silicon and pseudomorphic $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ fin structures using x-ray reciprocal space maps

Manasa Medikonda^{a)} and Gangadhara R. Muthinti
College of Nanoscale Science and Engineering, SUNY, New York 12203

Jody Fronheiser and Vimal Kamineni
GLOBALFOUNDRIES, Albany, New York 12203

Matthew Wormington and Kevin Matney
Jordan Valley Semiconductors Inc., 3913 Todd Lane, Suite 106, Austin, Texas 78744

Thomas N. Adam
College of Nanoscale Science and Engineering, SUNY, New York 12203

Evguenia Karapetrova
Advanced Photon Source, Argonne National Laboratory, 9700S Cass Ave., Argonne, Illinois 60439

Alain C. Diebold
College of Nanoscale Science and Engineering, SUNY, New York 12203

(Received 9 September 2013; accepted 13 January 2014; published 6 February 2014)

Characterization of the periodicity and strain state of an array of lithographically patterned silicon and silicon-germanium alloy on silicon fins using reciprocal space mapping of Bragg diffraction peaks is presented. Various patterned structures with different pitch values of 90 nm, 65 nm, and 42 nm have been studied and data for the 42 nm pitch sample is discussed in this paper. Diffraction from fin arrays is treated kinematically analogous to periodic surface grating structures. Diffraction from the symmetric 004 planes is used to calculate pitch and analyze the pitch walking pattern which appears as harmonic peaks on either side of the fin peaks. Pitch walking refers to the presence of two periodicities in the array due to the lithographic process. Longitudinal scans are evaluated at the fin peak positions to probe into the shape of the fin structure. Nonrectangular fin shapes resulted in peak splitting of the longitudinal scans of higher order fin peaks indicating a finite sidewall slope. Asymmetric 224 planes were analyzed to study the quality and strain-relaxation of the fin structures both parallel and perpendicular to the fin length using reciprocal space mapping techniques. © 2014 American Vacuum Society. [<http://dx.doi.org/10.1116/1.4863316>]

I. INTRODUCTION

Continued scaling of device sizes to achieve higher performance and/or lower power operation at lower cost is driving new transistor types. Historically, the silicon industry has relied on planar transistors and lithographic scaling to achieve these gains. Although the use of new materials and processes have advanced planar transistor performance, adequate control of the channel by a single gate has become increasingly difficult with each technology node. To this end, the silicon industry has been investigating new transistor types that provide both superior channel control and continued scaling. Nonplanar transistors based on single crystal Si “fins” with the gate wrapped around three sides of the fins have recently moved from research and development into high-volume manufacturing.^{1–3} Based on the International Technology Roadmap for Semiconductors, fin-based field effect transistors are the predicted device type for the next 15 years.⁴ The need for enhanced mobility has motivated research into $\text{Si}_{1-x}\text{Ge}_x$ alloys and III-V materials.⁴ The three-dimensional (3D) nature of the fin structure using these materials presents many challenges for manufacturing including the metrology of feature dimension, pitch, and

stress. There is a need for quick and nondestructive evaluation of these fin structures at each level of fabrication to monitor processes and maintain yield. X-rays have wavelengths comparable to atomic dimensions and can provide sufficient resolution to investigate the unique characteristics of these crystalline nanostructures. Figure 1 illustrates an array of Si fins with an average separation (pitch) P and a defect known as pitch walking that can cause yield loss if not monitored and controlled.⁵ Pitch walking is a pattern where every other fin is closer to the adjacent fin, which can arise from the multiple patterning lithography used to fabricate fins with critical dimensions of $1 \times -2 \times$ nm.

High-resolution x-ray diffraction (HRXRD) has been shown to provide unique capability for nondestructive measurement of the structural parameters of periodic features fabricated in single crystal materials.^{6–8} These parameters include the pitch, shape [height, width, and sidewall angle(s)], strain/stress, and defectivity in the array. However, the application of HRXRD to arrays of fins that are representative of those encountered in $1 \times$ silicon technology nodes has been limited. X-ray metrology is nondestructive and based on first principle quantities (wavelength, angle...) rather than uncertain material parameters and so does not need to be calibrated to provide accurate results. Complementary techniques such as optical critical dimension (OCD) measurement and critical

^{a)}Electronic mail: mmedikonda@albany.edu

FIG. 1. (Color) (a) Schematic illustration of trapezoidal fins with an overlay error resulting in an effective period of $2P$. (b) When the x-ray beam is perpendicular to the fin length, cross pattern of diffracted intensity in reciprocal space is characteristic of a trapezoidal fin shape.

dimension scanning electron microscopy (CD-SEM) measure pitch and shape. CD-SEM is evidently known to have sample charging effect and device structure damage problems. Reciprocal space maps (RSM) provide crucial information on particular set of lattice planes and RSM characterization of stress state and pitch walking can help guide OCD optical modeling.⁹

In this paper, we demonstrate the use of HRXRD scans and maps in reciprocal space to determine the pitch, pitch walking, and strain/stress state in periodic arrays of single crystal semiconductor fins. Section II gives an overview of the kinematic theory of x-ray diffraction from periodic arrays of fins including the origin of diffraction based observation of pitch walking. In Sec. III, the samples studied are described as are the experimental procedure used for the HRXRD measurements. In Sec. IV, the experimental results are presented including the observation of pitch, pitch walking, and stress in the SiGe layer of Si_{1-x}Ge_x/Si fin structures. In Sec. V, inferences from experimental measurements are presented.

II. THEORY

As will be shown, a periodic fin array will give rise to satellite diffraction peaks in addition to the Bragg peaks due to periodic arrangement of atoms within the crystalline material. These additional peaks measured by HRXRD scans and maps in reciprocal space provides a means of characterizing periodicity and changes in the strain/stress state of epitaxial layers on top of the fins. As introduced above, the term “pitch” defines periodicity of the fin array, and the term “pitch walking” relates to the additional periodicity induced due to overlay errors in fin arrays that were lithographically patterned using a multiple patterning process. In this

section, the theory behind these additional diffraction peaks is presented.

The theoretical basis for the x-ray diffraction from patterned nanostructures has been developed by a number of authors and in this work we follow the treatment by Pietsch, Holy, and Baumbach.¹⁰ The fin array is treated as lateral surface grating on a single crystal substrate and can be approximated using kinematical and semikinematical theory whereas the substrate diffraction can be treated dynamically. Since the vertical dimensions of the fin structures are typically a few tens of nanometers, which is much smaller than the extinction depth of the x-ray beam, then the kinematic theory is sufficient and higher order approximations are not necessary. The lithographically patterned structures have a regular 2D periodicity across a very large, flat area compared to the size of an individual fin, the magnitude of the polarizability $\chi(\mathbf{r})$ repeats itself with the grating period $\mathbf{P}_G$ as follows:

$$\chi(\mathbf{r}) = \chi(\mathbf{r} + \mathbf{P}_G). \quad (1)$$

Due to the periodicity, we can express the polarizability $\chi(\mathbf{r})$ of the fin array (grating) as a Fourier series in terms of reciprocal space vectors $\mathbf{G}$

$$\chi(\mathbf{r}) = \sum_{\mathbf{G}} \chi_{\mathbf{G}}(\mathbf{r}) e^{i\mathbf{G} \cdot \mathbf{r}}, \quad (2)$$

where the vector $\mathbf{r}$ represents the position of each fin and,

$$\mathbf{G} = \hat{\mathbf{x}}G_x + \hat{\mathbf{y}}G_y = \hat{\mathbf{x}}(2\pi m/P_x) + \hat{\mathbf{y}}(2\pi n/P_y). \quad (3)$$

The x - y coordinates (surface plane) are oriented perpendicular and parallel to the patterned structure, respectively, and z is normal to the surface plane. The components of $\mathbf{P}$ and $\mathbf{G}$ in the surface plane are denoted by subscripts and m and n are integers.

We can also write the polarizability as a Fourier series in terms of averaged reciprocal space lattice vectors $\mathbf{g}$

$$\chi(\mathbf{r}) = \sum_{\mathbf{g}} \tilde{\chi}_{\mathbf{g}}(\mathbf{r}) e^{i\mathbf{g} \cdot \mathbf{r}}. \quad (4)$$

Unlike an infinite crystal, the Fourier components $\tilde{\chi}_{\mathbf{g}}(\mathbf{r})$ depend on the position $\mathbf{r}$ because of the grating and so the components can also be expressed as a Fourier series in terms of fin array (grating) reciprocal space vectors $\mathbf{H}$ that belong to the in-plane component of $\mathbf{g}$

$$\tilde{\chi}_{\mathbf{g}}(\mathbf{r}) = \sum_{\mathbf{H}} \tilde{\chi}_{\mathbf{gH}}(z) e^{i\mathbf{H} \cdot \mathbf{r}}. \quad (5)$$

Further description of the vectors used above is useful for understanding Eqs. (1)–(5). $\mathbf{G}$ and $\mathbf{H}$ are both reciprocal grating vectors, while $\mathbf{g}$ and $\mathbf{h}$ are both reciprocal lattice vectors. However, $\mathbf{g}$ is general, while $\mathbf{h}$ corresponds to the particular reflection considered in the two-beam approximation, e.g., 004. $\mathbf{G}$ is general and $\mathbf{H}$ is associated with a particular satellite of a reciprocal lattice vector, so $\mathbf{G} = \mathbf{h} + \mathbf{H}$. In the

kinematical and semikinematical theories, they express the reciprocal grating vector for a reflection in terms of the vector offset of a satellite from the nearest lattice vector $\mathbf{h}$.

In our work, the fins are very long (mm) relative to their width (nm), and so we observe diffraction peaks form a structure with 1D periodicity in the x -direction. Furthermore, since this periodicity is much larger than the distances between atoms in the crystalline lattice, the diffracted intensity will only be significant in the vicinity of the reciprocal lattice points (Bragg peaks) from the crystalline lattice. Thus, the effects of multiple reciprocal lattice points can be ignored, and we may use the two beam approximation in which only the wavefields from the direct beam and one diffracted beam $\mathbf{h}$ are considered. Thus, in the two beam approximation, Eq. (4) can be replaced by $\chi(\mathbf{r}) = \tilde{\chi}_{0\mathbf{H}}(\mathbf{r}) + \tilde{\chi}_{\mathbf{hH}}(\mathbf{r})$ and specific examples of $\mathbf{h}$ include the 004 or 224 diffracted beams.

In the kinematic theory, the amplitude of the scattered wave $A(\mathbf{Q})$ is given by the Fourier transform of the polarizability as a function of the scattering vector $\mathbf{Q}$

$$A(\mathbf{Q}) = 4\pi^2 \int \chi(\mathbf{r}) e^{-i\mathbf{Q}\cdot\mathbf{r}} d\mathbf{r} \\ = \int [\tilde{\chi}_{0\mathbf{H}}(\mathbf{r}) + \tilde{\chi}_{\mathbf{hH}}(\mathbf{r}) e^{i\mathbf{h}\cdot\mathbf{r}}] e^{-i\mathbf{Q}\cdot\mathbf{r}} d\mathbf{r}. \quad (6)$$

Here, the first contribution is from the direct beam and the second contribution is from a single diffracted beam with the specific reciprocal lattice vector $\mathbf{h}$. After some manipulation, these contributions can be written in the following form:

$$A_0(\mathbf{Q}) = 4\pi^2 \sum_{\mathbf{H}} \delta(\mathbf{Q}_{\parallel} - \mathbf{H}) \int \tilde{\chi}_{0\mathbf{H}}(z) e^{-iQ_z z} dz, \quad (7a)$$

$$A_{\mathbf{h}}(\mathbf{Q}) = 4\pi^2 \sum_{\mathbf{H}} \delta(\mathbf{Q}_{\parallel} - \mathbf{h}_{\parallel} - \mathbf{H}) \int \tilde{\chi}_{\mathbf{hH}}(z) e^{-i(Q_z - h_z)z} dz. \quad (7b)$$

The diffracted amplitude from the fin array $A_{\mathbf{h}}(\mathbf{Q})$ is characterized by periodic fine structure in the vicinity of the reciprocal lattice points. The fine structure, known as “grating truncation rods” (GTRs), is parallel to the Q_z direction (perpendicular to the mean surface) and in the (Q_x, Q_y) plane is located according to the vector relation

$$\mathbf{Q}_{\parallel} = \mathbf{h}_{\parallel} + \mathbf{H}. \quad (8)$$

Thus, for our 1D array of fins, the GTRs are perpendicular to the sample surface and are equally spaced in the direction parallel to the fin width. As we shall see later, the GTRs are the origin of extra satellite peaks in $Q_x = [110]$ direction from the fins in transverse scans (sample scans in angle space). The substrate and satellite peaks are extended in the $Q_z = [001]$ due direction due to the height of the fins h and flatness of the substrate.

The amplitude of the diffracted wave $A_{\mathbf{h}}(\mathbf{Q})$ can be conveniently written as the product of two terms

$$A_{\mathbf{h}}(\mathbf{Q}) = S(\mathbf{Q})F(\mathbf{Q}), \quad (9)$$

where $S(\mathbf{Q}) = 4\pi^2 \sum_{\mathbf{H}} \delta(\mathbf{Q}_{\parallel} - \mathbf{h}_{\parallel} - \mathbf{H})$ is known as the structure factor and depends on the arrangement of the fins in the array. In practice, the GTRs are not delta functions but instead have finite width largely determined by the lateral coherence length of the x-ray beam and is normally modeled as a Gaussian functions of the appropriate width. Finally, the intensity is obtained from the diffraction amplitude according to

$$I_{\mathbf{h}}(\mathbf{Q}) = |A_{\mathbf{h}}(\mathbf{Q})|^2 = |S(\mathbf{Q})|^2 |F(\mathbf{Q})|^2. \quad (10)$$

Until now, we have considered a grating with a single pitch $P = P_x$, but when there is an overlay like error in lithography processes, for example, due to the critical dimension variation in spacer double patterning of fin structures, it manifests in a fin array with a pitch walking pattern. The pitch walking pattern can be treated as two interleaved arrays each with a pitch $2P$ and offset by $P + \Delta P$ where ΔP is the magnitude of the pitch walk error. The structure factor of the pitch walking pattern is then given by the sum of the structure factors from the two offset arrays taking into account the phase difference between them, such that

$$S(Q_x) = S_1(Q_x) + S_2(Q_x), \quad (11)$$

where

$$S_1(Q_x) \sim \sum_n e^{iQ_x 2nP} \text{ and } S_2(Q_x) \sim S_1(Q_x) e^{iQ_x (P + \Delta P)}. \quad (12)$$

After some manipulation, the structure factor contribution to diffracted intensity distribution is

$$|S(Q_x)|^2 = |S_1(Q_x)|^2 \{1 + \cos[Q_x(P + \Delta P)]\} / 2. \quad (13)$$

The effect of the pitch walk error ΔP gives rise to additional truncation rods with half the spacing in reciprocal space, due to the periodicity of $2P$ for the pattern. In the absence of pitch walking, $\Delta P = 0$ and the additional rods vanish as the periodicity of the pattern is again equal to that of a single array with pitch P . The effect of the pitch walking error results in an interference effect between the two gratings as expressed by the cosine term in Eq. (13).

The function $F(\mathbf{Q})$ is known as the form factor, which depends on the z -dependent Fourier components of the shape, composition, and strain profile in the fins. This form factor describes the amplitude of each GTR according to

$$F(\mathbf{Q}) = \int \tilde{\chi}_{\mathbf{hH}}(z) e^{-i(Q_z - h_z)z} dz. \quad (14)$$

For different lithography and etch processes, there is a possibility of achieving a variety of fin structures. For example, the fins in this study could be described as having a trapezoidal or triangular cross-section with a rounded top. The form factor for a strain free trapezoidal fin of width w , height h , and sidewall angle α is given by the expression

$$F(Q_x, Q_z) = \frac{F_h}{\sqrt{2\pi}} \left\{ \frac{e^{-iQ_x w/2} [e^{-ih(Q_z - Q_x \alpha)} - 1]}{Q_x(\alpha Q_x - Q_z)} + \frac{e^{iQ_x w/2} [e^{-ih(Q_z + Q_x \alpha)} - 1]}{Q_x(\alpha Q_x + Q_z)} \right\}, \quad (15)$$

where F_h is the structure factor for a unit cell of the crystal material for the reflection h . It should be apparent that the fin width will also impact the RSM diffraction pattern. For large fin arrays with many fins in the measurement area, the observed intensity depends on the relationship between pitch P and fin width w .¹¹ When $P = 2w$, the even order GTRs from a perfect array of rectangular fins are missing. When $P = nw$ and n is an integer, all n -th order peaks are missing. If n is not an integer, all order peaks are observed, but the intensity of the diffraction orders modulated by a function that depends on w . The finite sidewall angle α will also impact the RSM diffraction pattern by progressively splitting the main maxima of the GTRs. This splitting increases linearly with the order of the rod and forms a cross pattern of scattered intensity in the reciprocal space maps as shown schematically in Fig. 1.^{10,12} Further analysis is required before making conclusions about the detailed fin shape on the diffraction patterns reported here. This work is the subject of ongoing investigation.

III. EXPERIMENT

Silicon fins of various widths were patterned using optical lithography and reactive ion etching of 300 mm p-type (1–10 Ω -cm) silicon (100) wafers. SiGe fins were patterned by using blanket pseudomorphic $\text{Si}_{1-x}\text{Ge}_x$ films were grown on p-type (1–10 Ω cm) Silicon (100) wafers in an AMAT Centura 300 mm reduce pressure chemical vapor deposition epi reactor. Intrinsic SiGe films of 25% and 50% germanium concentrations were grown with a mixture of silane and germane at temperatures around 700°C and 450°C, respectively. The wafers were chemically cleaned for metallic contamination with subsequent native oxide etching before loading into the epitaxial growth tool. Prior to film deposition, the wafers were cleaned using an *in-situ* hydrogen bake at 1050°C to remove any additional contamination and native oxide. Fins of various widths of 90, 65, and 42 nm were patterned using lithography and reactive ion etching. Pattern sizes of several mm in dimensions were generated to obtain signals on the BedeMatrixL (BML). The fins are etched through the entire SiGe films to a depth of ~60 nm for all samples. A schematic illustration of the patterned silicon and SiGe fin arrays is shown in Fig. 2. The 3D fin gratings were several millimeters in spatial dimension to obtain the diffraction signal from x-ray methods.

The data presented here is obtained from three x-ray diffraction setups. A BML, Jordan Valley QC3 were used for lab-based measurements and as well as synchrotron measurements performed using Sector 33BM of the Advanced Photon Source (APS) at Argonne National Labs. Both the BML and QC3 tools are high resolution x-ray diffractometers capable of analyzing various parameters on multilayered

Fig. 2. (Color) (a) Schematic illustration of silicon fins and (b) SiGe fins samples depicting an overlay error.

structures. The tools are fitted with 2.2 kW Cu sealed tube sources, channel-cut collimator, and analyzer crystals and provide an x-ray beam a few millimeters in size that is set with mechanical slits. The QC3 is modern tool that provides higher intensity about 5×10^6 cps from Si(004) and higher resolution than the BML tool that is now more than 10 years old. The BML tool has an angular resolution of 25" and QC3 about 20". QC3 provides much quicker measurements in a time scale of 60 min for one set of scans compared to BML which requires a couple of days. The experimental station at Sector 33BM of the APS has a Huber 4-circle diffractometer and was fitted with an advanced 2D Pilatus 100 K detector. The imaging capability of the detector was used extensively for alignments, but measurements were done in 0D mode by integrating the intensity in a region of interest in the detector images. The extremely bright storage ring generated X-rays are useful by providing the highest quality data (10^{10} cps) for the in-depth study on these nanostructures. Measurements were done using a bending magnet beamline with a beam energy of 10 keV using Si(111) monochromator with a spot-size of 1 mm $\times$ 0.5 mm at the focal point of the beam.

The pitch P of the fins can be calculated from rocking-curves obtained by varying the incidence angle ω at a fixed detector angle 2θ with the x-ray beam direction perpendicular to the fins. The diffraction from the fins appears as satellite peaks at either side of the substrate diffraction peak, and the angular separation between the fin peaks is proportional to the pitch. The average separation between several satellite peaks can be used to give a more accurate result if present. It should be noted that the measurement of fin pitch does not require a very accurate alignment of the x-ray beam perpendicular to the length of the fins. The apparent pitch P' as a function of azimuth α with respect to the line direction varies as $P' = P \cos \alpha$ and so an error of $\pm 5^\circ$ will only result in a relative error of $\pm 0.5\%$ in the pitch. Pitch walking, or

overlay error, can be identified as additional harmonic peaks at either side of the fin peaks corresponding to a periodicity of 2P. Double crystal diffractometry with an open detector integrates the intensity over a large section of reciprocal space.⁷ Increasing the resolution of the detector by adding an analyzer crystal, i.e., triple-crystal diffractometry, notably decreases the detected angular range and allows the detection of pitch walking if exists in a sample using ω rocking-curves.

Information of layers in the samples can be extracted from so-called ω - 2θ scans of a symmetric reflection by varying the incident and diffraction angles in 1:2 ratio. Quality of the crystal can be assessed from the (thickness) fringes formed due to interference of x-rays from the interfaces between the layers in the structure, which can be seen only for high quality crystals with smooth and pseudomorphic layers.¹³⁻¹⁶ When the in-plane lattice of the epilayer is coherent with the lattice of the substrate (pseudomorphic), then ω - 2θ scans allow the determination of layer composition and film thickness. The angular spacing of the interference fringes is used to calculate film thickness, and the angular separation between the layer and substrate Bragg diffraction peaks is used to determine alloy strain and composition. ω - 2θ scans were measured from both symmetric (004) lattice planes, which are parallel to the surface and glancing exit asymmetric (224) planes that are inclined at an angle of 35.3° to the surface. The presence of sidewall angle in the fins can also be observed from 004 ω - 2θ scans on higher order fin peaks. The use of RSMs for the characterization of epitaxial layer quality is well known.¹³⁻¹⁵ The RSMs combine ω - 2θ coupled rocking scans and ω rocking scans to yield a complete picture of the scattering from selected planes in the sample. RSMs provide confirmation of the pseudomorphic nature of epitaxial layers such as Si_{1-x}Ge_x on Si (001) and provide information on tilts, surface roughness, and bulk defects, which appear as diffuse scatter.^{13-15,17} Tilt of the layer with respect to the substrate can be determined by plotting a reciprocal space map of the (004) planes. Tilted and untilted reciprocal lattice points of (004) planes lie on different crystal (h,k) or reciprocal (Q_x, Q_z) coordinates of x-axis and the position of the layer peak determines the amount of tilt in the sample. Reciprocal space maps of asymmetric 224 planes are used to determine lattice parameter of the epilayer (deformation with respect to the Si reference lattice parameter) in directions parallel and perpendicular to fin arrays in the case of Si_(1-x)Ge_x fins by changing the azimuthal angle and orienting the beam in that particular direction. If the composition is assumed or known from a complementary measurement, then the strain/relaxation parameters of the epilayer material can be determined.

IV. RESULTS

This section presents data collected from both silicon and silicon-germanium fin samples. Samples containing various lithographically patterned fin structures with pitch values of 42 nm, 65 nm, and 90 nm are studied for their characteristic signature using x-rays. Data presented here are from the pitch 42 nm set of samples. Data are collected in two

directions with the incoming x-ray beam perpendicular to fin length and with the beam parallel to fin length to study the structure in both directions. The fin structures will diffract when the x-ray beam is perpendicular to fin length and the satellite diffraction peaks from which the pitch can be determined can be prominently observed. When the sample is set to rock along H- or K-axis of the (hkl) planes by slightly changing the sample ω angle with fixed 2θ position, the reciprocal lattice point of those particular planes is scanned horizontally and the scans are equivalently called transverse scans H-, K-scans (HKL units), or Q_x-scans (reciprocal space units) or ω -scans (in angular units). A detector 2θ -scan samples the reciprocal space radially along the Ewald sphere. To sample reciprocal space in the longitudinal direction about reciprocal lattice points then equivalently an ω - 2θ coupled scan (angular units), Q_z scan (reciprocal space units) or an L-scan is used since they scan along the L-axis of the (hkl) planes. The angular, HKL and reciprocal space coordinates can be interchanged using the following geometric transformations:

$$Q_x = \frac{2}{\lambda} \sin \theta \sin(\theta - \omega) = \frac{2}{\lambda} a_{||} \sqrt{h^2 + k^2}, \quad (16)$$

$$Q_z = \frac{2}{\lambda} \sin \theta \cos(\theta - \omega) = \frac{2}{\lambda} a_{\perp} l. \quad (17)$$

Pitch can be calculated using^{10,18}

$$\begin{aligned} \text{Pitch} &= \frac{\lambda}{2 * \Delta\omega * \sin \theta} = \frac{2\pi}{Q_z * \Delta\omega_{\text{rod}}} = \frac{2\pi}{\Delta q_x} \\ &= \frac{0.5431 \text{ nm}}{\sqrt{2} * \Delta H}. \end{aligned} \quad (18)$$

Here, λ is the wavelength of the incoming x-ray beam, $\Delta\omega$ is the angular separation between the fundamental satellite peaks, and θ is the Bragg angle of diffracting planes. ΔH represents the difference between the substrate and fin peaks in HKL coordinates.

Figure 3 shows symmetric transverse scans in the vicinity of the 004 Bragg peak from an array of fins when the incoming x-ray beam is perpendicular to the fin length. Transverse scans were used to determine pitch from the angular separation between adjacent fundamental satellite peaks using Eq. (18), also verified using reciprocal space maps plotted in HKL units from Eq. (18). Figure 3(a) shows an ω -scan from a sample with silicon fins measured using the BML tool, which has been subjected to an overlay error during patterning stage. The pitch calculated using Eq. (18) resulted in a normal pitch value of 41.9 ± 0.5 nm from the higher intensity (fundamental) satellite peaks around ± 665 arcsecs and an effective pitch value of 84.3 ± 1 nm from the lower intensity (harmonic) peaks around ± 330 arcsecs. The intensity of the pitch walking harmonics measured using the BML tool is very low and was unable to see any pitch walking signature from SiGe/Si fins. Higher intensity measurements done using the QC3 and APS tools were used to analyze the SiGe/Si fin samples. Data taken using QC3 tool on Si_(1-x)Ge_x

Fig. 3. (Color) Transverse scans (TS) on 004 planes are shown for three fin arrays: silicon, $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$)/Si, and $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$)/Si. This data demonstrates the increase in signal intensity for the three measurement systems as follows $\text{BML} < \text{QC3} < \text{APS}$. (a) The TS for the silicon fin array while (c) and (e) show the TS for the silicon fin part of the $\text{Si}_{0.75}\text{Ge}_{0.25}$ /Si fin array measured using the QC3 and APS, respectively. (b) and (d) The TS for the SiGe epilayer part of the $\text{Si}_{0.75}\text{Ge}_{0.25}$ /Si array. Pitch-walking peaks are clearly present for the three samples. (g) and (i) The TS from the Si part of the $\text{Si}_{0.5}\text{Ge}_{0.5}$ /Si fin arrays using the QC3 and APS, respectively. (f) and (h) The TS from the epilayer part of the $\text{Si}_{0.5}\text{Ge}_{0.5}$ /Si fin arrays using the QC3 and APS, respectively. The synchrotron data shows many orders of diffraction peaks, indicating a high degree of periodicity in these structures. The main fin rods on the right are indicated as +1, +2, etc., and the ones on the left are indicated -1, -2, etc., in (i).

($x=0.25$) and $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) fins could distinguish pitch walking harmonics in the transverse scans through the silicon diffraction peak but the SiGe fin peaks were broad with insufficient resolution to separate out the pitch walking harmonics. Figures 3(b) and 3(c) show fin peaks on SiGe layer and silicon layer of a $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$) sample measured using the QC3 tool, the latter showing substrate peak, satellites from the fin pitch, and pitch walking harmonics. Figures 3(d) and 3(e) show K transverse scans on the same SiGe sample using the APS. Here, because of the high intensity source and increased resolution, we can clearly observe the pitch walking harmonics even on the SiGe layer scan and the peak positions exactly match that of the silicon layer peaks giving a normal pitch $P - \Delta P$ of 43.9 ± 0.5 nm and an effective 2P of 83 ± 1 nm. Figures 3(f) and 3(g) show QC3 scans through SiGe layer and silicon layer of $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) sample with the silicon layer plot displaying the substrate peak and pitch walking harmonics in addition to the fundamental satellite peaks. The SiGe layer fin peaks of the $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) sample measured on

Fig. 4. (Color) Reciprocal space maps in the vicinity of the (a) 004 and (b) 224 Bragg peak from Si for the $\text{Si}_{0.75}\text{Ge}_{0.25}$ /Si fin array. Transverse scans through the (c) $\text{Si}_{0.75}\text{Ge}_{0.25}$ layer and (d) Si layer from the same sample. All measurements were obtained using the BML.

the QC3 were also broad masking out the pitch walking harmonics. APS data on $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) sample shown in Fig. 3(h) displays pitch walking harmonics even on the SiGe layer scan and Fig. 3(i) plots the silicon layer diffraction peaks giving a pitch ($P - \Delta P$) value of 41.5 ± 0.5 nm and a 2P value of 84.51 ± 1 nm. The APS data exhibits higher order fin peaks labeled as +1, +2 for the fin peaks on the right and as -1, -2 for the fin peaks on the left as shown in

Fig. 5. (Color) (a) 004 ω_{Rel} line scan of the silicon layer of $\text{Si}_{0.5}\text{Ge}_{0.5}$ /Si fin array. Reciprocal space maps in the vicinity of (b) 004 Bragg peak displaying silicon and $\text{Si}_{0.5}\text{Ge}_{0.5}$ layer fin peaks. RSM of 224 planes (c) in the direction perpendicular to the fin length and (d) in the direction parallel to the fin length of $\text{Si}_{0.5}\text{Ge}_{0.5}$ array. All measurements are obtained using BML tool.

Figs. 3(d), 3(e), 3(h), and 3(i), where longitudinal scans along these peaks gives further useful information about the fin structure and will be discussed later. The pitch walking peaks that occur half way between these fin peaks are labeled as +0.5, +1.5, and +2.5 for the ones that appear on the right side of the main fin peaks and -0.5, -1.5 and -2.5 for the ones that appear on the left side of the main fin peaks.

Analysis of changes in strain/relaxation of the material is necessary after every step of the fin deposition and subsequent processing. Reciprocal space maps provide a detailed picture of the crystal lattice and allow us to probe into the quality of the sample. Reciprocal space maps on blanket $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$)/Si and $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$)/Si samples indicate high quality pseudomorphic SiGe layers on Si substrate with no trace of relaxation in the layers.¹⁹ Figure 4(a) shows an 004 RSM of the $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$)/Si fin sample displaying fin peaks around both the substrate and layer peaks. An RSM of the asymmetric 224 planes with the incoming x-ray beam parallel to fin length plotted in Fig. 4(b) displays SiGe layer peak on the same H-coordinate

value as that of silicon indicating that the SiGe fins are strained along the fin length. Figures 4(c) and 4(d) show transverse scans of 004 planes through the SiGe and silicon layers, respectively. The BML data for the $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$)/Si fin sample are shown in Fig. 5. Figure 5(a) shows a transverse scan through the 004 Si peak, and Fig. 5(b) displays an 004 RSM with the x-ray beam perpendicular to the fin length. The $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$)/Si sample displays broader fin peaks due to higher defect density or surface roughness compared to $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$)/Si fin sample. Figures 5(c) and 5(d) are asymmetric 224 reciprocal space maps with the x-ray beam perpendicular and parallel to the fin length, respectively. These asymmetric RSMs indicate that the 224 planes in the SiGe layer in the direction perpendicular to the fin length are at a different H coordinate, corresponding to a larger lattice parameter, compared to the silicon layer. Parallel to the fin length, the SiGe layer again shows strain with reference to the silicon layer indicating uniaxial strain in the sample. Figure 6 shows HK-maps of the 113 and 224 reflections done at the APS and these data

FIG. 6. (Color) 113 Reciprocal space maps of $\text{Si}_{0.5}\text{Ge}_{0.5}$ /Si fin array (a) in the direction parallel to fin length and (b) in the direction perpendicular to fin length. 224 RSMs of the same sample (c) in the direction parallel to fin length and (d) in the direction perpendicular to the fin length obtained using Advanced photon source at ANL.

are consistent with the BML measurements showing uniaxial strain in the epilayer—the SiGe lattice parameter being equal to that of the underlying Si, in the direction parallel to the fins and larger than the Si lattice parameter in the direction perpendicular to the fin length. The RSMs in Fig. 6 in the HK- plane depict the SiGe layer peaks and are analyzed taking 113 and 224 planes of silicon as a reference. Figure 6(a) shows an RSM of 113 planes of $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) layer in the direction parallel to the fin length. The peak center is at the $H=K=1$ coordinates, which are set in reference to the 113 plane of silicon and indicates that the fins are strained along the fin length. Figure 6(b) shows a 113 RSM for the same sample in the direction perpendicular to the fin length where the SiGe peak is not at the $H=K=1$ reference of the silicon peak, indicating a relaxed layer. Figures 6(c) and 6(d) show a similar situation using 224 planes where the silicon layer is referenced at $H=K=2$ coordinates. It should be noted that in long, narrow structure, this lattice relaxation can take place elastically due to lateral expansion of lattice without the creation of misfit dislocations that happens in blanket wafers or test pads.

Figure 7 show 004 longitudinal scans through the substrate peak and higher order fin peaks that are present in Fig. 3. Probing along these higher order fin rods using longitudinal scans provide insight into the shape of the fin structures. Due to the shape function introduced into the form factor of these grating rods, the sidewall slope has an effect on the scattered intensity. A peak splitting of the Si peak in higher order fin rods is observed in both the QC3 and APS data and is due to a finite sidewall slope of trapezoidal shape of the fins, displayed by all the samples. Using high intensity sources along with triple-axis detector settings, this peak split can be observed as a characteristic cross pattern in symmetric reciprocal space maps as shown in the illustration of Fig. 1. The average sidewall angle (SWA) can be estimated from RSMs or longitudinal scans through high-order fin peaks using the simple relation

$$\text{SWA} = \frac{1}{2} \frac{\Delta L}{\Delta H}. \quad (19)$$

Here, ΔL is the splitting of the Si peak along the L -direction and ΔH is the separation of the rod from the substrate peak in the H direction. For the samples studied in this work, SWA resulted in a value of approximately $9 \pm 1^\circ$.

Longitudinal L -value through substrate and fin rods for $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) in Fig. 7(f) show peak splitting though distorted compared to $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$) sample owing to increased roughness and higher density of defects in the crystal. Longitudinal scans through the substrate peak displayed excellent interference fringes, which allowed calculation of the SiGe fin height (thickness) of the structure with the spacing between the fringes being inversely proportional to the number of diffracting planes in the layer. Rod 0 of Fig. 7(b) exhibited fringe spacing $\Delta L_{\text{fringes}}$ of 0.0088 resulting in the height of the structure of silicon fins as 61.7 ± 0.5 nm using Eq. (20). From Fig. 7(d), the $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$) layer height is determined to be

FIG. 7. (Color) ω - 2θ scans on silicon substrate peak (Rod 0) and higher order silicon fin peaks (rods +1, +1.5, +2, +3) for silicon, $\text{Si}_{0.75}\text{Ge}_{0.25}$, and $\text{Si}_{0.50}\text{Ge}_{0.50}$ fin array samples measured using QC3 (a), (c), (e), and APS (b), (d), (f).

40.2 ± 0.5 nm for $\Delta\omega_{\text{fringes}}$ of 0.0993° using Eq. (20). Similarly $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) sample exhibited a fin height value of 47.9 ± 0.5 nm calculated from the fringe spacing $\Delta\omega_{\text{fringes}}$ of 0.0742°

$$\text{Thickness} = \frac{\lambda}{2\Delta\omega_{\text{fringes}} \cos \theta_B} = \frac{1}{\Delta q_z} = \frac{0.543102 \text{ nm}}{\Delta L}. \quad (20)$$

The fin height was also verified using x-ray reflectivity measurements done at the APS, which displayed high-quality Kiessig fringes for all the samples as shown in Fig. 8 and the thickness is calculated using Eq. (21), which is simply the small angle approximation of Eq. (20), where $\cos \theta_B \sim 1$. The fin height calculated using x-ray reflectivity (XRR) is comparable with the HRXRD longitudinal line scan thickness. The fin heights for silicon fins, $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.25$) fins and $\text{Si}_{(1-x)}\text{Ge}_x$ ($x=0.50$) fins samples were determined to be 56 ± 0.5 nm, 42.2 ± 0.5 nm, and 47.9 ± 0.5 nm, respectively

$$\text{Thickness} = \frac{\lambda}{2\Delta\omega_{\text{fringes}}}. \quad (21)$$

FIG. 8. (Color) X-ray reflectivity profile on (a) silicon fins (b) $\text{Si}_{(1-x)}\text{Ge}_x$ ($x = 0.25$) and (c) $\text{Si}_{(1-x)}\text{Ge}_x$ ($x = 0.50$) (Source: APS).

V. SUMMARY AND CONCLUSIONS

The characterization of advanced fin structures using x-ray diffraction techniques has been demonstrated to be an essential nondestructive approach for quick and reliable analysis. Essential structural parameters such as pitch, pitch walking, fin height, and shape of the fin structure are determined for both silicon and silicon-germanium fins directly from reciprocal space maps of reciprocal space around key Bragg diffraction peaks. The results were in good agreement with other complementary techniques including SEM and optical scatterometry. A model based on the kinematical theory is under development by one of the authors (M. Wormington) and will be detailed in a future paper.

ACKNOWLEDGMENTS

The authors acknowledge TEL Technology Center, America, for their generous contribution of analysis time on the BML tool. M. Medikonda gratefully acknowledges funding from the Center for Nanoscale Metrology. Use of the

Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. The authors acknowledge the integration support of Jeremy Wahl, Kerem Akarvardar, and Steven Bentley along with the management support from William Taylor and Ajey Jacob from GLOBALFOUNDRIES.

¹B. S. Doyle, S. Datta, B.-Y. Jin, N. M. Zelik, and R. Chau, U.S. patent 7,348,284 B2 (25 March 2008).

²R. S. Chau, B. S. Doyle, J. Kavalieros, D. Barlage, S. Datta, and A. Hareland, U.S. patent 7,514,346 B2 (7 April 2009).

³Intel Corporation, "Intel 22nm technology—Intel's 22nm technology moves transistor into the 3rd dimension," Silicon innovations, Intel, Santa Clara, California (May 2011).

⁴ITRS, "Process integration devices and structures," International Technology Roadmap for Semiconductors (2011).

⁵R.-H. Kim *et al.*, *Proc. SPIE* **7640**, 76400F (2010).

⁶J. V. Gilfrich, I. C. Noyan, R. Jenkins, T. C. Huang, R. L. Snyder, D. K. Smith, M. A. Zaitz, and P. K. Predecki, "Advances in X-ray analysis," in *Proceedings of the 44th Annual Conference on Applications of X-ray Analysis*, Colorado Springs, CO (1995), Vol. 39, pp. 615–626.

⁷G. T. Baumbach and M. Gailhanou, *J. Phys. D: Appl. Phys.* **28**, 2321 (1995).

⁸L. D. Caro, P. Sciacovelli, and L. Tapfer, *Appl. Phys. Lett.* **64**, 34 (1994).

⁹G. R. Muthinti, M. Medikonda, J. Fronheiser, V. K. Kamineni, B. Peterson, J. Race, and A. C. Diebold, *Proc. SPIE* **8681**, 86810M (2013).

¹⁰Ullrich Pietsch, Václav Holý, and Tilo Baumbach, *High-Resolution X-ray Scattering: From Thin Films to Lateral Nanostructures* (Springer-Verlag, New York, 2004).

¹¹L. Tapfer and P. Grambow, *Appl. Phys. A* **50**, 3 (1990).

¹²A. A. Darhuber, E. Koppensteiner, H. Straub, G. Brunthaler, W. Faschinger, and G. Bauer, *J. Appl. Phys.* **76**, 7816 (1994).

¹³D. Keith Bowen and Brian K. Tanner, *High Resolution X-ray Diffractometry and Topography* (CRC, Boca Raton, FL, 1998).

¹⁴D. Keith Bowen and Brian K. Tanner, *X-ray Metrology in Semiconductor Manufacturing* (CRC, Boca Raton, FL, 2006).

¹⁵Paul F. Fewster, *X-ray Scattering in Semiconductors* (Imperial College Press, Covent Garden, London, 2003).

¹⁶G. R. Muthinti, M. Medikonda, T. Adam, A. Reznicek, and A. C. Diebold, *J. Appl. Phys.* **112**, 053519 (2012).

¹⁷Thomas N. Blanton, "Applications of X-ray diffraction in the imaging industry," JCPDS—International Center for Diffraction Data, *Advances in X-ray analysis* (2003), Vol. 46.

¹⁸Pui Yee Hung, Nikolai Kasper, Jim Nadeau, Injo Ok, Chris Hobbs, and Assunta Vigliante, *J. Vac. Sci. Technol. B* **30**, 041211 (2012).

¹⁹See supplementary material at <http://dx.doi.org/10.1116/1.4863316> for 004 and 224 RSMs on blanket pseudomorphic $\text{Si}_{(1-x)}\text{Ge}_x$ ($x = 0.50$)/Si samples.