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# **New Journal of Physics**

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# Ordering of nanostressors on free-standing silicon nanomembranes and nanoribbons

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**Abstract.** Epitaxial growth of self-assembled quantum dots (QDs) on singlecrystal nanomembranes yields organized arrays of QDs via a growth mode mediated by QD-induced strains in the membrane. A crucial aspect of this effect arises because epitaxial growth on thin Si sheets and nanostructures derived from them can occur simultaneously on two surfaces separated only by the 10-nm-scale thickness of the membrane. A QD on one surface of a free-standing membrane causes the nucleation of QDs in specific positions on the opposite surface. Control experiments using molecular beam epitaxy to deposit QDs on a single surface do not yield long-range order. Through-membrane elastic interactions consistent with predictions from finite-element-based mechanics models are observed using synchrotron x-ray microdiffraction. The role of crystallographic anisotropy is evident in finite-element predictions of the strains that bias the nucleation events. The scaling of the dot spacing with membrane thickness is consistent with the spacing of nucleation sites predicted using the mechanical model.

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#### 1. Introduction

Mechanical properties arising from the nanometer thickness of free-standing nanomembranes make these materials fundamentally different from bulk materials or supported thin films [1]. In biological systems, for example, nanometer-scale molecular structures self-assemble into wellordered arrays via membrane distortions [2]. Membranes created from single inorganic crystals allow the mechanical effects arising from small thickness to be combined with the atomicscale precision of surface science and epitaxial growth. Inorganic membranes are unique in this range of thickness because single crystals have anisotropic elastic properties and longrange orientational and translational symmetry over length scales that are orders of magnitude larger than their thickness. As we illustrate here, these effects make inorganic single-crystalline membranes quite different from thin layers of materials with isotropic mechanical properties. Single-crystal nanomembranes have thicknesses from 5 to 1000 nm, and are derived from a number of sources ranging from semiconductors to complex oxides. Within this class of materials, silicon membranes are an ideal model system in which to explore the nanometer-scale consequences of the combination of thinness and crystallinity because the materials science of silicon membranes is highly advanced, enabled by the development of silicon-on-insulator (SOI) substrates for the semiconductor device industry [3, 4].

In this paper, we describe the crystallographic distortion created by the epitaxial growth of nanostressors on both sides of a nanomembrane and a statistical study of the degree of order achieved in large arrays. We quantitatively test detailed mechanical predictions of the mechanisms responsible for long-range ordering phenomena arising in the heteroepitaxial growth of SiGe on Si nanomembranes. Finally, by comparing adjacent regions of supported and free-standing membranes, we show that the size of the quantum dots (QDs) increases for thin nanomembranes and provide an explanation for this phenomenon.

#### 2. Background and experimental details

Heteroepitaxial growth of materials with mismatched lattice constants is particularly advantageous for the understanding of mechanical effects in nanomembranes. First, heteroepitaxy rapidly produces QD nanostructures [5, 6], which act as nanostressors, inducing

a large mechanical distortion of the membrane. In addition to the distortion, nanomembranes present the unique opportunity to allow deposition conditions to produce nanostressors simultaneously on both the surfaces. Chemical vapor deposition (CVD), for example, can expose both the surfaces of a membrane to precursor gases. This creates the situation that cannot arise in conventional heteroepitaxy: nanostressors can interact elastically across the thickness of the nanomembranes.

The degree of order among QDs on the surface of a bulk material is generally poor because the strain generated in a bulk material by a nanostressor is very small and localized near the QD [7, 8]. On bulk substrates the stress arising from heteroepitaxy results in strains that distort the QD and bulk substrate in proportion to their relative thicknesses. The mismatch between the nanometer-scale stressor and the millimeter-scale substrate results in very small substrate strains [9]. A degree of order can be induced in arrays of QDs by controlling their nucleation in multiple-layer growth or in a single layer via a nanopatterned ridge, mesa or array of holes [7], [10]–[12]. Arrays of QDs grown on bulk substrates via multiple-layer growth, for example, can be ordered because the strain induced by QDs can modify the nucleation of QDs in a subsequent layer [6, 7]. Similarly, a single layer of QDs spatially confined on a nanopatterned ridge or mesa [8], or in a single layer in curved nanostructures can become ordered simply due to the curvature of the surface [13]. In all of these cases, the local strain caused by the nanostressor is shared between the nanostressor and the very thick substrate. As a result, on thick substrates the QDs induce only weak localized strain fields [9, 13]. Additional island-island interaction occurring via surface diffusion and coarsening after the nucleation of QDs can improve ordering [14, 15].

On nanomembranes, long-range order in the spatial distribution of nanostressors results because the locally strained membrane provides strong feedback for subsequent self-organization of stressed nanostructures [16]. The consequence of this ordered QD array on the Si membrane is a strain lattice consisting of 100 nm regions of high tensile strain. The periodic strain resulting from the nanostressor array affects the local electronic band structure of Si membranes [17].

For thin membranes, the strain field on one surface appears as a strain distribution on the opposite surface, something obviously not found on bulk substrates. Strain fields reach through the thickness of the membrane and produce a large deformation of the entire substrate. The deformation induced in the membrane by a single QD in the ordering process can be understood by using a three-dimensional finite element simulation [16]. This in turn can be used to predict the distance and position of nearby sites at which the nucleation energy for QD formation is minimized. Earlier observations have already shown that curvature and strain can alter the elastic contribution to the nucleation barrier and thus select the nucleation sites of QDs [7, 18]. Following the random nucleation of an initial QD, the QDs on the other side of the membrane nucleate in sites of reduced surface chemical potential produced by the strain field of the original QD. The strain transfer is greater for higher Ge concentration in the QD and for thinner Si membranes, so local strain in the membrane extends laterally beyond the base of the QD only when the membrane is thinned down to the nanometer scale. The growth of QDs on both sides of the membrane is essential to the ordering because when growth proceeds on only one surface, as in molecular beam epitaxy (MBE), the mechanical interactions are far more limited, even in nanomembranes.

One can fabricate very thin Si membranes with (001) and, to some extent, with other orientations [19, 20]. The ultrathin Si nanomembranes for this study are created by thinning

and releasing the Si device ('template') layer of an SOI substrate with standard (001) orientation [21]. These Si(001) nanomembranes can be completely free-standing or partially attached to a substrate and either flat or curled into hybrid three-, two- or one-dimensional structures and can be as thin as 5 nm [22–25]. The membranes are patterned into ribbons of widths ranging from tens of nanometers to micrometers, supported at one or both ends [26]. We used SOI (001) with an Si template layer of initial thickness 200 nm bonded to a 3  $\mu$ m thick SiO<sub>2</sub> layer. We thinned the template layer to 25 nm using two cycles of thermal oxidation and etching in a 6:1 buffered oxide etching solution, in which a thickness of approximately 90 nm of Si was removed in each cycle. Further thinning is achieved in smaller steps by alternating chemical oxidation and etching in hydrofluoric acid (HF) [27]. The chemical oxidation in this step uses a modified SC1 solution consisting of concentrated NH<sub>4</sub>OH solution, 30% H<sub>2</sub>O<sub>2</sub> solution, and H<sub>2</sub>O in a ratio of 1:8:64 parts by weight. Each cycle of chemical oxidation and stripping removes 2 nm of Si. The thickness of the Si (001) template layer is measured using high-resolution x-ray diffraction and optical reflectivity, with an uncertainty in each case of  $\pm 1.5$  nm.

We use electron beam lithography to define the lateral shapes of nanomembranes and nanoribbons. A polymethylmethacrylate resist is patterned to produce windows for Si etching. To provide access to the underlying SiO<sub>2</sub> release layer, the Si template layer is removed, using reactive ion etching, in areas defined by photolithography. An extended immersion in 10% HF removes the oxide, leaving the membranes free standing and hydrogen terminated on both sides. Nanoribbons remain attached at their ends, spanning the regions in which SiO<sub>2</sub> was removed. Similarly, partially removing the SiO<sub>2</sub> from beneath the cantilever or mesa patterns created with optical lithography produces overhanging free-standing membranes attached only at one edge.

We use CVD or MBE under ultrahigh vacuum conditions to deposit Ge or a Ge-rich SiGe alloy on lithographically patterned but still supported template layers, on membrane cantilevers attached at one edge, or on free-standing, end-supported nanoribbons. The hydrogen termination produced during the HF etching release (and simultaneous surface cleaning) step is removed by briefly heating the sample to 900 °C before deposition. The key difference between CVD and MBE in the context of the present study is that CVD causes atoms to be deposited on both sides of an under-etched membrane, while MBE limits deposition to the top surface.

The spatial distribution of QDs on Si nanomembranes or nanoribbons can be probed quantitatively using scanning electron microscopy (SEM). SEM images of nanomembranes after the creation of SiGe QDs are shown in figure 1. Because of the thinness of the membrane, QDs on both sides of the Si membranes can appear in SEM images, as seen in figure 1(a), because the scanning 20 keV electron beam penetrates the entire membrane/QD structure. The top and bottom QDs can be easily distinguished by imaging the nanoribbons from the side, as shown in figure 1(b). An alternative imaging mechanism is provided by orienting the sample so that the electron beam is incident at 30° with respect to the surface normal, which yields contrast in SEM images that allows a distinction to be made between QDs on the top or bottom surfaces. The geometric projection of the incident beam onto the QDs causes the QDs on the top of membrane to be brighter in the upper half of their images and darker in the lower half. The opposite contrast variation occurs for QDs on the bottom surface of the membrane. An example of the difference between top-surface and bottom-surface QDs is illustrated in figure 1(c).

Synchrotron x-ray microdiffraction allows us to resolve the strain and bending induced in individual nanoribbons. X-ray microdiffraction experiments were conducted at station 2ID-D of the Advanced Photon Source at Argonne National Laboratory. An x-ray beam with a photon energy of 11.2 keV was focused with a Fresnel zone plate to a 200 nm spot size on the sample.



**Figure 1.** SEM images of SiGe QDs on Si nanomembranes and nanoribbons. (a) Two rows of SiGe QDs on a 20 nm thick Si nanoribbon. (b) A side-view image of the nanoribbon acquired with a sample tilt angle of 45°. Arrows indicate the anticorrelated positions of QDs on the top and bottom surfaces. (c) When the electron beam is incident on the sample at large angles with respect to the surface normal, the geometric projections of Ge QDs on the top (left) and bottom (right) surfaces of an Si membrane are different and the two types of QDs can be distinguished. (d) The global buckling of Si nanoribbons arising from the expansion of the Si lattice due to strain sharing with the SiGe QDs.

Focusing the x-ray beam introduces an angular divergence of approximately  $0.05^{\circ}$  and as a result effectively broadens the observed angular widths of x-ray rocking curves. Diffracted x-rays were collected with a charge-coupled device (CCD). This approach has previously been used to probe the structure distortion of a nanomembrane on which QDs were grown on only the top surface [28]. As we show below, the reflections observed in x-ray microdiffraction experiments provide both information on the local orientation of the nanomembrane crystallographic lattice and a spatially resolved measurement of the lattice parameter.

## 3. Results and discussion

## 3.1. X-ray microdiffraction

Isolated coherent QDs locally bend and deform a free-standing Si membrane because the 4% difference in the lattice constants of Ge and Si induces a mismatch strain [9, 29]. At the most local scale, x-ray diffraction directly probes the mechanical distortion produced by the QDs. The x-ray diffraction experiment produces measurements of the diffracted intensity in a slice through reciprocal space. The CCD image provides the intensity of the Si (004) Bragg reflection as a function of two angular directions,  $2\theta$  and  $\chi$ , defined in [28]:  $2\theta$  is the conventional opening



**Figure 2.** X-ray microdiffraction pattern of a single Si nanoribbon. (a) Broadened x-ray diffraction pattern arising from deformation by QD stressors. The vertical and horizontal axes represent the conventional  $2\theta$  scattering angle and the angle  $\chi$ , as defined in [28]. The large spread along the horizontal  $\chi$  axis arises from local bending of the nanoribbon due to SiGe nanostressors. (b) Sharp x-ray microdiffraction pattern of the undistorted supported region of the Si nanomembrane. (c) Integrated intensity as a function of scattering angle  $\chi$  for the nanoribbon and supported Si nanomembrane (inset). Features marked with (\*) in (a) and (c) arise from diffuse scattering near the handle wafer Si (004) reflection.

angle of the diffracted beam with respect to the incident beam direction and  $\chi$  is perpendicular to  $2\theta$ .

The broadening of the diffraction pattern along  $\chi$  induced by the QDs, as in figure 2(a), can be used to estimate the local curvature of the free-standing membrane beneath the QDs. The curvature  $\kappa$  is approximately given by  $\kappa = \Delta \chi / w$ , where w is the width of the QD and  $\Delta \chi$  is the angular width of the x-ray reflection. The microdiffraction results give  $\Delta \chi \approx 3^{\circ}$ , which, with a width w of 100 nm, gives a radius of curvature  $1/\kappa$  of  $1.9 \,\mu$ m. This radius corresponds to values obtained in theoretical calculations [9].

Evidence for the crucial role of the distortion of the membrane in ordering can be obtained by comparing the nanoribbon diffraction pattern with the diffraction pattern of an adjacent supported region. The diffraction pattern of a region in which QDs have been deposited on a supported nanomembrane, an unpatterned area of the SOI substrate, is shown in figure 2(b). The width of the reflection in figure 2(b) is limited only by the angular divergence of the focused beam, illustrating that there is no bending on the length scale of the width of the QDs in the supported membranes.

#### 3.2. Large-scale distortion of nanoribbons

When their mechanical stress is averaged over longer distances along the surface of membranes or ribbons, the QDs lead to an average distortion of the nanomembranes lattice. Once many QDs have formed, the overall elastic effect of the QDs is similar to that of continuous compressively strained films deposited on both surfaces [9]. The thinness of the Si nanomembranes leads to an average overall elastic distribution of the strain arising from epitaxial mismatch between the SiGe QDs and the nanomembranes. With an area-averaged thickness of the SiGe composition of the QDs, Si<sub>0.36</sub>Ge<sub>0.64</sub>, on the 23 nm nanomembranes, elastic strain sharing predicts an expansion of the Si nanomembrane enclosed by these compressively strained SiGe layers by 0.4% [30]. A ribbon constrained at both ends cannot accommodate this strain without buckling [29]. This behavior is the same as observed in strained nanomembrane tubes or 'rugs' and other structures [31], except that here the ribbon is constrained and thus must buckle, randomly up or down. Examples of buckled nanoribbons in these configurations are shown in figure 1(d). The global buckling is also observed when the QDs are deposited via MBE: in this case, the free-standing (cantilevered) portion of the membrane bends down toward the substrate, because the compressively strained material is deposited on the top surface.

#### 3.3. Ordering of nanostressors

We now address the ordering of the nanostressors into arrays. The formation of arrays of SiGe QDs on both sides of an Si nanomembrane or nanoribbon is immediately apparent across large distances (figure 3(a)). The QDs on the two surfaces of a free-standing two-dimensional membrane assemble in a highly ordered square lattice (figure 3(b)). Growth on nanoribbons produces one or more lines of QDs, depending on the width of the ribbon (figure 1(a)). For both sample geometries, QDs grow on both top and bottom surfaces of the ribbon. The rows of QDs on both surfaces are aligned with the  $\langle 100 \rangle$  elastically soft crystallographic directions of Si, but those on one surface are offset in the  $\langle 110 \rangle$  direction from those on the opposite surface. The alignment and offset are independent of the crystallographic direction in which a ribbon is cut. We have observed ordering of up to five rows of QDs on cantilever structures.

In order to describe the QD array quantitatively, we have calculated the positional pair correlation functions (PCFs; defined in [32]) of QDs. PCFs can be produced to determine the correlation for pairs of dots on the same surface or on opposite surfaces. Figure 4(a) shows the PCF for  $Si_{0.36}Ge_{0.64}$  QDs formed on opposite sides of a 23 nm thick membrane. The sharp peaks of the PCF in figure 4(a) correspond to the most common relative locations of a QD on the top surface of the nanomembrane relative to a QD on the bottom surface. The strong nearest-neighbor and second-neighbor peaks indicate a high degree of order. The full-width at half-maximum of the peaks in the PCF of the ordered layer is 40 nm, corresponding to the variation in the period of the superlattice throughout the entire array. For comparison, the PCF of QDs on the Si layer (of equal thickness) still attached to the oxide is shown in figure 4(b). This case corresponds to the conventional self-organization of strained nanostructures [6, 7] and shows only that two QDs cannot occupy the same area. The PCF of QDs on this rigid substrate indicates a minimum center-to-center distance of 100 nm, but no directional or long-range ordering.

The observed orientation of the square QD array with respect to the underlying Si lattice is consistent with mechanical arguments given in [16]. Peaks in the PCF are fourfold symmetric, falling along the  $\langle 110 \rangle$  crystallographic directions. Further evidence that the ordering direction



**Figure 3.** (a) QD array on a free-standing Si nanomembrane created by partially underetching an SOI structure. QDs on the supported region at right are not ordered. (b) Enlarged area of the organized QD array.

of the QDs is determined by the crystallographic anisotropy of the Si substrate is found by determining the effect of the presence of an edge of the membrane on the pattern. In other systems, the shape and elastic relaxation at edges and other linear features provide a strong impetus to organization [11]. In this case, however, the edge effect is absent. We have determined PCFs for QDs grown near the edges of membranes cut along both the  $\langle 100 \rangle$  and  $\langle 110 \rangle$  directions. Peaks in the PCF for both types of edges are found along the same directions with similar average top–bottom QD spacings to 111 and 108 nm, respectively.

#### 3.4. Nanomembrane thickness effects

Finite-element calculations predict that decreasing the thickness of the nanomembrane results in a decrease in the separation of the QDs in the array [16]. The results of new finite-element calculations using the model in [16] with parameters matching the experimental cases presented here are shown in figure 5. The preferred nucleation sites are apparent in figures 5(a) and (b) as minima in the normalized strain energy density, as defined in [16]. Changing the thickness of the membrane from 23 nm (figure 5(a)) to 6 nm (figure 5(b)) brings the energy minimum closer to the center of the QD. Plots of the normalized strain energy density as a function of all relative positions are shown for these two thicknesses in figures 5(c) and (d). To test the prediction that decreasing the nanomembrane thickness results in a more compact array, we have grown QDs of



**Figure 4.** PCF analysis of the ordering of QD stressors. (a) PCF for QDs on the top and bottom surfaces of a free-standing, 23 nm thick membrane. Sharp peaks indicate ordering of the QDs. (b) PCF of QDs grown on a supported silicon membrane. The PCF on this supported area is indicative only of the requirement that two QDs cannot occupy the same area. (c) Plots of selected lines through the PCF in (a). (red) A line through PCF for a 23 nm membrane with edge along  $\langle 110 \rangle$ ; (blue) a line through a PCF from a different area of the same sample near an edge cut along  $\langle 100 \rangle$  rather than  $\langle 110 \rangle$ ; and a (black) line through the PCF for a QD with the same composition grown on a 6 nm thick nanomembrane.

the same composition on a 6 nm Si nanomembrane. The average nearest-neighbor top-bottom separation of QDs on the 6 nm nanomembrane is 87 nm compared to 110 nm for the 23 nm nanomembrane, as shown in the PCF in figure 4(b).

For both the 6 and 23 nm thick membranes, the experimentally measured separation between the centers of QDs on opposite surfaces is larger than the separation distances predicted by the elasticity calculation. This may be a result of the asymmetry in the normalized strain energy distribution around the minima that is seen in both figures 5(a) and (b). Specifically, the normalized strain energy is lower on the sides of the minima that are farther away from the hut. As a result of this asymmetry, the growth that occurs after nucleation will be biased away from the hut, leading to a larger center-to-center separation.

Changing the thickness of the nanomembrane results in changes in the lateral size of the QDs. In both MBE and CVD, we observe that QDs in the free-standing area of the membrane are larger than in the areas supported by oxide. An increase in size is expected because the compliant free-standing nanomembrane shares strain with the QD, resulting in a larger critical size. As a result, the critical size of QD formation or nucleation is strain dependent [33, 34].

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**Figure 5.** Finite-element predictions of the normalized strain energy profile along the  $\langle 110 \rangle$  direction on the surface opposite to (a) a 115 nm SiGe QD on a 23 nm thick nanomembrane and (b) a 110 nm SiGe QD on a 6 nm thick nanomembrane. (c) Normalized strain energy contour on the surface opposite a 115 nm SiGe QD (outlined by the solid black square) resting on a 23 nm thick Si nanomembrane. Minima in the energy are marked with (+). (d) The analogous plot for a 6 nm thick nanomembrane with a 110 nm QD. Experimentally observed top-bottom nearest-neighbor distances are indicated by (X) in (c) and (d). The numbers on the scale represent normalized energies.

The QD volume can be estimated as  $V = h^3 \cot^2 \theta$ , where *h* is the QD height and  $\theta$  is the angle between the faces of the QD and the surface, respectively. The height *h* increase is inversely proportional to the square of the biaxial strain,  $h \propto \varepsilon^{-2}$ , and hence the hut volume scales as  $\varepsilon^{-6}$  [33]. The misfit strain in Ge QD decreases with decreasing the Si thickness and reaches a minimum of 2% as Si is 5 nm thick, which is the equivalent thickness of Ge QD [28]. Since we have already shown that the strain decreases with decreasing the Si thickness, we can expect that the dot volume would increase with decreasing the Si thickness. The ratio of the hut volume *V* to the volume  $V_0$  of QDs on bulk Si,  $V/V_0$ , increases to 6, meaning that QDs become sixfold larger in volume and a factor of 1.8 larger in linear extent than QDs grown on bulk Si. This increase becomes even more pronounced on thinner Si substrates.

#### 4. Summary and conclusions

To summarize, we describe a unique route for the fabrication of periodically strained nanostructures by depositing on both the sides of a nanomembrane or nanoribbon to produce ordered arrays. The combination of semiconductor nanomembranes with strain-driven self-organization of nanostructures significantly increases the degree of order of QDs in comparison with those obtained by heteroepitaxy on bulk substrates. As a consequence of this order, significant new electronic transport behaviors can be expected in membranes distorted by arrays of QDs. Transport in a thin sheet emphasizes the influence of the surface [35, 36], where it can easily be modified by strain, because the band structure of Si is quite sensitive to strain [37]. The ordered QDs introduce a periodic strain, creating effectively an electronic heterojunction superlattice made from a single element that may have an impact on optoelectronic and thermoelectric device structures [38].

A membrane-distortion-driven ordering mechanism will be accessible in membranes of all the inorganic systems that exhibit strain-mediated growth of nanostructures. Similar structural and electronic effects can be created in a broad range of functional materials through the combination of nanomembranes and strain-mediated self-organization of nanostructures. For example, QDs in the GaAs/InAs system are formed through the same self-limiting balance of elastic strain relaxation and surface energies as occurs in Si/SiGe, and such QDs are similarly difficult to organize on rigid substrates. Heterostructures are formed using epitaxial growth techniques in this system, and GaAs membranes can be released using several experimental methods [39]. Thermoelectric materials based on QDs in the PbSeTe/PbTe system face similar issues associated with the assembly of array nanostructures [40]. Finally, nanometer-scale crystals of magnetic oxides can be formed from a number of complex-oxide heteroepitaxial systems and can find nanomagnetic applications in ordered arrays [41]. The surprising benefit of creating novel strain and electronic lattices on the length scale of 100 nm in this manner provides an entirely new dimension to the use of nanostructures in the form of sheet, ribbons or dots.

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