

Size-Dependent Silicon Epitaxy at Mesoscale Dimensions

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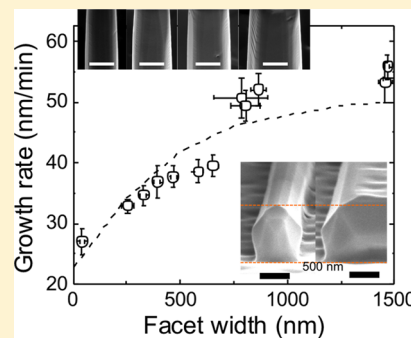
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S Supporting Information

ABSTRACT: New discoveries on collective processes in materials fabrication and performance are emerging in the mesoscopic size regime between the nanoscale, where atomistic effects dominate, and the macroscale, where bulk-like behavior rules. For semiconductor electronics and photonics, dimensional control of the architecture in this regime is the limiting factor for device performance. Epitaxial crystal growth is the major tool enabling simultaneous control of the dimensions and properties of such architectures. Although size-dependent effects have been studied for many small-scale systems, they have not been reported for the epitaxial growth of Si crystalline surfaces. Here, we show a strong dependence of epitaxial growth rates on size for nano to microscale radial wires and planar stripes. A model for this unexpected size-dependent vapor phase epitaxy behavior at small dimensions suggests that these effects are universal and result from an enhanced surface desorption of the silane (SiH_4) growth precursor near facet edges. Introducing phosphorus or boron dopants during the silicon epitaxy further decreases the growth rates and, for phosphorus, gives rise to a critical layer thickness for single crystalline epitaxial growth. This previously unknown mesoscopic size-dependent growth effect at mesoscopic dimensions points to a new mechanism in vapor phase growth and promises greater control of advanced device geometries.

KEYWORDS: Epitaxy, Nanowires, Mesoscale, Silicon, Chemical Vapor Deposition



At the heart of the dramatic transition in semiconductor device technology from planar to three-dimensional (3D) architectures is the ability to shrink devices from micro to nanoscale dimensions with near atomic level control of materials' structure and composition. The resulting 3D structures across this mesoscopic size regime serve as the building blocks for high-performance electronic and photonic devices, such as photovoltaic cells,^{1,2} light-emitting diodes,^{3,4} and multigate field effect transistors,^{5–7} and require unprecedented control in dimensions and materials properties. Epitaxial crystal growth in combination with lithographic and etching technologies provides one of the enabling approaches to create such structures and to tailor their 3D composition and doping profiles in accordance with desired properties. The electronics technology standard for epitaxial growth is chemical vapor deposition (CVD), which enables near atomic level control of layer thickness, composition, and electrical dopant profiles. New understanding of crystal growth kinetics at small sizes can shed light on such effects as dimensional control for new 3D device architectures and is also of great interest from a fundamental perspective for understanding mesoscopic scale science.

We show that Si epitaxial growth at the mesoscale is size dependent at dimensions significantly larger than the onset of thermodynamic limits. This previously unknown behavior for Si epitaxy, arguably the world's best studied crystal growth system, is unexpected and our studies of different geometries indicate this behavior is a general phenomenon. Motivated by radial junction nanowire (NW) arrays for novel solar cells,² radial Si epitaxial CVD growth was studied on Si NWs and compared to Si planar epitaxy on stripes with submicrometer widths. Results show a monotonic reduction in homoepitaxial growth rate with size for facet widths below $\sim 1 \mu\text{m}$, indicating an area-dependent Si incorporation rate, and modeling suggests the reduction is due to a new mechanism of edge-controlled desorption in CVD epitaxy at the mesoscale. The presence of *n*- and *p*-type dopants results in even greater reductions in low temperature radial growth rates. For phosphorus (P) (*n*-type doping) shell growth, a critical thickness for Si single crystal radial epitaxy is found. The present results provide new insights

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on the nature of CVD crystal growth at small dimensions and have significant implications for the fabrication of technologically important 3D crystalline Si electronic device architectures and for creating novel energy harvesting structures on low-cost substrates.

Si radial shells on high radius-of-curvature Si NWs (Supporting Information Figure S1, S2) and Si planar sheets on the flat surfaces of etched narrow Si stripes (Supporting Information Figure S3) were grown by low-pressure CVD in a cold wall reactor using SiH_4 in H_2 , with B_2H_6 or PH_3 precursors in H_2 added for the case of doped epitaxial growth (see Supporting Information). The starting NW and stripe substrates for size-dependent growth studies were fabricated by lithographic techniques and the growth conditions for radial shells and planar sheets were identical with the growth temperature fixed at 810°C except where otherwise noted.

The morphological features of Si 3D mesoscale epitaxy were observed by scanning electron microscopy (SEM). Figure 1a

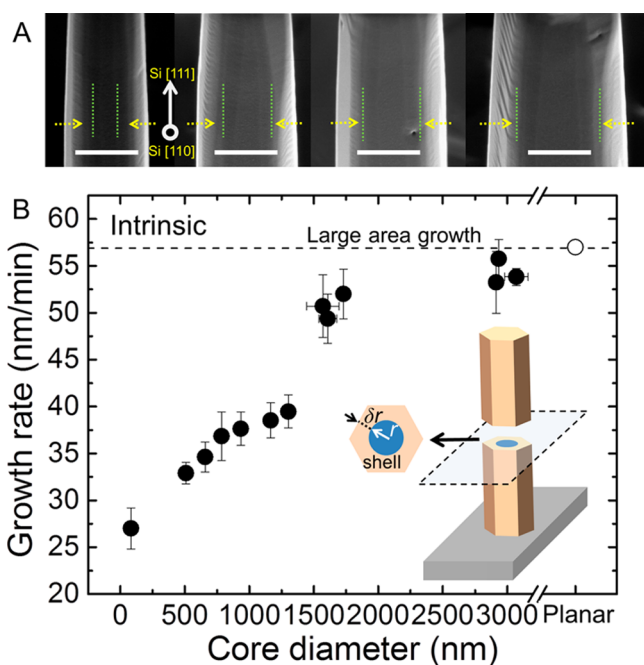


Figure 1. Images and growth rate of epitaxial silicon radial shell growth showing large reductions at small wire diameters. (A) High-resolution SEM images of undoped Si radial shells grown for 5 min on Si NWs with different core diameters showing {110} surface facets on NW sidewalls (scale bars: $1\ \mu\text{m}$). Yellow dashed arrow indicates the edges of a facet face after growth and the green dashed line shows the initial core NW diameter before growth. (B) Epitaxial growth rate of undoped Si {110} faceted radial shells on Si {111} NW sidewalls for a given NW core diameter. Each data point is the average of ~ 40 NW measurements. The open circle corresponds to the epitaxial growth rate of a Si (111) film for a large area $1 \times 1\ \text{cm}^2$ size substrate measured under identical growth conditions.

shows the radial epitaxial growth of undoped Si shells on the surfaces of Si [111]-oriented NWs with different diameters from $400\ \text{nm}$ to $1.8\ \mu\text{m}$ under the same growth conditions. The Si radial shells are single crystalline as determined by transmission electron microscopy (TEM; Supporting Information Figure S4) and are bounded by well-defined Si {110} facets with some surface steps⁸ visible on the faceted sidewalls. The Si {110} sidewalls of the Si [111] NWs, as shown in Figure 1a and Supporting Information Figure S5, form facets in the

initial stage of growth and retain the faceted structure throughout growth. The thermodynamically stable Si {110} planes form to minimize the surface energy on the high radius of curvature NW surfaces.^{2,9,10}

To quantitatively investigate the Si radial growth rate, shell thicknesses were measured for fixed growth times for different starting (core) Si [111] NW diameters from $110\ \text{nm}$ to $3\ \mu\text{m}$. Figure 1b shows that the Si {110} shell growth rate is significantly reduced for core diameters below $\sim 1.5\ \mu\text{m}$. The details of the growth rate determination for our geometry are described in Supporting Information. We note that the reduced radial growth rate at smaller dimensions did not depend on the NW spacings. At larger diameters the growth rate is observed to approach that for planar crystalline thin film growth, where the open circle in Figure 1b corresponds to planar, large area Si epitaxial growth measured under the same conditions. Epitaxial crystal growth is described by both thermodynamic and kinetic factors. In previous reduced-size semiconductor growth studies, thermodynamic effects have received considerable attention, for example, in liquid metal catalyzed vapor phase crystal growth for NW semiconductors.^{11–13} In that case, diameter-dependent growth rate effects were restricted to longitudinal NW growth rates for metal-catalyzed vapor–liquid–solid growth for diameters below $100\ \text{nm}$.^{11–14} In this subhundred nanometer size regime, the well-known Gibbs–Thomson effect can result in decreased growth rates at smaller sizes due to an increase in the equilibrium chemical potential, a thermodynamic effect that only becomes significant at sizes below a few 10s of nanometers. Also, both thermodynamic and kinetic effects have been examined in noncatalyzed solution phase colloidal growth.^{15,16} The present mesoscopic size regime, spans hundreds of nanometers to $1.5\ \mu\text{m}$, thus excluding explanations based on thermodynamic size effects. In contrast to the above nanoscale liquid phase growth studies, a size dependence of vapor phase crystal growth kinetics for Si in the mesoscale transition region has not, to our knowledge, been reported previously. We also note that recent studies of 2-dimensional artificial crystal growth have reported effects of curved surfaces on growth stability for significantly larger radii of curvature ($\sim 10\text{s}\ \mu\text{m}$) due to curvature-induced elastic strain.¹⁷ However, the rapid formation of facets on the curved NW surfaces here indicate such curvature effects are not a significant factor and instead suggests that a kinetic origin may be responsible for this unexpected behavior.

To further explore possible kinetic effects and exclude curvature effects we investigated CVD growth of Si on planar substrates at reduced dimensions along the surface normal direction. Size-dependent Si growth rates were measured on the top of narrow Si stripes, comparable in width to the Si NW sidewall facets. Figure 2a shows Si (100) planar stripes, with the same initial height of $800\ \text{nm}$ and initial widths ranging from $500\ \text{nm}$ to $3\ \mu\text{m}$, after Si thin film growth for 5 min under CVD growth conditions identical to the radial NW growth (Figure 1b). As seen in the example shown in Figure 2a, the top of the $500\ \text{nm}$ -wide stripe is lower than that of the $3\ \mu\text{m}$ -wide Si stripe, indicating a decrease in Si planar growth rates for smaller stripe widths. Reduced growth rates with decreasing width were also observed for other Si stripe crystal orientations (see Supporting Information Figure S9). The similarity in reduced growth rate behavior for both Si radial shells and planar stripes at small dimensions suggests that there is a universal size-dependent growth behavior at such mesoscopic dimensions. To confirm this possibility, the growth rates of the Si radial shell

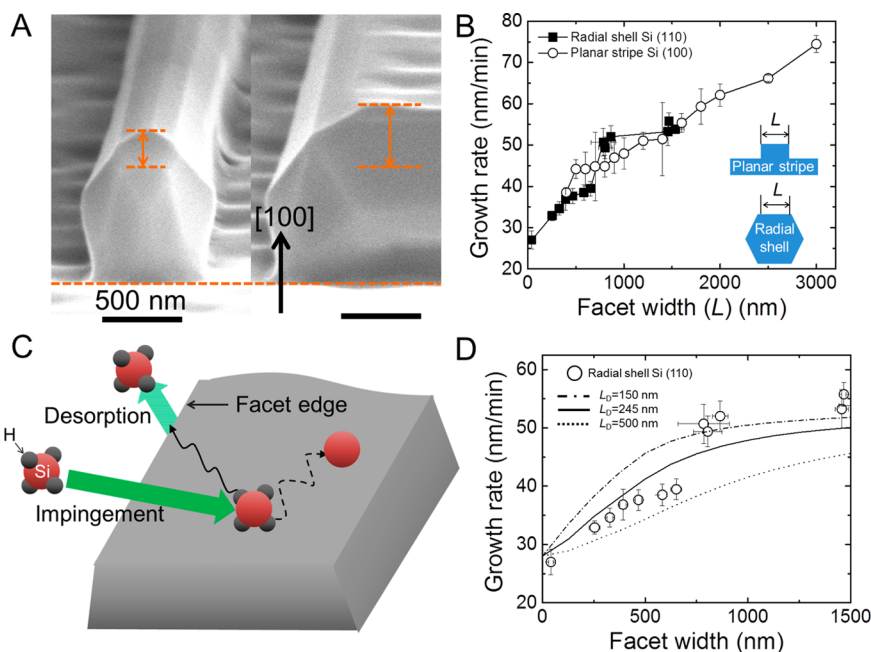


Figure 2. Images, measurements, and model for size-dependent epitaxial silicon growth on narrow planar silicon stripes and radial wire facets. (A) Low magnification, 80°-tilted SEM image of Si (100) planar stripes after Si epitaxial thin film growth for 5 min for etched initial stripe widths of 500 nm (left) and 3 μm (right). (B) Comparison between Si (100) growth rate on planar stripes and Si (110) radial shell growth rate for the same facet width as the corresponding planar stripe. (C) Schematic of the area-dependent SiH₄ adsorption model. Two different paths of an incident SiH₄ molecule followed by desorption at the edges and decomposition to Si adatom are depicted. (D) Fitted curves based on our area-dependent growth model with enhanced facet edge precursor desorption compared to the measured diameter-dependent growth rates of Si {110} radial shells. The dash-dot, solid, and dot curves represent SiH₄ surface diffusion lengths (L_D) of 150, 245, and 500 nm, respectively.

facets and planar sheets were compared for the same NW facet and planar stripe widths. Figure 2b shows that the Si growth rates on Si {110} sidewall facets and planar Si (100) stripes with the same facet width are in close agreement, indicating that the size-dependent growth rates exhibit consistent behavior for similar growth areas.

The apparent universality of a size-dependent growth rate observed here for both radial and planar sheet-like growth indicates that the conventional growth kinetics of vapor phase epitaxy requires further examination. Conventional kinetic models of vapor phase Si epitaxy consist of consecutive steps from precursor adsorption to thermal decomposition and incorporation of Si atoms into the growing crystal surface. The epitaxial growth rate is then governed by the rate-limiting step, as determined by the growth conditions. For the present case, the rate-limiting step in large area conventional growth models is the SiH₄ precursor adsorption.^{18,19} At lower temperatures, the precursor adsorption in conventional Si planar growth is affected by hydrogen coverage;^{18,20} however, at our growth temperature of 810 °C, the fractional hydrogen coverage is less than 0.1. This small hydrogen coverage is insignificant in hindering precursor adsorption. Thus, the conventional model of Si epitaxy requires additional considerations to explain the present mesoscopic growth effects.

To quantitatively analyze and better understand the size-dependent growth rate, we propose a model of “area-dependent precursor adsorption”. In our model, the Si precursor molecules migrate on the Si surface before adsorption. The diffusing precursor molecule, SiH₄, can be either adsorbed at available sites on the Si solid surface, followed by thermal decomposition and subsequent epitaxial growth (incorporation into the crystal), or be desorbed without decomposition. At our growth conditions of 15 mTorr, a growth rate of 50 nm/min (see

Figure 1b) corresponds to 1 in 1300 (0.08%) of the incident SiH₄ molecular flux incident on the Si surface incorporating Si atoms into the growing epitaxial layer, a reactive sticking coefficient consistent with previous studies.²¹ As a result, a small shift in the adsorption rate can have a large effect on the Si incorporation and resulting growth rate. Previous studies, for InAs and GaAs,²² for example, have suggested that surface topography such as step edges can influence the desorption rate of precursor molecules from semiconductor surfaces. Here, we postulate that the desorption coefficient of the incident precursor molecules is different at the edge and the center of the facets of our structures. We hypothesize that the SiH₄ desorption is enhanced at the edges of a single facet on the sidewalls of NWs and, similarly, at the edges of planar stripes as depicted in Figure 2c. Thus, for small area epitaxy, in which at least one dimension is smaller than the characteristic surface diffusion length for SiH₄ molecules, the rate of Si incorporation will be reduced and become size dependent. To model this area-dependent precursor adsorption effect, we first assume that the rate of SiH₄ decomposition, and thus of growth, is proportional to the concentration of adsorbed SiH₄ molecules, c . On a macroscopic facet, c is determined by when the rates of decomposition and evaporation are balanced by the incoming flux f , that is, $c/\tau = f$, where τ is the lifetime of a SiH₄ molecule on the surface before evaporation or decomposition. Now, suppose in the regions of the crystal between {110} facets (the facet edge) the evaporation rate is higher, giving a locally reduced value of τ . Because molecules deposited on the facet can diffuse to the facet edges and evaporate before decomposing, this will lead to a decreased concentration on the terrace and, thus, lower the growth rate compared to a macroscopic facet. How much this enhanced evaporation decreases the growth rate depends on the facet size and

diffusivity D of a SiH_4 molecule. To calculate the decrease, we solve the one-dimensional diffusion equation $D\nabla^2 c + f - c/\tau = 0$ with τ different on the facet surface and the facet edges. The concentration and the flux $D\nabla c$ are assumed continuous between the two regions, and the width of the facets is taken to be much larger than the region between them. Because the decomposition rate is proportional to c , the growth rate of the facet is proportional to the integral of c over the facet. Figure 2d shows that the size dependence of the growth rate observed in experiments is reproduced when the diffusion length of a SiH_4 molecule ($L_D = (D\tau)^{1/2}$) on the terrace is 245 ± 80 nm. As seen for the predicted dependence for 150 to 500 nm diffusion lengths (Figure 2d), a decrease in diffusion length results in a delay in the onset of growth rate reduction with size. More surface and growth studies are needed to fully understand this new area-dependent precursor adsorption mechanism. There have been few studies of the diffusion of silicon hydride species on Si surfaces,²³ and none for silane or related precursors on crystalline Si surfaces to our knowledge. We suggest that the influence of surface area on CVD growth may be a general phenomenon, extending to other growth systems. To treat such effects for three-dimensional nanoscale structures in detail, including growth rates and surface facet evolution, the above treatment could be extended to a two-dimensional diffusion model. We believe these effects are important to understand in the future because they can significantly influence growth kinetics at small sizes and strongly affect the resulting architectures in the fabrication of 3D mesoscopic structures.

Silicon (Si) epitaxy is also affected by dopant incorporation because dopants may segregate to the surface, and thereby change step formation energies, alter Si adatom migration lengths, and influence precursor adsorption and desorption. Figure 3 shows the effect of dopant incorporation on the size-dependent Si {110} radial shell growth rate. The growth rates are again found to decrease with decreasing diameter at the ~ 1

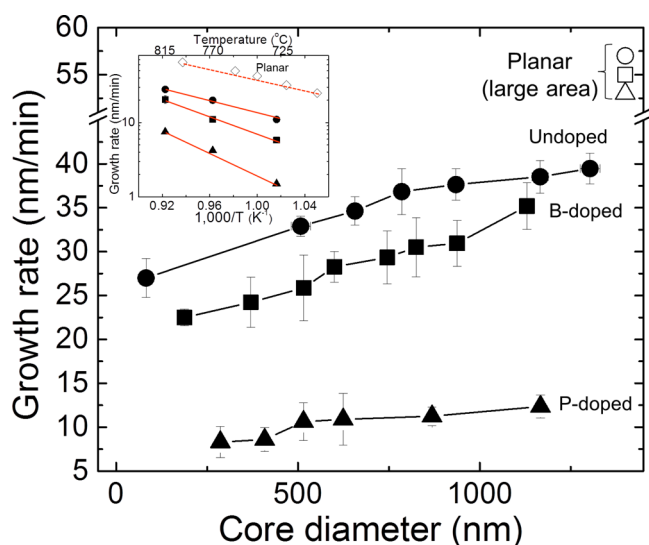


Figure 3. Effect of dopants on size-dependent silicon radial shell growth rates. Core diameter-dependent radial shell growth rate of undoped, B-doped ($\sim 1.3 \times 10^{19}/\text{cm}^3$), and P-doped ($\sim 3 \times 10^{19}/\text{cm}^3$) Si. Growth rates for large area surfaces ($\sim 1 \text{ cm}^2$) are shown by open symbols (with standard deviation < 3 nm). Inset shows the undoped, P-doped, and B-doped temperature dependent growth rates in an Arrhenius plot, indicating similar activation energies (~ 250 vs 220 meV) for radial growth compared to planar growth.

μm to 100 nm regime, consistent with our intrinsic Si growth results. Incorporation of boron (B) and phosphorus (P) at high dopant concentrations is known to result in substantial segregation of B and P to the surface and a lowering of the crystalline Si growth rate in conventional large area epitaxial growth.^{24–26} Similar lowering of the growth rates were observed here in comparison to planar large area growth rates for undoped (57 ± 3 nm/min), B-doped (55 ± 3 nm/min), and P-doped (53 ± 3 nm/min) Si shells (filled vs open symbols, Figure 3). Also, the P-doped Si radial shell growth rate, for example, 11 nm/min for 1500 nm-diameter NWs, is much lower than the undoped Si radial shell growth rate of 37 nm/min for the same NW core diameter, indicating an even stronger effect on growth with electrical dopants present. Previous reports reveal that phosphorus atoms on the Si surface hinder Si adatom migration, result in lower Si growth rates and induce surface roughening due to the formation of Si clusters on Si surfaces.²⁷ Moreover, the clusters can act as nucleation sites for Si grains with crystallographic orientations different from the Si epitaxial layer. Thus, the observed remarkably low growth rate of P-doped Si radial NW shells suggests that the crystallinity of the P-doped Si radial shells may be affected by P incorporation for these small surface areas. Figure 4a–c show high-resolution TEM images of P-doped Si radial shells after growth for increasing growth times, demonstrating for the first time the existence of a dopant-dependent epitaxial critical thickness for CVD grown Si NWs. The crystallinity of the P-doped Si radial shell changes from epitaxial single crystalline for a 60 nm-thick shell (8 min growth) to defective crystalline containing stacking faults for a 130 nm-thick shell (15 min growth) to polycrystalline for a 600 nm-thick shell (40 min growth). This transition of crystallinity from epitaxial to polycrystalline Si layer growth is analogous to the critical thickness behavior for undoped large area Si planar growth at low temperatures ($< 300^\circ\text{C}$),²⁸ where the behavior originates from the blocking of Si adatom migration by hydrogen atoms occupying vacant sites on the Si surface. For the P-doped Si radial shell grown at 810°C , the hydrogen coverage is very low and the entities blocking Si adatom migration and adsorption of Si adatoms and SiH_4 molecules are inferred instead to be P atoms. Figure 4d shows the temperature-dependent critical thickness of a P-doped Si radial shell. This critical thickness increases with growth temperature from 15 nm at 710°C to 75 nm at 810°C . The inset in Figure 4d shows that the growth rate for a fixed temperature (810°C) increases significantly as the shell layer is transformed from single crystalline epitaxial growth to polycrystalline growth. This behavior is consistent with previous large area CVD Si growth studies which show faster polycrystalline Si growth compared to single crystalline Si epitaxy.²⁹ Both Si adatom migration and phosphorus incorporation during growth are thermally activated processes, and thus, the critical thickness for P-doped Si radial shell growth should be affected by growth temperature. At higher temperatures, the Si adatom migration length increases and the phosphorus incorporation coefficient decreases,²⁶ resulting in the observed increase in critical thickness with temperature. Another notable feature is that the critical thickness of ~ 75 nm at 810°C for radial shells is even thinner than that of 120 nm at only 300°C for undoped large area planar Si epitaxial growth.²⁸ Thus, in combination with the lower growth rate of radial shells for small area growth, the presence of P dopants further hinders the Si epitaxial growth, with the dopants most likely changing

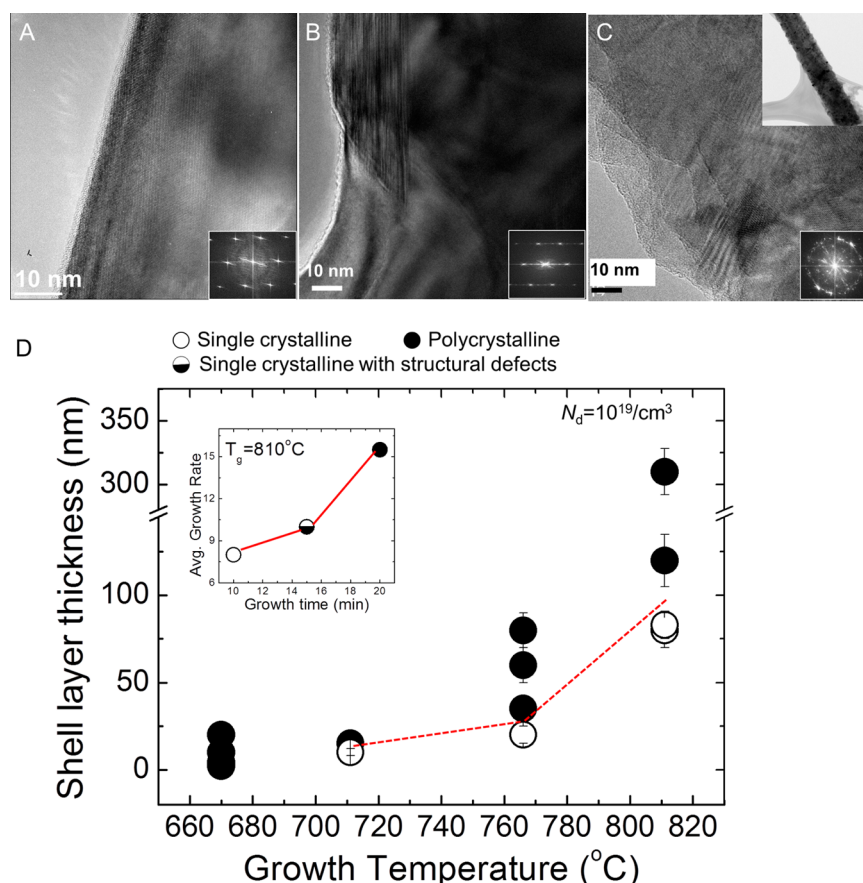


Figure 4. Images and measurements for phosphorus doped silicon radial growth showing epitaxial to polycrystalline transition with increasing shell thickness. (A)–(C) High resolution TEM images of phosphorus-doped Si radial shells for growth times of 8, 15, and 40 min, respectively, resulting in an increase in the shell layer thickness and a transition from single crystalline (A) to crystalline with low defect density (B) and to polycrystalline (C) growth. (D) Critical thicknesses for single crystalline epitaxial growth of P-doped Si shells (P concentration $\sim 10^{19}/\text{cm}^3$) as a function of growth temperature (filled symbols are polycrystalline and open symbols are single crystalline shells). Inset shows growth time (thickness) transition in growth rate from crystalline epitaxy (open circle) to defected crystalline epitaxy (half filled circle) and to polycrystalline (filled circle).

the surface adatom migration and precursor adsorption dynamics compared to planar growth.

These results place new emphasis on understanding fundamental processes at mesoscopic dimensions and provide a route to model growth rates for increased control in nanoscale device fabrication. Our observation of a size-dependent Si epitaxial growth rate and a critical thickness for crystalline P-doped Si shell growth demonstrates that in addition to surface atomic arrangements, the microscopic structuring of surface boundaries influences CVD growth at the mesoscale. Our proposed model describes this previously unknown size-dependent growth effect based on a new mechanism for CVD growth of enhanced edge desorption and implies surface diffusion lengths for silane on Si of ~ 200 nm for our growth conditions. We anticipate that the observed size effects for Si epitaxy may be relevant to other vapor phase material growth systems in the mesoscale regime. Given the high electrical doping required for many nanoscale device structures to achieve desired functionalities, the large dopant effects on Si crystal growth rates at small dimensions reported here also have important technological implications. For example, in controlled epitaxial growth for nanowire electronic and photovoltaic structures a critical thickness for crystalline growth for *n*-type doping sets a limit on shell thickness and doping order in radial junction device design. Understanding of such mesoscale growth effects is essential in the quest of

semiconductor technology to meet future design criteria and achieve atomic level control of 3D device architectures.

■ ASSOCIATED CONTENT

⑤ Supporting Information

Experimental details such as preparation of Si nanowires and planar stripes, electron microscopy of Si nano/mesostructures, Chemical vapor deposition kinetics of Si epitaxy in nano/mesoscale. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

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