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Citation: Appl. Phys. Lett. **100**, 263117 (2012); doi: 10.1063/1.4731777

View online: http://dx.doi.org/10.1063/1.4731777

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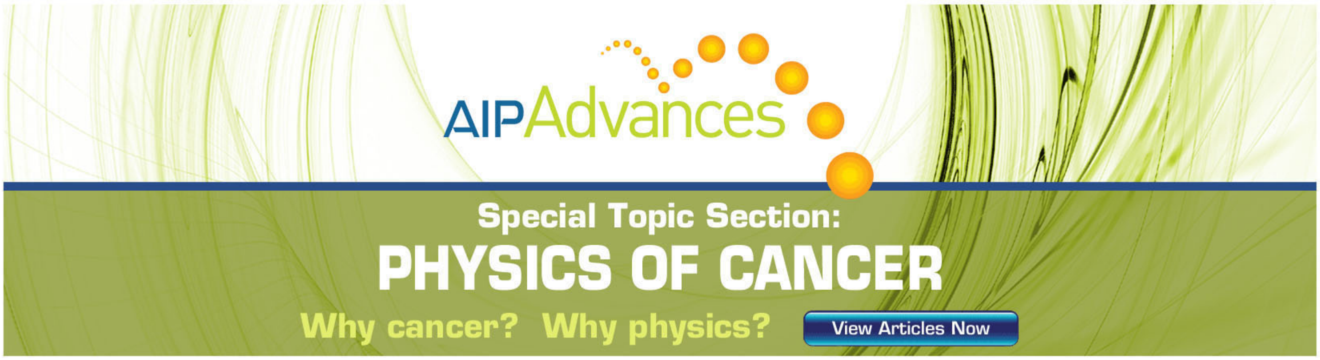

APPLIED PHYSICS LETTERS **100**, 263117 (2012)

Nanodot to nanowire: A strain-driven shape transition in self-organized endotaxial $CoSi_2$ on Si(100)

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(Received 14 May 2012; accepted 12 June 2012; published online 29 June 2012)

We report a phenomenon of strain-driven shape transition in the growth of nanoscale self-organized endotaxial $CoSi_2$ islands on Si(100) substrates. Nanodots of $CoSi_2$ grow in the square shape following the four fold symmetry of the Si(100) substrate, up to a critical size of $67 \times 67\,\mathrm{nm}^2$, where a shape transition takes place. Larger islands grow as nanowires with ever increasing length and the width decreasing to an asymptotic value of $\sim 25\,\mathrm{nm}$. This produces long nanowires of nearly constant width. The endotaxial nanostructures grow into the Si substrate with a small extension above the surface. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4731777]

Interaction of metals on silicon and the consequent growth of metal silicides are both of fundamental and technological interest, including modern silicon-based integrated circuit technologies.[1–4] In microelectronic devices, metal silicides, e.g., titanium silicide, nickel silicide, cobalt silicide, etc., are used as interconnects, Ohmic contacts, Schottky barrier contacts, and gate electrodes.[1,5,6] Synthesis and properties of nanoscale metal silicides on silicon are of tremendous current interest.[2–7,9] A bottom up approach in many cases provides self-organized nanostructures, including metal silicide nanostructures on silicon. When such self-organized single-crystalline metal silicides are grown on silicon substrates, there are two possibilities: (i) epitaxial growth on the surface of the substrate and (ii) endotaxial growth,[7,8] where epitaxial growth occurs into the substrate. In the present study we concentrate on the self-organized growth of endotaxial nanoscale silicides. Growth of endotaxial silicide nanowires has been investigated for a variety of systems.[9] Here, we report on the phenomenon of shape transition, for the endotaxial nanoscale systems, which was not reported earlier.

Self-organized epitaxial island growth usually occurs on a substrate via Stranski-Krastanov (SK) and Volmer-Weber (VW) growth modes.[10] While SK mode describes island formation on a wetting layer, the VW mode describes island formation directly on a substrate, in heteroepitaxial systems with different lattice constants. Because of the lattice mismatch between the materials of the islands and the substrate, the islands are inherently strained. In this self-organized growth, coherent islands of shapes following the symmetry of the substrate have been observed. This symmetry may be broken in many cases leading to the growth of elongated islands due to a strain relaxation mechanism causing shape-transition.[11] Such long and narrow self-organized islands, in fact, constitute quasi-one dimensional "quantum wires."[11] Elongated island formation via shape transition in the epitaxial growth of strained islands in lattice-mismatched systems has been observed.[12,13] Elongated islands can also grow due to anisotropic lattice mismatch,[14–18] the elongation being along the direction of smaller lattice mismatch. Epitaxial growth usually refers to growth on a substrate. However, there are cases where epitaxial growth occurs into the substrate; this is known as "endotaxy" as originally called by Fathauer *et al*.[8] The topic of growth of endotaxial silicide nanowires has recently been reviewed by Bennett *et al*.[9] Growth of endotaxial silicide nanowires of a variety of systems like Ti, Mn, Fe, Co, Ni, Pt, and several rare earth metals on Si(111), Si(110), and Si(100) substrates are discussed in Ref. 9. These are all lattice mismatched systems and hence the nanowires are strained. However, in none of these cases of endotaxial growth, the phenomenon of shape transition was reported. In these studies the growth of self-organized silicide nanowires has been found to follow a constant-shape growth model in which length, width, and thickness all change in proportion as the nanowire grows. Among these silicides, the case of endotaxial growth of cobalt disilicide ($CoSi_2$) nanowires on Si(111), Si(110), and Si(100) was extensively investigated by He *et al*.[7] These authors also did not observe shape transition from nanodot to nanowire or the growth of nanowires of nearly constant width, as predicted by the theory of shape transition in Ref. 11 for strained islands in heteroepitaxial growth. Compared to all other cases of endotaxial growth,[7,9] we have deposited a smaller amount of Co on Si to form endotaxial $CoSi_2$ with the expectation that we would observe growth of smaller $CoSi_2$ islands and possibly shape transition. In contrast to all previously reported endotaxial systems, we find that small self-organized endotaxial $CoSi_2$ nanoislands grow on Si(100) in the square shape following the four-fold symmetry of the Si(100) substrate. Up to a critical size of the islands, the length and the width of the islands are equal. At the critical size a nanodot to nanowire transition occurs. As the $CoSi_2$

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nanowires grow larger, the width gradually reduces and approaches an asymptotic value while the length keeps on increasing, with the aspect ratio (length/width) becoming ever larger. We have observed an aspect ratio as large as ~20:1. This indicates that even in the endotaxial growth, the shape is determined by a strain-driven energetic mechanism as in the Tersoff and Tromp model,[11] introduced for heteroepitaxial growth of strained islands. $CoSi_2$ has 1.2% smaller lattice constant compared to Si and hence epitaxy (or endotaxy) gives rise to the growth of strained islands due to lattice mismatch. Larger islands, adopting a nanowire shape, have better elastic relaxation of the island stress.

The experiments were performed in an ultra-high vacuum (UHV) system for molecular beam epitaxy (MBE) growth and *in situ* scanning tunneling microscopy (STM) experiments similar to the one in Ref. 19. The UHV chambers of MBE and STM are interconnected, and the samples are transferred from one to the other *in situ*. The base pressure in the MBE and the STM chamber was 5.2×10^{-11} mbar and 2.3×10^{-10} mbar, respectively. P-doped, n-type Si(100) wafer with resistivity of 10–20 Ω cm was used as substrates. Atomically clean Si(100)-2 × 1 surfaces were prepared by degassing the Si(100) substrate at 750 °C for about 14 h and then flashing the substrate at ~1250 °C for 1 min. The clean Si(100)-2 × 1 surface with dimmer rows was checked with STM. A PBN-crucible was used to produce Co (purity 99.9999%) atomic beams. During Co deposition, the substrate was kept at 600 °C. Co reacts with Si to form $CoSi_2$ at 600 °C.[20] Co atoms of 0.6 ML coverage were deposited at 0.2 monolayer (ML)/min (1 ML = 6.78×10^{14} atoms/cm^2) rate. Following Co deposition the sample temperature was brought down to room temperature (RT). A tungsten tip was used in STM. All STM images were recorded and current (I)-Voltage (V) measurements were made *in situ* at RT. Samples were then taken out for *ex situ* imaging using scanning electron microscopy (SEM) with a field emission gun based microscope and high-resolution transmission electron microscopy (HRTEM). Cross-sectional TEM (XTEM) specimens were prepared from the above samples in which electron transparency was achieved through mechanical thinning followed by low energy Ar^+ ion milling. The TEM characterization of the samples were done with 200 keV electrons (2010, JEOL HRTEM).

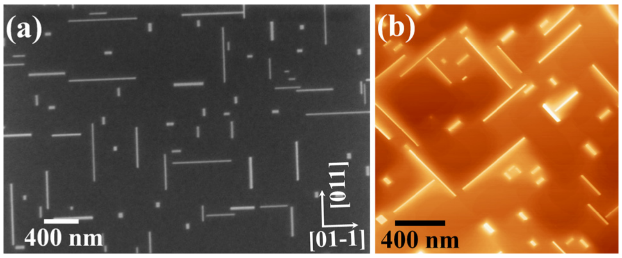

FIG. 1. Cobalt disilicide islands grown by 0.6 ML Co deposition on Si(100) at 600 °C: (a) SEM image showing small square shaped as well as elongated islands. The elongated islands grow along [011] and [01-1] directions without any preference. (b) STM image shows small square shaped $CoSi_2$ islands as well as nanowires.

Figure 1 shows cobalt disilicide islands grown on Si(100). Figure 1(a) shows a SEM image and Fig. 1(b) shows a STM image. Growth of small square and rectangular islands as well as long nanowires is observed. Elongation of the islands is in [011] and [01-1] directions without any preference. In our growth condition, nanowires as long as 800 nm have been found. We will show latter with cross-sectional HRTEM images that these islands have grown predominantly into the substrate, i.e., this is endotaxial growth; outward growth is limited to ~3 nm–5 nm. The observed shapes are discussed below and analyzed in the light of the theory of strain-driven shape transition.[11] It is evident from Fig. 1 that the island formation has followed two distinct kinds of symmetry. Measurements show that the smallest islands are square in shape, following the four fold symmetry of the underlying substrate; the 2 × 1 reconstruction of the Si(100) surface is destroyed upon Co deposition. As island area increases, square shaped islands grow up to a critical dimension, at which transition from square to a rectangular shape occurs. For very long islands, the width reduces from its critical value towards an optimal value, making the islands thinner leading to the formation of quasi-one dimensional nanowires. Thus a symmetry breaking is involved in the phenomenon of shape transition. From a large number of islands of various sizes, we have obtained a plot of island length/width versus area. Island length and width have been determined from the FWHM of the line profile of intensity on individual islands in the STM images. The plot is shown in Fig. 2 which displays shape transition.

We will resort to the theory of Tersoff and Tromp[11] in order to explain our results. According to the theory, island shape and dimensions are basically dictated by the interplay between two contributions—one from the relevant surface and interface energies and the other from elastic relaxation of the strained-islands. The energy per unit volume (E/V) of a rectangular strained epitaxial island is

$$\frac{E}{V} = 2\Gamma(s^{-1} + t^{-1}) + h^{-1}(\gamma_i + \gamma_t - \gamma_s) - 2ch\left\{ s^{-1}\ln\left[\frac{s}{\phi h}\right] + t^{-1}\ln\left[\frac{t}{\phi h}\right]\right\}, \qquad (1)$$

where s, t, and h denote island width, length, and height, respectively; $\varphi = e^{-3/2}\cot\theta$, θ being the contact angle; $\Gamma = \gamma_e \csc\theta - (1/2)(\gamma_s + \gamma_t - \gamma_i)\cot\theta$, where γ_s, γ_t, and γ_e

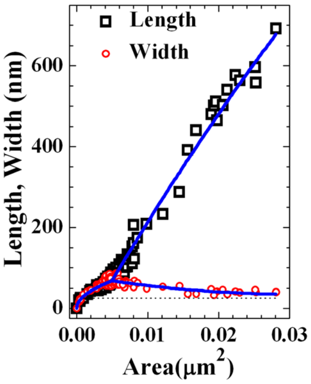

FIG. 2. The plot shows length (t) and width (s) of $CoSi_2$ islands vs. island area. The critical size at which the shape transition occurs is $s = t = e\alpha_o = 67$ nm. For longer islands the width approaches towards $s \approx 25$ nm, marked by the dashed horizontal line.

are the surface energy (per unit area) of the substrate and that of the top surface and the edge facets of the island, respectively, and γ_i is the island-substrate interface energy; $c = \sigma_b^2\,(1-\nu)/2\pi\mu$, where ν and μ are the Poisson ratio and the shear modulus of the substrate, respectively, and σ_b is the island bulk stress. The optimal tradeoff between surface energy and strain is obtained by minimization of E/V with respect to s and t treating h as constant. This gives $s = t = \alpha_o$, where, $\alpha_o = e\varphi h e^{\Gamma/ch}$. Square island shape ($s = t$) is stable for island sizes s, $t < e\alpha_o$. As soon as the island dimension exceeds its optimal value α_o by a factor e, the square shape becomes unstable and a transition from square shape to rectangular shape occurs. As the island grows, the island width tends to go back to its optimal value α_o whereas island length keeps increasing rapidly. From the plot in Fig. 2 it is seen that the critical dimension is $e\alpha_o \approx 67$ nm and the optimal value of width α_o is ~ 25 nm. From these experimentally obtained values we have calculated $\Gamma/ch = 0.11$ using the above model, taking $\theta = 25°$ and average island height (or rather depth, as shown later in XTEM images), $h = 17.0$ nm. A $CoSi_2$ square island near the critical size is shown in the STM image in Fig. 3. We find from the edge profiles (not shown) of the STM image (shown in Figs. 3(a) and 3(b)) that the contact angle of the large facets in all four directions is $\sim 25°$ indicating that the facets are {311} which are one of the low energy facets. The corners of the island are rounded. However, this does not affect the analysis. The theory in Ref. 11 assumes square shaped islands. It mentions that realistic islands may have complex shape including rounding. However, the assumption of square shape would be sufficient to capture the important feature such as size and aspect ratio.

It should be noted that values of h or θ used in the calculation shown in the previous paragraph, only change the depth of the energy minimum, while the obtained values of s and t remain unchanged. Also, in the theoretical calculation in Ref. 11 the top of the island surface has been assumed to be flat; however, when h is larger, for $\Gamma/ch \leq 0.5$ the island will have a triangular cross-section, as we observe in our case (see Fig. 4).

In the absence of any explicit theory for endotaxial systems explaining shape transition, we have used the theory of shape transition in strained heteroepitaxial islands,[11] the predictions of which are in agreement with our experimental results.

Upon deposition of cobalt atoms on the heated substrate, the (2×1) reconstruction of the Si(100) surface is destroyed and cobalt reacts with the silicon atoms to form cobalt silicide. Cross-sectional TEM images of nanowires are shown in Fig. 4. The spacing of atomic rows, as observed in cross-sectional TEM images in Fig. 4 of silicide nanowires indicates that the silicide is $CoSi_2$ and the nanowires are endotaxial. The $CoSi_2(111)//Si(111)$ interface has the lowest interface energy than the other possible interfaces. The epitaxial growth of $CoSi_2$ on Si(100) is far more difficult than that on Si(111) substrates due to the higher interfacial energy of $CoSi_2(100)//Si(100)$.[21] This prevents island growth above the surface as that would require formation of $CoSi_2(100)//Si(100)$ interface. Instead this facilitates ingrowth or "endotaxy" and drives the growth of inverted pyramidal shaped $CoSi_2$ islands with the sharp interfaces along {111} planes, which is clearly visible in cross-sectional TEM micrograph in Fig. 4. Here, $CoSi_2(111)//Si(111)$ and $CoSi_2(1\text{-}1\text{-}1)//Si(1\text{-}1\text{-}1)$ suggest that the interfaces in our system are symmetrical in square island as well as in nanowires. In Ref. 7, this type of nanowires is called rectangular islands while square islands have not been observed at all. Our present results show the formation and evolution of square islands up to a critical size ($e\alpha_o$) and a shape transition to rectangular nanowire islands, establishing a strain-driven energetic mechanism[11] for this shape transition. The critical size observed in the endotaxial growth here is also the smallest compared to the values obtained in earlier cases of shape transition in epitaxial growth.[12,13]

An interesting feature of the islands is seen in Fig. 5 which shows several height profiles of nanoislands. Their side facets have one major inclination [{311} facets as marked in Fig. 3] around $\theta \approx 25°$ which has been used in the calculation of Γ/ch. There is another feature of much smaller gradually varying inclinations. This feature follows a staircase-like structure. The average terrace width is ~ 5 nm. The average step height of the staircase is ~ 0.26 nm, which is equal to (200) planar spacing of $CoSi_2$. In other words, this is the separation between Co planes along the [100] direction. The step heights and the $CoSi_2$ structure are shown in Fig. 5(c). Whether these step-like features are related to the recently reported geometrical frustration in nanowire growth,[22] which produces sawtooth faceting of nanostructure sidewalls, will be explored in future. The staircase-like

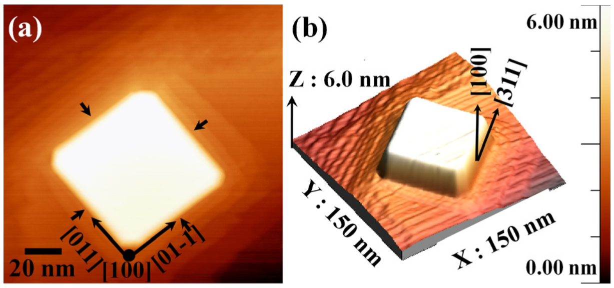

FIG. 3. STM images of one square island of approximately the critical size. Sample bias = 1.6 V and tunneling current = 0.2 nA; (a) longer arrows show the crystallographic directions of the island and shorter arrows indicate facets, (b) three dimensional plot of the island in (a), measured angles of the facets with respect to the (100) plane indicates that the facets are {311}.

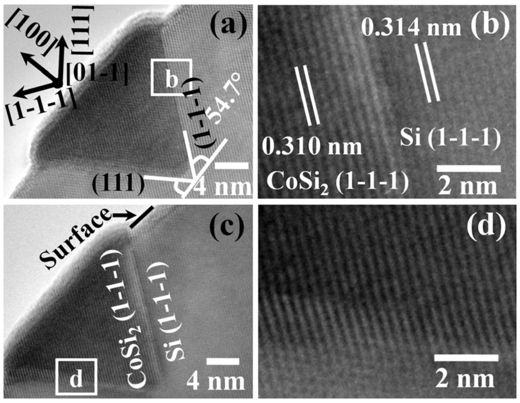

FIG. 4. XTEM images showing two nanowires: (a) crystallographic orientations of the nanowire, (1-1-1) and (111) planes are shown, (b) enlarged portion in (a) marked "b," showing the $CoSi_2(1\text{-}1\text{-}1)//Si(1\text{-}1\text{-}1)$ interface and $CoSi_2(1\text{-}1\text{-}1)$ and Si(1-1-1) planes, (c) similar image as in (a) for another nanowire, (d) enlarged portion in (c) marked as "d."

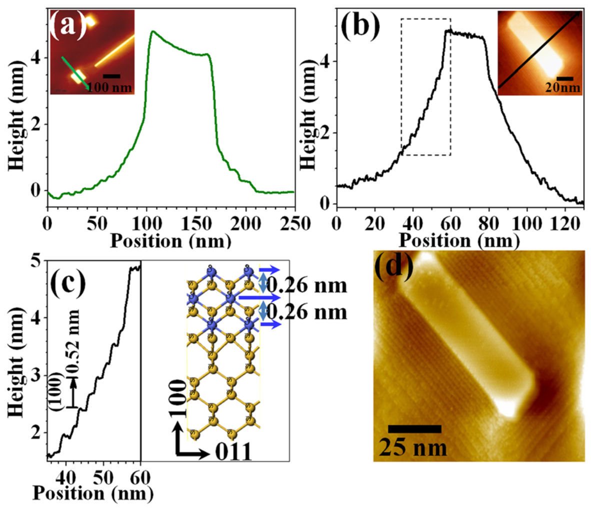

FIG. 5. STM images show staircase-like edge facets of the islands of different sizes, such as those in the image in (a). This staircase structure extends up to a large distance from the main body of the nanostructure. Height profiles of two islands are shown in (a) and (b). The enclosed portion in (b) is shown in (c) along with the schematic of the $CoSi_2(100)/Si(100)$ structure. Blue (darker) and yellow (lighter) spheres denote Co and Si atoms, respectively. Spacing between consecutive Co atomic planes (0.26 nm) agrees with the experimental observation. These staircases of atomic-step are also seen in the STM image in (d).

feature has an average inclination of ∼5°. There are no low-energy facets at this inclination. As mentioned in Ref. 22, kinetic factors could in principle allow growth of a sidewall with "forbidden" orientation, such as a sidewall consisting of a vicinal facet with a staircase of atomic steps. This is what we observe here.

It is known that a Schottky barrier is formed at the $CoSi_2/Si$ interface.[23] We have investigated the formation of Schottky barrier for our nanoscale $CoSi_2/Si$ interfaces. We have carried out scanning tunneling spectroscopy [Current (I)-voltage (V)] measurement on $CoSi_2$ islands. In this measurement current flows from the W-tip to the Si substrate or vice versa via tunneling through a $CoSi_2$ island. Thus the presence of a Schottky barrier at the $CoSi_2/Si$ interface, as one would expect, is revealed in the I-V curve (not shown here). The endotaxial growth of $CoSi_2$ nanoislands and nanowires and the consequent nanoscale Schottky barrier opens up new possibilities in nanoelectronics with the introduction of self-organized semiconductor-metal-semiconductor (SMS) junctions. SMS junctions are used in permeable base transistors (PBTs), for high speed electronic devices.[24,25] $Si/CoSi_2/Si$ vertical structures formed by growing a $CoSi_2$ layer on Si and a Si layer on top have been used for PBTs.[25] Our results on self-organized nanoscale Schottky diodes offer the possibility to fabricate lateral PBTs without the necessity of artificially sandwiching a metal layer between two semiconductors. Partially embedded $CoSi_2$ nanostructures in Si offer the possibility of fabricating nanoscale lateral PBTs if we isolate the upper layer of the Si substrate (∼10-20 nm thick) by forming a sub-surface insulating layer of SiO_2 as in Silicon-on-Insulator (SOI) technology.[26] This is done by implanting the Si substrate with oxygen ions.[26,27] The conceptual steps are shown schematically in Fig. 6. We illustrate in Fig. 6 a possible method for the fabrication of lateral permeable base nanoscale transistors using such structures. This PBT would work like a lateral field effect transistor (FET).

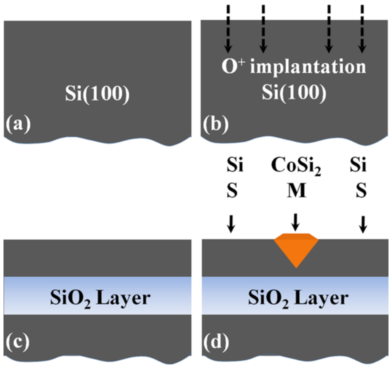

FIG. 6. Schematic illustration of a self-organized semiconductor-metal-semiconductor structure fabricated on a silicon-on-insulator substrate that can serve as a lateral PBT. (a) a Si substrate, (b) oxygen implantation to incorporate O at a desired depth, (c) annealing causes O to react with Si and form a buried SiO_2 layer, (d) endotaxial metallic silicide (M) and semiconductor (S) on sides forms lateral PBT structure.

In conclusion, we have presented experimental results showing shape transition from nanodot to nanowire in endotaxial growth of strained islands on Si. Although we have shown the shape transition in the $CoSi_2$ islands, we believe that this should be observed in other systems. The results are consistent with the theory of shape transition based on a strain-driven energetic mechanism. Earlier, shape transition was observed in epitaxial growth. Here we have demonstrated that shape transition also occurs in endotaxial growth. Small nanoscale islands of square shape have been found to grow up to a critical dimension $e\alpha_0$. A shape transition from nanodot to nanowire occurs at this critical size and larger islands grow in rectangular shape with a rapid increase in length and a reduction in width, which approaches the optimum value α_0. In the present example $\alpha_0 = 25$ nm. That is, long nanowires have approximately the same width. Our results also reveal the smallest critical size and narrowest nanowires compared to other cases where shape transitions have been observed. It has been shown that these endotaxial islands function as nanoscale Schottky diodes, and the possibility of fabrication of nanoscale lateral permeable base transistors with such structures has been illustrated.

Jagadish Chandra Mahato and Debolina Das are supported by a CSIR Fellowship [(09/080(0674)/2009-EMR-I] and [09/080(0725)/2010-EMR-I], respectively.

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