Tuning the length/width aspect ratio of epitaxial unidirectional silicide nanowires on Si(110)-16 × 2 surfaces

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Abstract

The reactive deposition epitaxy growth of self-organized cobalt silicide nanowires (NWs) on clean Si(110) surfaces has been investigated by in situ scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) as well as by scanning electron microscopy (SEM). Half a monolayer of cobalt was deposited on the Si(110) surfaces at ~600 °C substrate temperature. Following cobalt deposition, the substrates have been annealed for different durations. Cobalt forms aligned cobalt disilicide nanowires upon reaction with the silicon substrate, following the twofold substrate symmetry. With increasing duration of annealing, the NWs have been found to grow with larger aspect ratio (length/width), eventually producing narrower NWs. These self-organized unidirectional NWs of sub-hundred nanometer width and ~4–7 nm height produce a Schottky barrier with the silicon substrate and are expected to find applications in nanoelectronic devices.

1. Introduction

Achieving the control on the dimension of the nanostructures in the bottom-up approach of nanotechnology is the coveted goal for their importance in possible application in nanoelectronic device fabrication and also for fundamental research in surface and interface science [1–4]. Self-organized nanostructures have attracted much interest and attention since it promises to overcome the limitations on resolution and throughput of the conventional lithography technique for novel device fabrication at nano scale [5]. Among all self-organized methods, the growth of single-crystal epitaxial silicide nanowires (NWs) has been widely investigated in recent years for its potential applications in nanoelectronics [6–13]. In addition, the studies on electrical characteristics of epitaxial silicide NWs have shown interesting characteristics [14–17]. Growth of epitaxial and endotaxial self-organized cobalt silicide nanostructures on silicon substrates of various crystallographic orientations is of tremendous current interest [11, 12, 14, 15, 18–21] and new phenomena in self-organized nanowire growth are still being discovered [22–25]. The main drawback in applying self-organized silicide NWs to practical devices is that it is challenging to control their spatial location, morphology as well as size distribution. The formation of silicide NWs preferably at the step edges on Si surface has helped overcome partially the spatial location issue [26, 27]. However, the control over the morphology of NWs is more problematic. Chen et al have combined reactive deposition epitaxy and nitride mediated epitaxy to grow epitaxial Fe and Ni silicide NWs with controlled sizes [28]. They used Si 3N 4 as buffer layer to control the amount of Fe or Ni that diffuses and reacts with the substrate silicon to form silicide NW. Recently, we carried out detailed investigations of CoSi 2 nanowire growth on Si(110) surfaces [29]. Shape transition of epitaxial islands was proposed to grow long narrow NWs, even quantum wires [30]. Growth of such elongated narrow NWs of CoSi 2 via shape transition was observed on Si(100) surface, which has fourfold symmetry [19]. However, for Si(110) surface, the underlying twofold symmetry guides the unidirectional growth of NWs. Earlier we have extensively investigated the epitaxy of CoSi 2 NWs on Si(110) and their other crystallographic details [29]. From these studies we know that these
nanowires grow endotaxially on Si(110) substrates. Here, we focus on another aspect, namely the control of the dimension of the already grown self-organized CoSi$_2$ nanowires. We show that the length/width aspect ratio of the NWs can be readily controlled by tuning the post-deposition annealing time.

2. Experimental details

Clean Si(110)-16 × 2 surfaces were prepared by degassing the substrate at ∼700 °C for 14–16 h and then flashing the sample at ∼1250 °C for one min under UHV condition (∼5 × 10$^{-10}$ mbar). We raised the substrate temperature to ∼1250 °C, kept there for one min, and then cooled it down to ∼850 °C and kept at that temperature for 30 min and then allowed the substrate to attain the room temperature by slowly decreasing the sample heating current to zero. The clean surface thus prepared possesses domains with Si(110)-2×2 reconstructed areas. Half a monolayer (ML) (1ML = 9.59 × 10$^{14}$ atoms/cm$^2$) Co was deposited on these substrates at ∼600 °C to grow CoSi$_2$ NWs in a molecular beam epitaxy (MBE) growth chamber. The substrates were annealed for different durations after Co deposition at that substrate temperature. The morphology of these cobalt silicide structures and their length/width aspect ratio were investigated by in situ scanning tunneling microscopy (STM) and ex situ scanning electron microscopy (SEM). The in situ scanning tunneling spectroscopy (STS) measurements were carried out on the silicide NWs as well as on the clean silicon surface. The UHV STM equipment (Omicron Nanotechnology) is connected to the MBE growth chamber, which allows in situ STM and STS measurements. The samples were taken out from the MBE-STM system and then the ex situ SEM measurements were carried out with a field emission gun based scanning electron microscope (ZEISS SUPRA 40) operated at 5.0 keV. We prepared a set of three samples where the substrates were annealed after Co deposition for different durations. The annealing after Co deposition was carried out at ∼600 °C in the UHV environment of the MBE chamber for a time period of 10 min (Sample A), 40 min (Sample B) and 70 min (Sample C). All the samples were annealed at ∼600 °C for 25 min prior to Co deposition.

3. Results and discussion

3.1. Effect of prolonged annealing on the average length/width aspect ratio of the silicide nanowires on Si(110) surfaces

Si(110) surface undergoes wide variety of complex surface reconstructions, such as 5 × 1, 5 × 4, 16 × 2, 5 × 8 etc. The Si(110)-16 × 2 surface reconstruction has been investigated by a number of groups [31–40]. Various authors have interpreted this surface reconstruction with models of octets [37], pentagons [38], centered stretched hexagons [39], or adatom-tetramer-interstitial (ATI) [40]. The clean Si(110)-16 × 2 reconstructed surfaces have been prepared to grow cobalt disilicide nanowires on them in a molecular beam epitaxy growth chamber under ultrahigh vacuum condition.

Figures 1(a)–(b) show the STM images of a clean Si(110) reconstructed surface. The reconstructed surface predominantly contains (16 × 2) reconstruction along with some (5 × 8) reconstructed areas. The image shows protrusions of parallel chains that are arranged up and down alternatively with a monatomic height difference. The separation between successive up or down chains is ∼5 nm. The atomic chains of the Si(110)-16 × 2 reconstructed surface are aligned to the [-112] and [1-12] directions as shown in figure 1(b).

Figures 2(a)–(c) show typical SEM images of Sample-A, Sample-B and Sample-C, respectively. Sample-A, Sample-B and Sample-C, have been annealed for 10 min, 40 min and 70 min after Co deposition at ∼600 °C, respectively. We have calculated the length (L) and the width (W) of a large number of nanowires for Sample-A, Sample-B and
Sample-C. The width has been measured from the full width half maximum of the line profile across the nanowire in the SEM images. Similarly, the length has been calculated for the nanowires for all three samples. We plot the length/width (L/W) aspect ratio frequency distribution for the three cases (figures 2(d)–(f)), obtained from many SEM images. The data have been fitted with a Gaussian function. From the distribution plot, we find that the maxima of the aspect ratio distribution increase with the increase of annealing time. This is evident from the plots in figures 2(d)–(f).

For sample-A, sample-B and sample-C, the most probable aspect ratio are at ∼7.4, 11.4 and 12.6, respectively. As the annealing time increased from 10 min to 70 min the average NW width decreases from ∼60 nm to ∼35 nm.

From figures 2(a)–(c), we notice two major aspects - NWs become narrower and their density increases with longer duration of annealing. We have plotted the average length/width (L/W) aspect ratio versus the annealing time in figure 3, which shows that the L/W aspect ratio of the nanowires increases with the annealing time. The average width of the NWs of 70 min annealed sample (Sample-C) is much narrower (35 nm) than that of the 10 min annealed sample (60 nm) (Sample-A). The decrease of the NW width and increase in nanowire length results in the increase in length/width aspect ratio of the nanowires. This indicates that upon prolonged annealing material from the NW side-walls diffuse to the NW ends. This type of mass diffusion has also been
earlier modeled for the silicide nanowire elongation on Si(100) surfaces [41]. This observation has also been supported by the SEM as well as STM measurements in the present case.

3.2. Effect of prolonged annealing on Nanowires areal density

The areal density of the NWs has been calculated for the three samples. The areal density of NWs (i.e., number of nanowires per cm²) increases with the annealing duration as revealed from the nanowire density versus post-deposition annealing time plot shown in figure 4. In the sample-A i.e., in the sample which is annealed for 10 min after cobalt deposition, nanodots have been formed on the surface along with the nanowires (figure 2(a)). From this plot in figure 4, we can see that the nanowire density increases with the post deposition annealing time. This indicates that the small silicide nanodots or clusters, which were present on the surface, grow or agglomerate to form new NWs upon further annealing (like those in Sample-B and Sample-C). The nanodots are visible on the surface as shown in figure 2(a). A small segment of the area containing two nanodots have been magnified for clarity and it has been shown in the inset at the right bottom corner in figure 2(a). In figure 2(a), there are smaller nanodots, which are not visible in this scale of magnification. As annealing is continued for a longer duration these nanodots disappear by forming new NWs. This is consistent with the increase of NW density with annealing time (figure 4). Bennett et al have investigated the CoSi₂ NW growth in situ at...

Figure 3. The average length/width (L/W) aspect ratio CoSi₂ NWs as a function of post-deposition annealing time.

Figure 4. Variation of CoSi₂ NW density with post-deposition annealing time.
\[ T \approx 750 \degree C - 850 \degree C \] and found the length and width evolve with time as \( t^{1/3} \) [12]. They provided continuous supply of cobalt during the growth of silicide NWs. In our case, we deposited a fixed amount of cobalt on the heated substrate and varied only the post-deposition annealing time. During this period of sufficient annealing, the material of the silicide diffuses from sides towards the length of the NW overcoming the corner diffusion barrier. This might be the possible reason for the decrease of width of NWs. Chu et al have found that if the aspect ratio of the NW is increased keeping the height to be same, the strain energy decreases [42]. In our case of silicide growth on silicon, strain reduction appears to be the driving force for the narrowing and elongation of the NWs.

Figure 5 shows the NW length and width versus area plot for the Sample-A, Sample-B and Sample-C. Two different length scales have been used for the length and the width in figure 5. When the width data are plotted in the same length scale as that of the length data (not shown here) they appear to fall on a horizontal straight line, indicating

![Figure 5](image-url)
that the widths of the NWs are nearly the same irrespective of their lengths. The details of the variation of width with NW area are seen in figure 5. The plot for Sample-A in figure 5 shows both nanodots (the group with small length and small width values) and nanowires. (For the growth of CoSi2 NWs on Si(100) substrates, a nanodot-to-nanowire shape transition was observed [19]). For Sample-B and Sample-C we do not observe this group of nanodots. Apparently they have transformed into NWs. Additionally, the width of the NWs become smaller (∼35 nm) compared to Sample-A (∼60 nm). Comparison between Sample-B (40 min annealed) and Sample-C (70 min annealed) shows that longer annealing has not produced further narrowing of NWs. Narrowing of NWs indicates a reduction of strain and apparently an asymptotic limit [30] has been reached. Annealing for longer duration (Sample-C) has rather produced a more uniform width distribution of NWs. NW growth under longer duration of annealing brings the nanowires thermodynamically nearer to the equilibrium condition. The result can be explained in the light of the general concept of the theory of shape transition by Tersoff and Tramp [30]. According to the theory, epitaxial island shape and dimensions are basically dictated by the interplay between two contributions—one from the relevant surface and interface free energies and the other from elastic relaxation of the strained-islands. The optimal tradeoff between surface free energy and strain is obtained by minimization of energy per unit volume of the elastically strained island with respect to its length and width treating height as constant. As soon as the island dimension exceeds an optimal value the symmetry-determined shape becomes unstable and a transition from this shape to an elongated structure occurs. On a threefold substrate, such as Si(111), this transition occurs from equilateral triangular to trapezoidal shape [43]. On a substrate of fourfold symmetry, such as Si(100), the transition occurs from square to rectangular shape [19, 30] leading to narrow nanowire for larger islands with the nanowire width approaching an asymptotic value. On the other hand on a substrate of twofold symmetry, such as Si(110), rectangular islands appear to grow from the beginning [29, 44]. As the islands grow into nanowires, their widths tend to attain an asymptotic value [29, 44, 45]. No further reduction in the width of the nanowires can be achieved even when they grow in length. Li et al [46] also have shown theoretically that in heteroepitaxial growth when the step energies are isotropic, an island adopts an isotropic shape at small sizes and transforms into an anisotropic (elongated) shape beyond a critical size, as observed in our earlier studies of CoSi2 growth on Si(100), where small square-shaped islands undergo a shape transition at a critical size and large islands are nanowires approaching an asymptotic width [19]. However, for anisotropic step energies, as one would have for growth on a Si(110) substrate, the island always has an anisotropic shape [length (l) ≠ width (w)], but its aspect ratio increases continuously with increasing island size as the strain energy becomes a more significant contribution to the total free energy. For the sample annealed for the shortest duration, Sample-A, we notice more scatter in the size distribution (figure 5); although most of the islands are nanowires, there is a group of smaller islands which also have l ≠ w. The larger group of islands in Sample-A, as well as practically all islands in Sample-B and Sample-C, are nanowires with their aspect ratio increasing with island size.

In the present case of CoSi2 growth on Si(110), the biaxial misfit strain is isotropic as both CoSi2 and Si have cubic structure; however, step energies are anisotropic (E1 ≠ E2). This condition leads to an anisotropic shape even for very small islands [46], unlike the isotropic to anisotropic shape transition observed for growth on Si(111) [43] and Si(100) [19] surfaces. An anisotropically strained island, on the other hand, has always an anisotropic shape, elongating along the less-strained direction and adopting a narrow width in the more-strained direction [47]. This has been observed for the growth of AuSi on Si(110) [44, 45], where misfit strain is smaller along the [−1 10] direction and larger along the [0 0 1] direction; as a consequence long narrow islands grow along the [−1 10] direction with a practically constant narrow width along the [0 0 1] direction. Essentially, in all cases, the width of the nanowires is determined by the strain, the larger the strain the narrower is the width.

3.3. STM measurement of silicide nanowires

We have also investigated the NW structures by STM and STS. Figure 6(a) shows a STM image from Sample-C. The NWs are all aligned along the [1−10] direction. The line profile across NWs along the marked line in (a) is
shown in figure 6(b). From this height profile it is seen that the NW height lies in the range of 3.8–7.0 nm. The height distribution of fifty nanowires, obtained from different regions of the sample (not shown here), has provided the average height of 5.2 nm with a standard deviation of ±1.1 nm. In conjunction with the results in figure 5, we find that the NWs of varying length have a nearly constant width and height.

3.4. Electronic property of silicide nanowire-silicon surface junction

We have probed the electronic transport behaviour of the NWs by STS. Figure 7 shows a typical tunneling current versus bias voltage plot recorded on the silicon surface as well as on the cobalt disilicide NW flat top. The tunneling current-voltage measurements were carried out with set point $V_g = 1.9V, I_t = 0.2$ nA. From the plot it is clear that the silicide NW shows rectifying character due to metal-semiconductor junction formation at the NW/Si(110) interface. This type of cobalt-disilicide/silicon junction may be utilized for the Schottky diode at nanoscale.

4. Conclusions

Self-organized endotaxial CoSi$_2$ NWs have been grown on Si(110) substrates by reactive deposition epitaxy. The width of the NWs can be decreased and the length/width aspect ratio of the NWs can be significantly increased by post-deposition annealing. By prolonged post-deposition annealing narrower nanowires of uniform width and height can be obtained. Theoretically, epitaxial nanowires reach an asymptotic width, which is determined by the amount of lattice mismatch and the consequent strain [30]. For the CoSi$_2$/Si system this limiting width was found to be around 25 nm for CoSi$_2$ nanowires on Si(100) substrates [19]. In the present case, a similar nanowire width has been obtained in a prolonged post-deposition annealing.

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