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Directional scrolling of hetero-films on Si(1 1 0) and Si(1 1 1) surfaces

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Abstract

Freestanding micro-tubes were obtained by directional scrolling of SiGe/Si/Cr and SiGe/Si hybrid- and hetero-structures on Si(1 1 0) and Si (1 1 1) substrates. In addition, on a Si(1 1 1) surface, helical nanobelts and vertical structure were achieved too. Compared to a Si(0 0 1) substrate, hetero-films on Si(1 1 0) and Si(1 1 1) substrates present different preferred scrolling directions for tubes resulting from the strong anisotropic underetching rate in directions parallel to the substrates surface. The design rules for the fabrication of micro- and nanotubes on Si(1 1 0) and Si(1 1 1) substrates are elucidated. The flexibility in the design is promising for applications in micro- and nano-electromechanics.

Keywords: Si(1 1 0); Si(1 1 1); Micro-tube; Scrolling; Anisotropic underetching

1. Introduction

The methodology to scroll strained hetero-structures to form tubes, helices and other 3D nano-objects is promising for a variety of devices in nanotechnology [1–4]. The self-scrolling technique is based on the strain relieved in hetero-films when they are detached from the substrate by selective wet etching. By this rolling up method, several different semiconductor material systems such as SiGe/Si and InGaAs/GaAs have been successfully used to fabricate micro- and nano-structures as well as semiconductor/metal hybrid systems. The technology offers a large flexibility in materials and design of the 3D structures, thus tubes with a diameter varying from several nanometers to several micrometers have been fabricated [2]. The details of the scrolling process are determined by the anisotropy of Young's modulus as well as of the etchant used to lift the hetero-strucutre [4]. Si surface with (1 1 0) and (1 1 1) orientations are frequently used for MEMS/NEMS [5,6]. A pronounced anisotropy of Young's modulus is found in directions parallel to the Si(1 1 0) surface [7]. The main advantage of using Si(1 1 1) as substrate is that the etching rate in the $\langle 111 \rangle$ direction is 7.5 times lower than that in the $\langle 110 \rangle$ direction [8]. The latter permits a rapid underetching in the $\langle 110 \rangle$ direction of a mesa holding the Si/SiGe bilayer. Thus the requirements on the etching selectivity between a SiGe/Si bilayer and its substrate is less demanding compared to processes using Si(0 0 1) as a substrate [2]. These peculiarities of the (1 1 0) and (1 1 1) substrates make them of particular interest to study the fabrication of nano-objects using the scrolling of hetero-structures. The purpose of this paper is to discover the scrolling behavior of SiGe/Si and SiGe/Si/Cr semiconductor and hybrid 3D freestanding structures on these two important surfaces. The acquired insights are expected to direct to new approaches in Si based nanotechnology for the fabrication of sensors, actuators and MEMS.

2. Experiment

The SiGe/Si hetero-structures were epi-grown by ultra-high vacuum chemical vapor deposition (UHV-CVD) on low resistivity 4″ Si(1 1 0) and Si(1 1 1) substrates. The Cr layer was deposited by electron beam evaporation in a high vacuum deposition system. Details of the SiGe/Si and

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SiGe/Si/Cr pattern fabrication and applied wet etching technology for the scrolling process have been described elsewhere [9]. The structures deposited on Si(1 1 0) substrates consist of SiGe/Si/Cr layer stacks with a thickness of 5/4/10 nm, respectively. The SiGe/Si/Cr layer sequence deposited on Si(1 1 1) substrate contains thicknesses of the individual films of 3/4/13 nm, respectively. The Ge concentration of all of the above SiGe films is about 30% Ge. On a Si(1 1 0) oriented substrates, mesas long sidewalls aligned along the [0 0 1] and [1 −1 0] direction as well as in different directions ranging from 13° to 33° off the [1 −1 0] were fabricated. On Si(1 1 1) substrates, different patterns were designed to test the scrolling direction of both helical coils and tubes. The tube formation was investigated using triangular shaped mesa patterns aligned perpendicular to the ⟨1 1 2⟩ direction, and directions off from the ⟨1 1 2⟩ by 10° to 350° in steps of 10°. In the fabrication of helices and vertical structures [9,10] on Si(1 1 1) surface, stripe patterns with 1 or 2 μm width and "T"-shaped patterns were used respectively. In addition, wagon wheel shaped patterns [11] were designed for both types of substrates to investigate the dependence of the lateral underetching rate on the crystallographic orientation of the mesa. For this determination of the rate of underecthcing, a 15 nm thick Cr film was patterned and the silicon underneath was etched in 3.7% NH_4OH. For quantitative analysis, the widths of the freestanding underetched Cr membranes were measured in a field emission scanning electron microscope.

The hybrid- and hetero-structures were fabricated into 3D micro-structures by underetching the mesa in 3.7% NH_4OH aqueous solution either at room temperature (300 K) or at 350 K. Finally, the samples were rinsed in deionized water and subsequently dried by pulling them out of boiling isopropyl alcohol [12].

3. Results and discussion

The SEM images in Fig. 1 show the micro-tubes produced from a "butterfly"-shaped pattern (see Fig. 1 inset) using a Si(1 1 0) substrate. The long side of the pattern is either aligned along the direction of ⟨1 −1 0⟩ (Fig. 1a) or ⟨0 0 1⟩ (Fig. 1b). The diameter of the tubes covered with and without 10 nm of Cr is about 1.9 μm and 0.9 μm, respectively. Although the ⟨0 0 1⟩ direction has the smallest value of Young's modulus, the scrolling direction of tubes on this pattern is along ⟨110⟩ and not along ⟨0 0 1⟩ directions. It implies that Young's modulus is not the dominating factor deciding on the scrolling direction. The anisotropic underetching of the (1 1 0) substrate leading to fast etching rates in the ⟨110⟩ direction and to small rates in the ⟨0 0 1⟩ direction determines the scrolling direction. At the sidewalls aligned along the ⟨1 −1 0⟩ directions (Fig. 1a) rapidly inclined {1 1 1} planes form during etching. It is well known that {1 1 1} planes are slow etching planes if 3.7% NH_4OH aqueous solution or other alkaline etchants [11] are used. Consequently, the underetching rate in the ⟨0 0 1⟩ direction is very small and the scrolling of the hetero-films along this direction is suppressed. In contrast, sidewalls aligned along the ⟨0 0 1⟩ directions are underetched rapidly and the tubes form with their axis along the ⟨0 0 1⟩ direction. Thus, the fast underetching in the ⟨1 −1 0⟩ direction defines the preferred scrolling direction. In contrary, on a Si(0 0 1) surface the {1 1 1} planes intersect with the surface along ⟨011⟩ and mesa sidewalls aligned along the [0 1 0] and [0 0 1] directions will be underetched leading to tube formation, supported by the lowest value of Young's modulus.

To investigate the detailed anisotropic etching rate profile Si(1 1 0) in a 3.7% NH_4OH solution wagon wheel shaped patterns were designed. Fig. 2a shows the speed of underetching in dependence on all directions for Si(1 1 0) substrates after etching at room temperature. The maximum etch rate on Si(1 1 0) is about 11 μm/h in directions ±22° off ⟨1 −1 0⟩ which leads to the fastest scrolling speed. For directions with faster underetching rates than ⟨0 0 1⟩ and smaller Young's modulus than that of the ⟨1 −1 0⟩, preferred scrolling directions can be expected. This prediction is confirmed by the experiments. Indeed tubes containing SiGe/Si/Cr and SiGe/Si layer sequences can be rolled up easily when their sidewalls are aligned from about 13° to about 33° off ⟨1 −1 0⟩. Some examples are shown in Fig. 3.

Among different Si substrates orientations, Si(1 1 1), the one of highest symmetry, is the only one having an almost

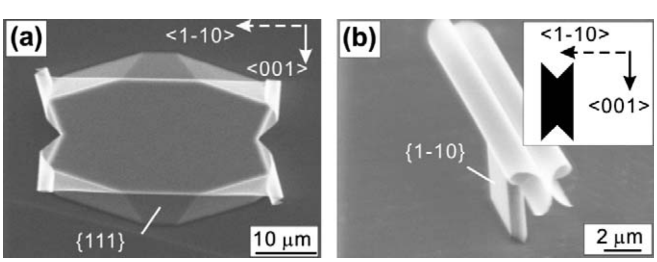

Fig. 1. SEM images of the SiGe/Si/Cr micro-tubes fabricated on Si(1 1 0) surfaces. (a) Mesa pattern aligned along ⟨1 −1 0⟩ direction, etched at 300 K. (b) Mesa pattern aligned along the ⟨0 0 1⟩, etched at 350 K. Inset illustrates the "butterfly" shape of the mesa for alignment along the ⟨0 0 1⟩ direction.

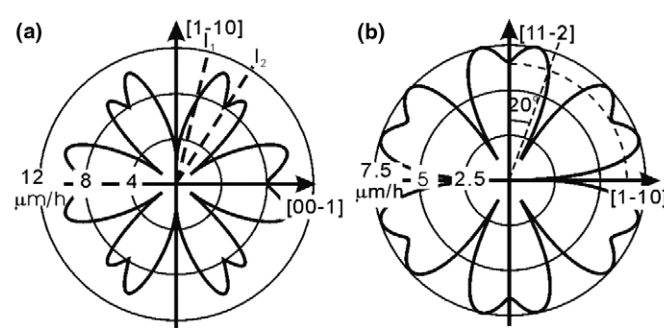

Fig. 2. Dependence of the lateral underetching speed on the crystal orientation for directions (a) parallel to the Si(1 1 0) and (b) parallel to the Si(1 1 1) surface for etching in 3.7% NH_4OH aqueous solution at 300 K. (a) l_1 is 13° off [1 −1 0] and l_2 is 33° off [1 −1 0].

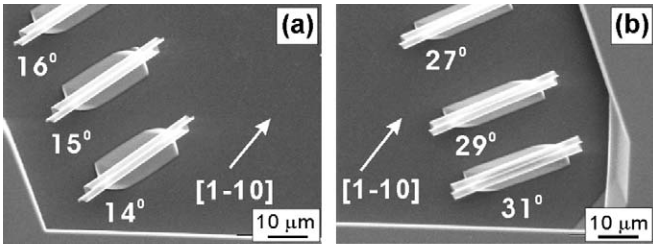

Fig. 3. SEM images showing freestanding tubes on a Si(1 1 0) surface after etching in 3.7% NH_4OH solution at 300 K. (a) The numbers indicate the misalignment towards the [1 −1 0] orientation, which is indicated by the white arrows in (a) and (b).

isotropic Young's modulus on its surface [7]. Hence, it can be expected that the preferred scrolling direction will also strongly depend on the anisotropic underetching rate and only weakly on the anisotropy of Young's modulus. Fig. 2b depicts the dependence of the underetching rate on the crystallographic orientation for a Si(1 1 1) wafer, which reflecting the sixfold symmetry. Three {1 1 1} planes intersect the (1 1 1) surface with three ⟨110⟩ directions. Our wagon wheel pattern test shows that the fastest lateral underetching direction is several degrees (about 10°) away from ⟨1 1 2⟩ (Fig. 2b). In contrast, the ⟨110⟩ directions have the slowest lateral underetching speed again due to the formation of {1 1 1} planes, which will block the scrolling of the hetero-films. Fig. 2b indicates that patterns designed for scrolling in directions close to the ⟨1 1 2⟩ directions should easily roll up. Fig. 4 shows that SiGe/Si/Cr micro-tubes can be formed on a Si(1 1 1) surface in expected directions when their scrolling direction is no less than 10° off from the ⟨1 1 2⟩ direction. The fabrication of helical structures on Si(1 1 1) substrates is less favorable due to the less anisotropy of Young's modulus [7] and the threefold symmetry of the crystal orientation in the plane of the surface. Even stripes have the same orientation and shape, various helicity can be observed when helices form. However, this feature allows to forming rings from narrow mesa lines oriented in various directions on the surface. Vertical ring structures can be produced by forcing the rings to tip over using "T"-shaped mesa structures (see Fig. 5b). The vertical rings, depicted in Fig. 5a, are fabricated from SiGe/Si/Cr (3/4/13 nm, respectively) layer stacks resulting in a wall thickness of the rings of 20 nm. The width of the rings, which is the height of the vertical rings is lithographically defined by the design of the mesa to 300 nm, resulting in

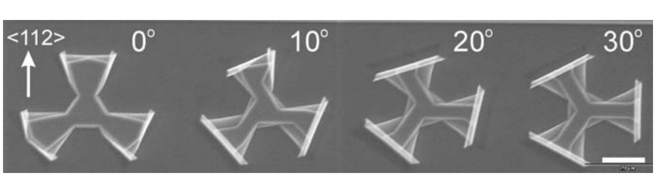

Fig. 4. SEM image of threefold symmetrical patterns employed to test the preferred scrolling direction on Si(1 1 1) substrates. In the top right corner of each pattern, the misaligned angle to ⟨1 1 2⟩ (white arrow) is marked. The scale bar is 10 µm.

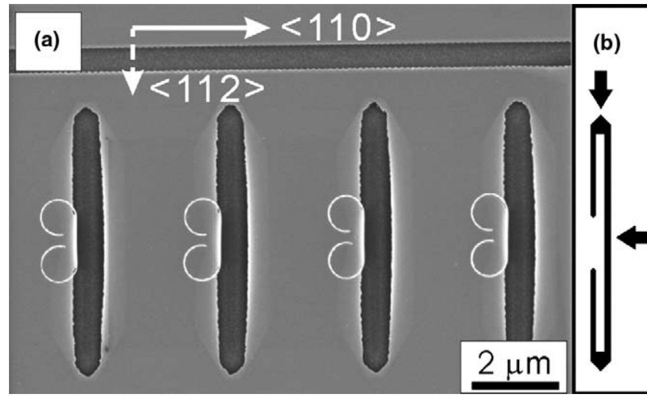

Fig. 5. (a) SEM top-view image showing an array of vertical ring structures on a Si(1 1 1) surface. (b) The initial "T" shape mesa pattern used to fabricate the vertical structure. The black arrows indicate the scrolling directions of the hetero-films.

an aspect ratio of 15 vertical walls of the rings shown in Fig. 5a. Those metal-coated vertical walls are of large mechanical strength and have atomically smooth sidewalls, which makes them suitable candidates to be used as masters containing nano-structures for hot embossing nano-replication technology. The Cr top layer can be removed easily by RIE with Cl_2 and CO_2. In this case, the aspect ratio increases and the radii of the rings decrease.

4. Conclusions

In summary, the preferred scrolling direction of the 3D micro- and nano-objects on Si(1 1 0) and Si(1 1 1) surfaces were compared to those on Si(0 0 1) substrates. It has been found that on Si(1 1 0) and Si(1 1 1) substrates, the scrolling direction of micro-tubes is dominated by the anisotropic underetching of mesa structures, whereas anisotropy of Young's modulus is of minor importance. The high symmetry of (1 1 1) substrates makes it difficult to control helix formation, but allows the formation of rings and tubes in various directions. The investigated micro- and nano-structures on Si(1 1 0) and (1 1 1) substrates add a high degree in flexibility for the design of three-dimensional nano-structures using the roll-up technology, which makes this technology particularly suitable for integration of micromechanical components.

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