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Fabrication and characterization of freestanding Si/Cr micro- and nanospirals

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Abstract

We report on the scrolling of two-layer Si/Cr hybrid films on Si(001) wafers to form rolled-up micro-and nanostructures. Mesa stripes with width ranging from 3.0 μm to 60 nm have been used to fabricate the spirals. Decreasing the width gradually changes the scrolling direction of the Si/Cr stripe from the ⟨100⟩ direction to the longitudinal axis of the stripe. Moreover, the diameter of the Si/Cr rings decreases significantly with decreasing stripe width, which can be explained by edge effects at the side walls of the bilayer film. Based on this effect, freestanding Si/Cr spiral nanobelts can be fabricated. The flexibility of such spirals has been probed with a nano-manipulator showing their excellent shape-memory properties.

Keywords: Spiral nanobelts; Coiling up; Nano- manipulation; Shape-memory

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1. Introduction

After the discovery by Prinz et al. [1,2] that strained InGaAs/GaAs and SiGe/Si hetero-films can roll-up to form nanotubes and helical nanocoils, new strategies to scale down 3D structures in the micro- and nanometre range have been envisioned by using various materials, including metals, semiconductors and dielectrics. Two types of structure have been fabricated, i.e. tubes and helixes [2–5]. The diameter of these freestanding structures is related to the Young's modulus of the layers, the layers' thickness and the mismatch between the layers [2–5].

Recently, a simple way to fabricate micro- and nanotubes has been reported based on strained semiconductor–metal (InGaAs/metal) double layers [6]. In this paper, we present results obtained for the Si/Cr double layer system. In particular the changes of the scrolling direction and of diameter of the rolled-up nanostructures have been investigated in dependence on the stripe widths. Insights into these dependencies allow for new strategies to fabricate 3D nanoscale devices in Si, which may include nanoelectromechanical systems (NEMS). Thus, their mechanical properties, i.e. elasticity have been investigated by using a nano-manipulator.

2. Experiments

A 35 nm thick p-type Si layer was epitaxially grown by ultrahigh vacuum chemical vapour deposition (UHV-CVD) on Si(001) at 550 °C followed by the deposition of a 10 nm thick amorphous Cr layer by e-gun evaporation. E-beam lithography, reactive ion etching (RIE) and wet etching were applied to form the rolled-up silicon–metal (Si/Cr) hybrid structures on Si(001). Details of the Si/Cr stripe pattern fabrication and wet etching have been described in [5]. Here, mesa stripes of 40 μm length and 3.0 to 0.2 μm width were fabricated. The stripe patterns have been aligned to the ⟨110⟩ direction and with several degrees deviation from the ⟨110⟩ direction. To probe the

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scrolling direction dependence of the mesa stripe orientation on the Si(001) substrate over a larger range, a wagon wheel pattern [7] was examined. The maximum width of all stripes in the wagon wheel pattern is 1.4 μm in the outer most region and 60 nm close to the centre of the wheel.

To synthesize spiral nanobelts, tapered stripes were designed with one narrow and one broad end. The 3D structures were formed by lifting the Si/Cr bilayer from the Si substrate in a 3.7% ammonia water solution. Subsequently, all samples were dried in a supercritical point dryer to eliminate the impact of capillary forces during drying [8]. The structures were inspected by a field-emission scanning electron microscopy (Zeiss SUPRA 55VP). The elasticity of spiral nanobelts was studied by using a commercial nano-manipulator (Kleindiek, MM3A) mounted in an SEM (Zeiss DSM962). For this elasticity experiment the manipulator probe was inserted sidewise into the center of a spiral nanobelt. Then the probe is translated along the longitudinal axis of the stripe to extend the coiled nanobelt until the probe passed the front end of the spiral.

3. Results and discussion

The SEM image in Fig. 1 shows that the scrolling direction of a Si/Cr bilayer stripe gradually shifts from ⟨100⟩ to the ⟨110⟩ direction, i.e. to the longitudinal axis of the stripe, when the stripe width is reduced from 3.0 to 0.5 μm. In this case all stripes with width less than 1.5 μm rolled-up in the ⟨110⟩ direction. Fig. 2 shows an overview SEM image of the rolled-up Si/Cr stripes fabricated from a wagon wheel pattern. Without exception all stripes with a width w <1.4 μm are coiled up in the direction along their longitudinal axis. These results indicate that the scrolling direction of a Si/Cr bilayer depends on the width of the mesa stripe. It has been reported [3,4] that in a stack of SiGe/Si/Cr films, the e-beam evaporated Cr layer has tensile stress and applies a compressive stress on the Si layer. The tensile stress in the Cr layer leads to the scrolling of the Si/Cr bilayer after it has been detached from the Si substrate. Thus after the rolled-up structure has formed, the Si layer will harbour compressive stress and the tensile stress in the Cr film is reduced. The Young's modulus of the amorphous Cr film can be considered as isotropic, thus the preferred scrolling direction of the top Cr layer is the longitudinal axis of the stripe. However, the underneath Si layer would prefer to scroll along ⟨100⟩ because of the smaller Young's modulus. Moreover, as can be seen by the progress in the underetching of the stripes in the wagon wheel geometry (Fig. 2), the ⟨100⟩ direction is a preferred etching direction. However, for wide stripes, the Si layer determines the scrolling direction apparently, whereas for narrow ones the Cr layer dominates. This is surprising because the ratio of Si to Cr material is independent from the stripe width and should depend only on the thickness of those layers. Thus attention has to be paid to the edges along the mesa stripe. Here, two effects may occur, namely relaxation of the strained films perpendicular to the direction of the stripe and a change of the vertical profile of the mesa walls during the lateral underetching of the bilayer in the ammonia solution. Careful SEM observations reveal that the p-type Si layer is underetched with respect to the Cr layer by typically 10–20 nm (see Fig. 3, inset). It is suspected that this narrowing occurs because the p-type Si film is attacked during the RIE and wet etching processes, whereas the Cr layer maintains the same width. Moreover, it has been reported that the tensile stress at a polysilicon–oxide interface can enhance spontaneous

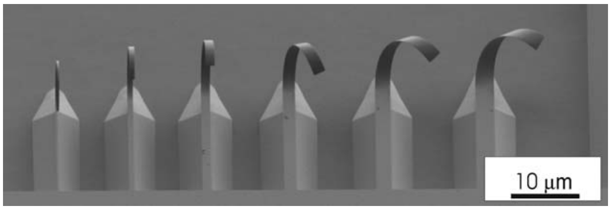

Fig. 1. SEM image of Si/Cr (35/10 nm) 3D structures. The width of the six stripes from left to right is 0.5 μm, 1.0 μm, 1.5 μm, 2.0 μm, 2.5 μm and 3.0 μm. The stripes are aligned along the ⟨110⟩.

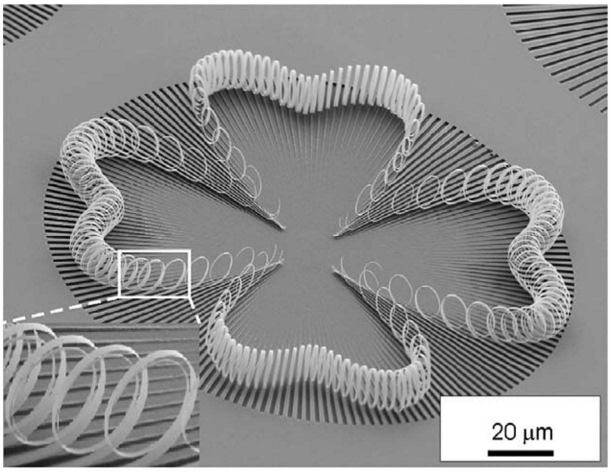

Fig. 2. SEM image of an array of Si/Cr spirals formed from a wagon wheel patterned Si/Cr bilayer. The inset shows a local magnified SEM image.

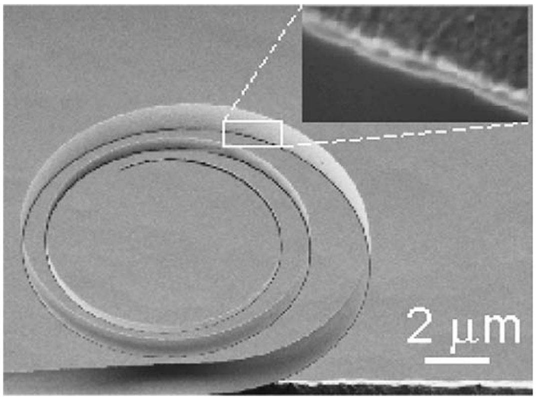

Fig. 3. SEM images of a freestanding Si/Cr spiral nanobelts fabricated from a tapered mesa line. Inset visualizes the 15 nm wide Cr overhang at the rim of the Si/Cr bilayer.

etching of a poly-Si layer [9]. The tensile stress at a Si/Cr interface may induce a similar effect in a Si layer. However, the observation indicates that the ratio of Si to Cr material does depend on the width of the stripe and changes in the favour of the Cr material. Additional relaxation at the edges will occur. We make these two effects responsible for the change over from the Si to the Cr dominated scrolling of the stripes with decreasing stripe width.

The second effect clearly visible in Fig. 1 is that the radius of the rolled-up structure decreases with the stripe width. This becomes even more evident in Fig. 3 showing a rolled-up tapered mesa line. Here the turning radius gradually increases with the width of the mesa stripe. To analyse this effect a series of mesa stripes were fabricated with width decreasing from 1.2 to 0.2 μm. This range of widths warrants that the scrolling direction is controlled by the Cr film and rings are formed independently from the orientation of the mesa stripe. The dependence of the diameter of the rings on the width of the strips is shown in Fig. 4. When the stripe width decreases from 1.2 to 0.2 μm, the diameter of rings reduces by about 30% from 14.1 to 9.8 μm.

The force F leading to the roll-up can be expressed by [10]

$$F = \varepsilon b \frac{d_{\mathrm{Cr}} d_{\mathrm{Si}} E_{\mathrm{Cr}} E_{\mathrm{Si}}}{d_{\mathrm{Cr}} E_{\mathrm{Cr}} + d_{Si} E_{Si}} \tag{1}$$

in which ε is the misfit strain, E is the elastic modulus, d gives the layer thickness, and b is the width of the stripe along the scrolling direction. According to Eq. (1), the roll-up force F for a narrow stripe decreases proportionally with reduced stripe width b. The pair of equal and opposite forces on the Si and Cr layers generate a bending moment M. M is given by [10]

$$M = F\left(\frac{d_{\mathrm{Cr}} + d_{\mathrm{Si}}}{2}\right) \tag{2}$$

The curvature of the stripe κ can be described as [11]

$$\kappa = \frac{M}{EI} = \frac{F(d_{\mathrm{Cr}} + d_{\mathrm{Si}})}{2(E_{\mathrm{Cr}} I_{\mathrm{Cr}} + E_{\mathrm{Si}} I_{\mathrm{Si}})} \tag{3}$$

In Eq. (3), EI is the stiffness of the stripe for bending which is proportional to the bilayer stripe width b [10,11]. In the ideal case, the Si and Cr layers should have the same width, so the curvature κ is independent of b. However, since the width of the Si layer is reduced compared to the Cr layer due to the lateral underetching of the Si on the edges, the actual EI is smaller than the ideal value, thus the counter moment for bending decreases, and the curvature κ becomes larger.

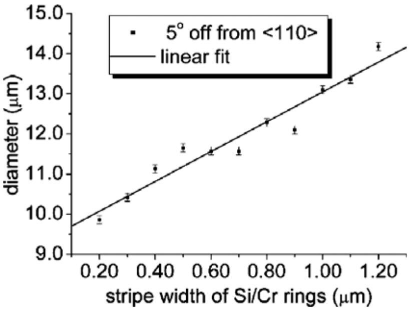

Fig. 4. Dependence of ring diameters on Si/Cr mesa stripe widths. The mesa stripes were oriented 5° off from the ⟨110⟩ direction.

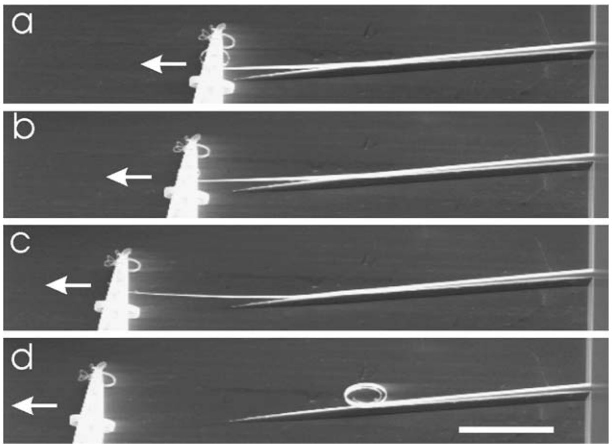

Fig. 5. (a–c) The nano-manipulator probe moves to left (white arrow) to extend the Si/Cr spiral nanobelt. (d) The spiral returns to its original shape. The scale bar is 25 μm for all images.

Using a manipulator (Kleindiek MM3A) installed in an SEM, the as-fabricated Si/Cr spiral nanobelts are manipulated for characterization. A commercially available tungsten sharp probe (Picoprobe T-1-10-1 mm) mounted on the manipulator was inserted sidewise into the spiral. To investigate the flexibility of the spiral belts, the probe is used to extend, i.e. unroll them as shown in Fig. 5, where a spiral is completely extended along its longitudinal axis, and subsequently recovers to its original shape after being released from the extension (Fig. 5d). No significant change in its curvature due to the extension was observed, which means that these freestanding spirals have a strong "memory" of their shape after being formed.

4. Conclusions

The scrolling behavior of Si/Cr stripes has been investigated. It was found that the scrolling direction and the curvature of 3D structures varied with the stripe width. Based on this edge effect, freestanding Si/Cr spiral nanobelts can be fabricated. These spiral nanobelts are very elastic and have a strong "memory" of their original shape.

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References

[1] V.Ya. Prinz, V.A. Seleznev, A.K. Gutakovsky, Inst. Phys. Conf. Ser. 166 (2000) 199.

[2] S.V. Golod, V.Ya. Prinz, V.I. Mashanov, A.K. Gutakovsky, Semicond. Sci. Technol. 16 (2001) 181.

[3] S.V. Golod, D. Gruetzmacher, C. David, E. Deckardt, O. Kirfel, S. Mentese, B. Ketterer, Microelectron. Eng. 67–68 (2003) 595.

[4] S.V. Golod, V.Ya. Prinz, P. Waegli, L. Zhang, O. Kirfel, E. Deckardt, F. Glaus, C. David, D. Gruetzmacher, Appl. Phys. Lett. 84 (2004) 3391.

[5] L. Zhang, E. Deckardt, A. Weber, C. Schoenenberger, D. Gruetzmacher, Nanotechnology 16 (2005) 655.

[6] O. Schumacher, S. Mendach, H. Welsch, A. Schramm, Ch. Heyn, W. Hansen, Appl. Phys. Lett. 86 (2005) 143109.

[7] H. Seidel, L. Csepregi, A. Heuberger, H. Baumgärtel, J. Electrochem. Soc. 137 (1990) 3612.

[8] V. Seleznev, H. Yamaguchi, Y. Hirayama, V.Ya. Prinz, Jpn. J. Appl. Phys. Pt. 2 (42) (2003) 791.

[9] J.P. Chang, H.H. Sawin, J. Vac. Sci. Technol. B 19 (5) (2001) 1870.

[10] Y.C. Tsui, T.W. Clyne, Thin Solid Films 306 (1997) 23.

[11] J.M. Gere, Mechanics of Materials, sixth ed., Brooks/Cole-Thomson learning, Belmont, USA, 2004.