Freestanding SiGe/Si/Cr and SiGe/Si/Si_xN_y/Cr microtubes

S. V. Golod and V. Ya. Prinz

Institute of Semiconductor Physics, Siberian Branch of Russian Academy of Sciences, 13, Lavrent'ev Avenue, Novosibirsk 630090, Russia

P. Wägli

Laboratorium für Festkörperphysik, Federal Institute of Technology Zürich, CH-8093 Zürich, Switzerland

L. Zhang, O. Kirfel, E. Deckhardt, F. Glaus, C. David, and D. Grützmacher^{a)}

Laboratory for Micro- and Nanotechnology, Paul Scherrer Institute, CH-5232 Villigen-PSI, Switzerland

(Received 13 August 2003; accepted 3 March 2004)

We report on hybrid microtubes and rings fabricated from rolled-up strained metal–semiconductor SiGe/Si/Cr and metal–insulator–semiconductor SiGe/Si/Si_xN_y/Cr films. For making suspended microtubes, a method of directional rolling of the patterned films by anisotropic underetching of silicon substrate was introduced. It is shown quantitatively that Cr and Si_xN_y layers are highly strained, the tensile stress being sufficient to cause the rolling-up of the hybrid films into microtubes of preset diameter. The proposed controllable and reproducible technology is promising for fabricating cylindrical-shaped microcapacitors, induction coils, transistors, and building blocks of microelectromechanical devices. © 2004 American Institute of Physics.

[DOI: 10.1063/1.1736317]

Recently, a self-rolling procedure for forming three-dimensional micro- and nano-objects from planar semiconductor films has been introduced.^{1–3} For practical applications, micro- and nanotubes are required, fabricated not only from semiconductor heterostructures, but also from more complex multilayered hybrid structures composed of metal, dielectric, and/or magnetic-material layers. This would substantially widen the possible applications of such tubes and related objects in micromechanics and in micro- and nano-electronics. Device elements that can be prepared in this way are cylindrical-shaped capacitors, inductors, and building blocks for tube-like field-effect transistors composed from freestanding and/or rolled-up hybrid films. Some of these applications would require freestanding tubes; that is, tubes detached from substrates over a considerable part of their length or protruding over substrate edges. The ability to manufacture truly freestanding tubes would also enable the in-depth study of the electrical and mechanical properties. The present work is intended to show, by the example of strained SiGe/Si/Cr and SiGe/Si/Si_xN_y/Cr structures, the possibility of fabricating metal–semiconductor (MS) and metal–insulator–semiconductor (MIS) hybrid freestanding microtubes using the rolling procedure. Methods to control the diameters of hybrid tubes and their fabrication processes are described.

In the present study, the SiGe/Si bilayers were grown on a (001) *n*-type Si wafer at 400 °C by molecular-beam epitaxy (MBE) or by ultrahigh-vacuum chemical vapor deposition (UHV/CVD) at 550 °C. The thickness of the pseudomorphic SiGe layer was always below the critical thickness; thus, relaxation via formation of misfit dislocations can be excluded, even after annealing steps. The fabrication process of the SiGe/Si/Cr tubes started with the growth of strained

pseudomorphic SiGe/Si heterolayers heavily doped with boron (10^{20} cm^{-3}). Subsequently, a Cr top layer was deposited by thermal evaporation. The high-purity Cr was deposited at a rate of 5 nm/min using a working pressure of $5 \times 10^{-5} \text{ Pa}$. Chromium was chosen for the upper metal layer since it provides a well-established technology for electron-beam (e-beam) lithography, it is stable against alkaline etchants, and it is highly tensile strained when deposited onto silicon.⁴ E-beam lithography was used to pattern the initial planar structure. After developing the resist, the defined pattern was transferred onto the underlying SiGe/Si/Cr film using $\text{Cl}_2 + \text{CO}_2$ and $\text{CHF}_3 + \text{O}_2$ reactive ion etching. The initial mesa pattern for freestanding SiGe/Si/Cr tubes is schematically shown in Fig. 1(c) as a white region against a gray background (gray area is etched). Next, the low-doped substrate underlying the patterned p^+ -SiGe/Si/Cr structure was selectively etched in aqueous solutions of NH_4OH (Refs. 2 and 3) or KOH .⁵ In the initial structure, the Cr film deposited onto Si is under tension, and the SiGe layer is under compression. As a result, the detached SiGe/Si/Cr film undergoes bending due to the internal elastic stress and rolls into a tube. This process is schematically shown in Fig. 1(a).

To control precisely the fabrication of a freestanding tube, it is not only necessary to detach the stack of films from the substrate, but also to determine the direction under which the initial planar film, patterned as a mesa strip, starts to roll into a tube. In the present work the directional rolling of a narrow strip of the strained film was achieved by anisotropic etching of the silicon substrate. This results in the formation of (111) facets at the sides of the mesa oriented along the (110) directions; these facets prevent lateral underetching and rolling of the SiGe/Si/Cr strip in the direction indicated by the open white arrow. Mesa edges oriented along the (100) direction are underetched most rapidly and as a result, the strained film starts rolling in the direction indicated by the thick black arrow in Fig. 1(c). Figure 1(b) shows a scan-

^{a)}Electronic mail: detlev.gruetzmacher@psi.ch

FIG. 1. (a) Schematic view of the scrolling process of a semiconductor-metal hybrid tube. The arrows F_1 and F_2 symbolize the forces induced by the stretched Cr and compressed SiGe films, respectively, and which give rise to the momentum M scrolling the stack of films. (b) SEM image of a freestanding $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}/\text{Cr}$ tube of 210 μm length and 3.5 μm diameter. The initial stack consisted of an MBE $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}$ (20/20 nm) bilayer and a 20-nm-thick top Cr layer. (c) The initial mesa pattern used to fabricate the freestanding tube. The thick black arrow indicates the rolling direction of the white figure; the rolling process is suppressed in the direction indicated by the open white arrow.

ning electron microscopy (SEM) image of 210- μm -long suspended hybrid tube formed by $1\frac{1}{2}$ turns of the initially planar $\text{SiGe}/\text{Si}/\text{Cr}$ (20/20/20 nm) film. The diameter of this tube is 3.5 μm . It should be noted that the predominant portion of the tube (about 80% of its total length) is completely detached from the substrate forming a truly free-standing tube. In Fig. 1, it is clearly seen that the tube is slightly bent and the initially free-standing end is attached to the substrate. This bending occurred during the SEM study and is due to the accumulated static electrical charge. From this experiment it can therefore be inferred that flexible freestanding hybrid tubes are able to be substantially bent by external electric field or under mechanical actions.

The possibilities provided by the self-rolling method are by no means restricted to the case of films composed by semiconductor and metal layers. Figure 2 shows a SEM im-

FIG. 2. SEM micrograph of two suspended $\text{SiGe}/\text{Si}/\text{Si}_3\text{N}_4/\text{Cr}$ tubes (scrolls). The diameter of the inner tube of the $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}_3\text{N}_4/\text{Cr}$ (10/10/10/18 nm) scrolls is 3.8 μm .

FIG. 3. SEM images of rolled objects. (a) A bent $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}/\text{Cr}$ cantilever of 0.3 μm width, 5 μm length, and 1.15 μm curvature radius. (b) A ring 1.6 μm in diameter formed after the Cr layer was selectively removed from the initial planar stack. The thicknesses of the UHV/CVD grown $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}$ and evaporated Cr layers were 10 nm/10 nm and 18 nm, respectively.

age of two hybrid MIS tubes prepared from the four-layered stack containing a Si_xN_y dielectric film sandwiched between the Si and the Cr layer. In detail, the stack was composed of a UHV/CVD grown $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}$ (10/10 nm) bilayer, a 10-nm-thick Si_xN_y film deposited by low-pressure CVD at 800 $^\circ\text{C}$ from a dichlorosilane/ammonia gas mixture, and a 18-nm-thick top Cr layer. The growth rate of Si_xN_y was 3.5 nm/min and the refractive index of the Si_xN_y film was very close to 2. Olson⁶ showed that Si_xN_y films obtained under similar conditions exhibit a considerable residual tensile stress. This stress is generated as Si_xN_y undergoes shrinkage during high-temperature treatments and subsequent continuous post-growth cooling.⁷ The tubes depicted in Fig. 2 contain two turns. The inner diameter of the tubes fabricated from this stack is about 3.8 μm . The same layer sequence despite the silicon nitride layer leads to tubes having an inner diameter of 2.3 μm [Fig. 3(a)]. Thus, it can be concluded that the nitride film supports the scrolling process rather than hampering it. For technological processing, it is important that after a 2-hour-long high-temperature anneal (650–800 $^\circ\text{C}$), the grown SiGe heterostructures are still highly strained, yielding tubes of the same diameter as those fabricated from as-grown structures. However, one of the most important advantages of the produced MS and MIS tubes (Figs. 1 and 2, respectively) is that they are freestanding; that is, a predominant proportion of their length has a significant separation from the substrate surface. Thus, parasitic impacts of the substrate will be reduced in analogy to planar spiral inductors and capacitors,⁸ for which a detachment from the substrate leads to an increase of the quality factor.

In order to roll up hybrid films containing semiconductor, dielectric, and metal layers into a tube of a preset diameter, it is essential to be able to control the stresses in individual layers. Previous studies^{1,2,9} showed that the diameter of tubes formed from two-layer MBE-grown semiconductor films obeys the elasticity-theory relations even for tubes down to a few nanometers in diameter; thus, to predict the tube diameters, the formula proposed by Tsui¹⁰ can be used. A simplified formula for the diameter D of tubes prepared from three-layer films is given subsequently. This formula was derived by assuming the elastic moduli of all three layers to be identical and by using the approaches described by Tsui¹⁰ and Timoshenko.¹¹ In the case of thicknesses and Young's moduli of tube layers differing within a factor of 2, the tube diameters obtained by exact formulas are 3%–8% greater than the approximate diameter, given by

$$D = \frac{1}{3} \frac{(d_1 + d_2 + d_3)^3}{f_1 d_1 (d_2 + d_3) + f_2 (d_1 + d_2) d_3}, \quad (1)$$

where d_1 , d_2 , and d_3 are the thicknesses of the SiGe, Si, and Cr layers, respectively; f_1 is the strain due to lattice mismatch between Si and SiGe; and f_2 accounts for the strain of the Cr layer on Si. With $d_3=0$, formula (1) reduces to the well-known formula for a two-layer film.¹⁰

Whereas the mechanical deformations of semiconductor structures can easily be predicted based on lattice-mismatch considerations, there is no easy prediction of deformations in bent structures containing additional metal or dielectric layers, since the total stress σ in such structures depends not only on the thermal-expansion mismatch between the film and the substrate, but also on the intrinsic stress.⁴ In turn, the intrinsic stress depends on the conditions under which those layers were deposited and on the properties of the semiconductor surface.⁴ Considering the SiGe/Si/Cr tubes fabricated in the present study, the strain in the Cr layer was calculated by formula (1) from the diameter (3.5 μm) of the freestanding SiGe/Si/Cr microtube in Fig. 1. The obtained tensile strain f_2 amounts to 0.95%. Assuming that the stress of the Cr layer is given by $\sigma = f_2 E_{\text{Cr}}$ (for the generalized biaxial Young's modulus of Cr $E_{\text{Cr}} = 377 \text{ GPa}$ ¹²), one readily obtains the tensile stress $\sigma = 3.6 \text{ GPa}$.

Figure 3 shows SEM micrographs of ring-like structures obtained from $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}/\text{Cr}$ 10/10/18 nm stacks before (a) and after (b) selective removal of the Cr layer in $\text{Cl}_2 + \text{CO}_2$ plasma. Before the removal of the Cr layer, the predefined mesa strip was not sufficiently long enough to roll it into a closed ring, and only $\frac{3}{4}$ of a single turn was obtained. The measured diameter is 2.3 μm [see Fig. 3(a)]; after the selective etching of the Cr film a ring with a diameter of 1.6 μm is obtained [see Fig. 3(b)]. The experimental values are in good agreement with those predicted by formula (1) assuming a tensile stress of the Cr film of 3.6 GPa and by the formula of Tsui,¹⁰ which yield diameters of 2.3 and 1.64 μm , respectively. Our results [Figs. 1 and 3(a)] have shown conclusively that the radius of curvature of the scrolled MS films are controlled and can be reproduced from one structure to another. On the one hand, it should be stressed that it is possible to choose the thicknesses and stresses in the layers such that the diameters of bilayered and multilayered hybrid tubes are almost identical. Using formula (1) and the experimental value of the tensile stress in the Cr layer, it is deduced that the diameters of the $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}$ semiconductor and the $\text{Si}_{0.6}\text{Ge}_{0.4}/\text{Si}/\text{Cr}$ hybrid tubes are identical if 10-nm-wide semiconductor films and an 8-nm-wide Cr film are used. Such a choice of thicknesses prevents substantial deformation of complex tubes composed of sections along the tube having different number of layers, which might occur in tubes used as building blocks for micromechanical cantilevers or transistors having source, gate, and drain regions located along the tube length. On the other hand, it is possible, using lithography and selective etching of the metal layers, to control the diameter of hybrid tubes and obtain tubes with locally modulated thicknesses.

In conclusion, we have introduced a controllable and reproducible procedure for fabricating SiGe/Si/Cr and SiGe/Si/Si_xN_y/Cr freestanding hybrid microtubes. The contribution of the highly strained Cr, Si_xN_y, and SiGe/Si layers to the rolling process of the hybrid films is quantitatively determined. For making suspended microtubes a method of directional rolling of the patterned films caused by anisotropic underetching of silicon substrate was proposed and realized. It is shown that the diameter of hybrid tubes can be precisely controlled through variation of layer thicknesses and strain in initial structures, and also using selective etching of individual layers. The hybrid rolled objects offer a path to create compact induction coils, fixed and variable cylindrical capacitors, and tube-like field-effect transistors with the potential of easy integration of these elements into Si-based microelectronics. Moreover, the possibilities offered by the rolling method are by no means restricted to the use of only Cr and Si_xN_y; they can be extended to other materials displaying substantial etching selectivity and tensile stress. Magnetic memory chips, quantum magnetic rings, and hard disk read heads¹³ can be developed from the freestanding flexible hybrid tubes, rings, and cantilevers containing thin Cr, Co, Ni, Mn, or Fe magnetic layers. In addition, freestanding tubes provide fresh possibilities for the investigation of the electrical, optical, and mechanical properties of rolled-up structures. Further reduction of tube diameters by using thinner films and increasing the Ge content of the bottom layer for raising the general strain level in the structure seem to be promising lines for future studies.

This work was supported by the Swiss National Science Foundation, by SCOPES contract no. 71P065794, and by the Russian Foundation for Basic Research (grant 03-02-17600).

¹V. Ya. Prinz, V. A. Seleznev, A. K. Gutakovsky, A. V. Chehovskiy, V. V. Preobrazhenskii, M. A. Putyato, and T. A. Gavrilo, *Physica E (Amsterdam)* **6**, 828 (2000).

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

³V. Ya. Prinz, D. Grützacher, A. Beyer, C. David, B. Ketterer, and E. Deckardt, *Nanotechnology* **12**, 399 (2001).

⁴J. A. Thornton and D. W. Hoffman, *Thin Solid Films* **171**, 5 (1989).

⁵H. Seidel, L. Csepregi, A. Heuberger, and H. Baumgärtel, *J. Electrochem. Soc.* **137**, 3626 (1990).

⁶J. M. Olson, *Mater. Sci. Semicond. Process.* **5**, 51 (2002).

⁷E. B. Gorokhov, V. Ya. Prinz, A. G. Noskov, and T. A. Gavrilo, *J. Electrochem. Soc.* **145**, 2120 (1998).

⁸J. Chen, J. Zou, C. Liu, J. E. Schutt-Ainé, and S.-M. Kang, *IEEE Trans. Electron Devices* **50**, 730 (2003).

⁹Ch. Deneke, C. Müller, N. Y. Jin-Phillipp, and O. G. Schmidt, *Semicond. Sci. Technol.* **17**, 1278 (2002).

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

¹¹S. P. Timoshenko and J. M. Gere, *Mechanics of Materials* (Van Nostrand Reinhold, New York, 1972).

¹²F. Elstner, C. Gautier, O. Piot, G. Contoux, F. Cosset, F. Nardou, and J. Machet, *Phys. Status Solidi A* **154**, 669 (1996).

¹³S. A. Wolf, D. D. Awschalom, R. A. Buhrman, J. M. Daughton, S. von Molnár, M. L. Roukes, A. Y. Chtchelkanova, and D. M. Treger, *Science* **294**, 1488 (2001).