Microelectronic Engineering 153 (2016) 88–91

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Top–down SiGe nanostructures on Ge membranes realized by e-beam lithography and wet etching

V. Mondiali [a,b,*], M. Lodari [a,b], M. Borriello [a], D. Chrastina [a], M. Bollani [b]

[a] L-NESS, Dipartimento di Fisica, Politecnico di Milano, Polo di Como, via Anzani 42, I-22100 Como, Italy
[b] IFN-CNR, L-NESS, via Anzani 42, I-22100 Como, Italy

ARTICLE INFO

Article history:
Received 27 October 2015
Received in revised form 31 January 2016
Accepted 10 February 2016
Available online 13 February 2016

Keywords:
Ge membrane
Nanoscale etching
Wet-anisotropic etching
Electron beam lithography

ABSTRACT

SiGe nanostructures on Ge membranes have been fabricated by electron beam lithography and anisotropic wet-chemical etching, starting from SiGe/Ge heterostructures epitaxially deposited on Si substrates. Two different top–down approaches have been studied in order to obtain the best freestanding structures. We find that the process in which the Ge membrane is suspended after the lithography of the SiGe nanostructures leads to high quality SiGe nanostructures without damage to either the SiGe nanostructures or the Ge membrane. The structures have been systematically analyzed at every step of the fabrication process, by scanning electron microscopy and by atomic force microscopy.

1. Introduction

Novel approaches are needed to improve the performance of opto-electronic devices in the Si-based information and communication technologies [1,2]. Strain engineering is one of the approaches used to increase the performance of the device through the ability to control and to engineer the band structure of the semiconductor [3,4]. Moreover, the modulation of the strain allows the control of the electron/hole mobility [5] and thereby the conductivity of the system, with great benefits for the performance of the devices [6]. Germanium, for example, is fully compatible with Si-technology. So, transforming it in a direct-gap semiconductor would open up new interesting scenarios in the field of the mid-infrared signal detection and emission [7], in the realization of on-chip CMOS systems and of integrated waveguides [8–10]. We have already demonstrated that the edge forces exerted by epitaxial SiGe nanostructures induce high levels of strain in the substrate and that the resulting strain can be exploited to engineer the functional properties of the substrate [11,12]. In addition, such high strain values are theoretically predicted to transform germanium from an indirect to a direct gap semiconductor [13]. However, finite-element simulations suggest that the use of SiGe nanostructures realized on Ge suspended membranes instead of bulk substrates should allow a great strain-level improvement which leads to larger areas where the Ge has a direct conduction band [14,15]. Moreover, recent analyses also show how the improved quality and the lower thickness of the membranes influence the optical and electronic properties of semiconductors [16].

Free-standing membranes are usually fabricated by wet anisotropic etching because of the high etch rate and the low costs. SiGe and Ge thin films grown on ultrathin Si-on-insulator (SOI) substrates can be suspended removing the oxide layer through the use of hydrofluoric acid (HF) solutions [17–19]. Also alkaline solutions, such as potassium hydroxide (KOH) and tetramethylammonium hydroxide (TMAH), are widely used to suspend nanostructures by etching the Si substrate [20–22], due to their high material selectivity and crystallographic anisotropy [23]. So, a combination of lithographic process and dry and wet anisotropic etchings (with KOH solution) has been used in a previous work for the fabrication of Ge membranes without damaging the Si material present in the SiGe alloys [24]. Therefore, in this work the SiGe nanostructures have been fabricated on top of the suspended Ge thin film. The aim is to obtain the best profile quality of the nanostructures, thus two different approaches have been investigated. In the first case the suspension of the structure was performed before the realization of the SiGe nanostructures by electron beam lithography (EBL) and reactive ion etching (RIE); in the second approach the last step is the suspension of the structure via wet-chemical etching. The obtained structures have been systematically analyzed step by step by scanning electron microscopy (SEM) and the morphology characterized by atomic force microscopy (AFM). The optimized SiGe nanostructures can be used to create deformations in the underlying layer: in this work this layer is a Ge membrane, but this approach can be used in principle for any other semiconductor alloys taking into account the changes in etching solutions.

* Corresponding author at: via Anzani 42, I-22100 Como, Italy.
E-mail address: valeria.mondiali@polimi.it (V. Mondiali).

http://dx.doi.org/10.1016/j.mee.2016.02.015
0167-9317/

2. Material and methods

2.1. Material details

A layer of Ge (100 nm thick) has been grown on a HF-dipped Si(001) wafer by low-energy plasma-enhanced chemical vapor deposition (LEPECVD) at 500 °C and with a growth rate of 0.4 nm/s. Since the Ge film thickness is much greater than the critical thickness [25], the Ge film is completely relaxed. Then, a $Si_{1-x}Ge_x$ film ($x = 0.5$) has been epitaxially deposited on the Ge layer at 400 °C (Fig. 1(a)). The nominal thickness of the SiGe film is 50 nm, so the SiGe film results to be under tensile strain [26] and it can be used, after a proper patterning, as stressor for the underlying Ge film. X-ray diffraction measurements confirm that before patterning, the Ge layer is fully relaxed and the SiGe film is lattice-matched to the Ge layer.

2.2. Fabrication process: results and discussion

First, EBL (acceleration voltage of 30 kV) and RIE have been used to define mesa structures along the <100> directions to allow the underetching of the Si substrate (Fig. 1(b)). For these kind of structures a negative resist has been employed (AR-N 7520.07) and the dose used for the membrane was 200 μC/cm^2. As developer a solution of TMAH (tetramethylammonium hydroxide) and deionized water in the 4:1 ratio is used for 90 s. Then, the pattern was transferred to the Ge epitaxial film by RIE, using a CF_4 plasma, 80 W RF power and a total gas pressure of 5.4 mTorr. Finally the resist is removed using acetone. The realized mesas were characterized by SEM. Fig. 2(a) shows different SiGe/Ge mesas in the same structure, the black square marks the area shown in Fig. 2(b): the resulting thickness of the SiGe/Ge stack is about 160 nm. At this point several fabrication tests have been carried out in order to optimize the patterned SiGe/Ge membranes. Two different approaches have been used. In the first approach, the mesas have been suspended before the realization of the SiGe nanostructures (Fig. 1(c)). In this case, the samples have been cleaned by dilute HF to remove the native silicon oxide layer to make it more rapid and effective. Afterwards the samples have been etched with 40 wt.% KOH at 70 °C in order to suspend the SiGe/Ge mesas: in fact KOH etches the Si substrate allowing a high selectivity between etching rates in <100> and <111> directions, while Ge is chemically resistant. The resulting structure is shown in Fig. 2(c): the SiGe/Ge membranes are completely suspended from the Si(001) substrate. Finally, to realize the nanostructures, the SiGe thin layer is exposed by EBL and etched by RIE. The exposures have been performed with a positive resist (PMMA E-Beam Resists AR-P 950) and with single pixel line method (dose = 930 pC/cm). In this case, a solution of methyl isobutyl ketone (MIBK-$C_6H_{12}O$) and isopropanol (IPA) in a 1:3 ratio is used for 50 s. Then, the pattern is transferred to the SiGe film through RIE, using a CF_4 plasma (13 sccm), 50 W RF power and a total gas pressure of 6.4 mTorr. Finally the resist is again removed with acetone. Since the RIE does not etch selectively the SiGe film respect to the Ge epitaxial material, several samples have been realized changing the etching time. The etched samples have been characterized by AFM and μRaman spectroscopy. Using AFM some thickness measurements were carried out, while by micro Raman the presence/absence of the Ge peak was observed determining if the pattern was correctly transferred in the SiGe layer.

A representation of the obtained structures is reported in Fig. 1(e). All samples have been analyzed by SEM in order to measure the etched thicknesses and to verify the quality of the structures. Fig. 3(a)–(b) shows different patterns of the SiGe layer on the Ge membrane: stripes and crosses, respectively. Fig. 3(b) shows that the crosses are connected to each other in the direction perpendicular to the bridge due to a not perfect PMMA deposition and, therefore, to a not uniform EBL exposure. In fact, when the SiGe/Ge mesa is suspended by wet chemical etching, the resulting membrane is bent downwards because of the force exerted by the partial release of the strain stored in the SiGe layer [27] (Fig. 2(c)). This effect can affect negatively the quality of the patterning: in fact, due to this bending, the PMMA is not spin-coated in a homogeneous way.

Another unexpected result is that the final thickness of the SiGe structures is lower than the initial thickness. We have already demonstrated that TMAH and KOH etching solutions do not damage the SiGe layer on top of the structure for a Ge content in the range of 60%–90% [24], so this effect is not due to the wet etching. At first, the pattern seemed to be overexposed in correspondence of the membrane (Fig. 3(b)) respect to the pattern on the surrounding regions which was, on the contrary, perfectly exposed. If this hypothesis was true, after the development, the resist on the membrane would be very thin, due to the over-exposition, so during the RIE process the thin residual layer of resist would be completely removed and the SiGe layer would be also damaged, resulting in a thinner SiGe/Ge stack. In literature, however, membranes with a thickness of tens of nanometers have been used as substrates for ultrahigh-resolution EBL [28–30] because of significant reduction of backscattering effects. This means that using the same dose for all the nanostructures, the pattern should be underexposed on the membrane respect to the surrounding regions,

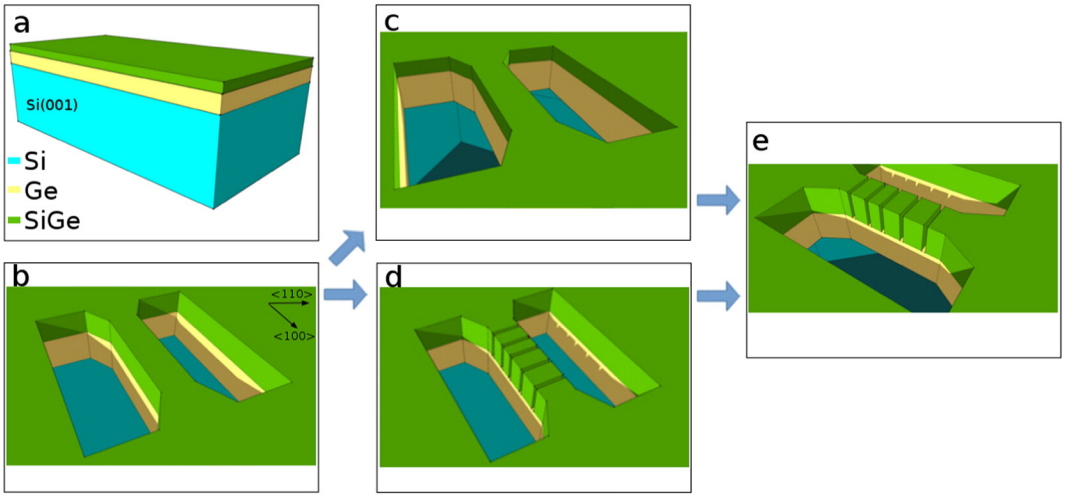

Fig. 1. (a)–(e) Schematic sequence of the fabrication steps of SiGe nanostructures on Ge membranes, the size of the nanostructures is not in scale. (a) 100 nm of Ge and the $Si_{0.5}Ge_{0.5}$ layer (50 nm thick) are grown by LEPECVD on a Si(001) substrate. (b) The SiGe/Ge mesa is realized by EBL and RIE. (c) The SiGe/Ge mesa is suspended by wet chemical etching. (d) The SiGe nanostructures are obtained by EBL and RIE on the mesa. (e) The final structure is composed of SiGe nanostructures on top of the Ge membrane.