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Three-dimensional fabrication of free-standing epitaxial semiconductor nanostructures obtained by focused ion beam

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ABSTRACT

We target the nanofabrication of free-standing nanostructures made of epitaxial semiconductor material layers of high crystallinity quality and high heterostructure complexity for optical applications at the nanoscale. Here we demonstrate the fabrication method in the case of epitaxial germanium grown on a silicon substrate but the method can be applied to any heterostructure material. The nanostructures are fabricated out of planar epitaxial wafers in the form of pillars with arbitrary section and high aspect ratio by electron-beam lithography and deep reactive-ion etching. The patterned SiGe structures are then released by focused ion-beam milling of the pillar base. In this way, they become free-standing and can be relocated on a suitable substrate by using a nanomanipulator. Microscopic characterizations are ongoing to verify that the high crystal quality typical of epitaxial layers grown on a large-area substrate is preserved throughout the different fabrication steps.

1. Introduction

Epitaxial semiconductor layers grown by chemical vapor deposition (CVD) and molecular beam epitaxy (MBE) are at the core of many microelectronic devices and of almost all optoelectronic ones. Lattice-parameter matching and strain relaxation techniques are both routinely employed to form heterostructures made of different semiconductor materials, including semiconductor–semiconductor junctions [1], strain engineering for the control of important functional properties in optoelectronics fields [2–4], quantum barriers for the confinement of electrons [5], quantum wells for the creation of discrete states with optically-active transitions [6], refractive index profiles for light guiding [7]. The required high level of control at the atomic scale is obtained by growing the epitaxial materials in ultra-high vacuum chambers with controlled temperature and reagent concentration profiles. Apparently, this control can be achieved only on large-area substrates that are previously cleaned, mechanically polished, and chemically etched to realize a flat surface at the atomic scale, where temperature and concentration gradients in the directions orthogonal to the growth axis are minimized. The resulting epitaxial material is indeed the ideal one to fabricate devices and integrated circuits by standard planar lithography.

A large variety of attempts have been made to grow epitaxial layers on nanostructured substrates, and excellent results have been achieved e.g. in the growth of quantum dots in etched pits [8–11] or, at the opposite end of the nanostructure shape spectrum, in the growth of ultra-strained epitaxial layers on substrates patterned with pillar fields [12,13]. However, if one simply wants to transfer the known properties of planar epi-layers on free-standing nanostructures (e.g. for localized light emission [14] or for mid-infrared absorption spectroscopy in the molecular fingerprint region [15]), these approaches cannot be employed, since the nanostructure properties are lost once it is released from the substrate. The aim of this work is to describe a fabrication method that allows one to produce nanostructures with well defined

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geometry out of epitaxial material layers grown in ideal conditions on large-area substrates, so as to deploy their electronic and optical functions at the nanoscale. In order to describe our fabrication method, we provide an experimental demonstration by fabricating nano-rods and nano-cones out of germanium layers hetero-epitaxially grown on silicon wafers, but the present method could be applied to many different semiconductor materials and heterostructures. In the case of germanium, for example, such a result would allow to transfer the mid-IR plasmonic properties of the epitaxial material to the free-standing nanostructures. Up to now, different designs of 3D metal nanostructures have been proposed as plasmonic tips for spectroscopic investigation at the nanoscale in the visible range [16,17] and in the mid-infrared [18]. Epitaxial semiconductor materials with high doping level could be used for the same range of applications.

2. Experimental

2.1. Epitaxial material growth

Among the possible semiconductors, we individuated epitaxial SiGe alloys with high Ge content, therefore with small effective mass and high plasma frequency, as the most promising and interesting material to study. The epitaxial material used in this first demonstration is a thick Ge layer grown on Si (001) substrate by low energy plasma enhanced chemical vapor deposition (LEPECVD). While also being an indirect gap semiconductor, Ge features a direct gap that is only 140 meV larger than the indirect gap. Due to its light emission properties at its bandgap energy of 0.8 eV, germanium is attracting considerable interest from semiconductor chip manufacturers and from telecommunication industry. Moreover, Ge epilayers can be electron- or hole-doped and they can be grown in stacks with epilayers of the Ge-rich SiGe alloy, hence enabling access to the different degrees of freedom of the Ge/SiGe heterostructure [19]. By quantum-mechanical design, the band structure of Ge and Ge/SiGe epitaxial layers can be modified to optimize the emission and detection properties at the standard telecom wavelength of 1550 nm (0.8 eV photon energy) and indeed a large number of integrated devices have been developed, proposed, and designed along these lines [20]. Clearly, spectroscopic chemical sensors based on free-standing epitaxial Ge and SiGe nanostructures, capable of enhanced photoluminescence emission at 1550 nm [21], would take advantage of the existing telecom technology for detection. Starting from HF-dipped Si (001) 4-in wafers, a 2 μm Ge layer was deposited at 500 °C at a growth rate of 4.3 nm s^{-1} and then annealed in-situ over six cycles between 600 and 800 °C in order to reduce the threading

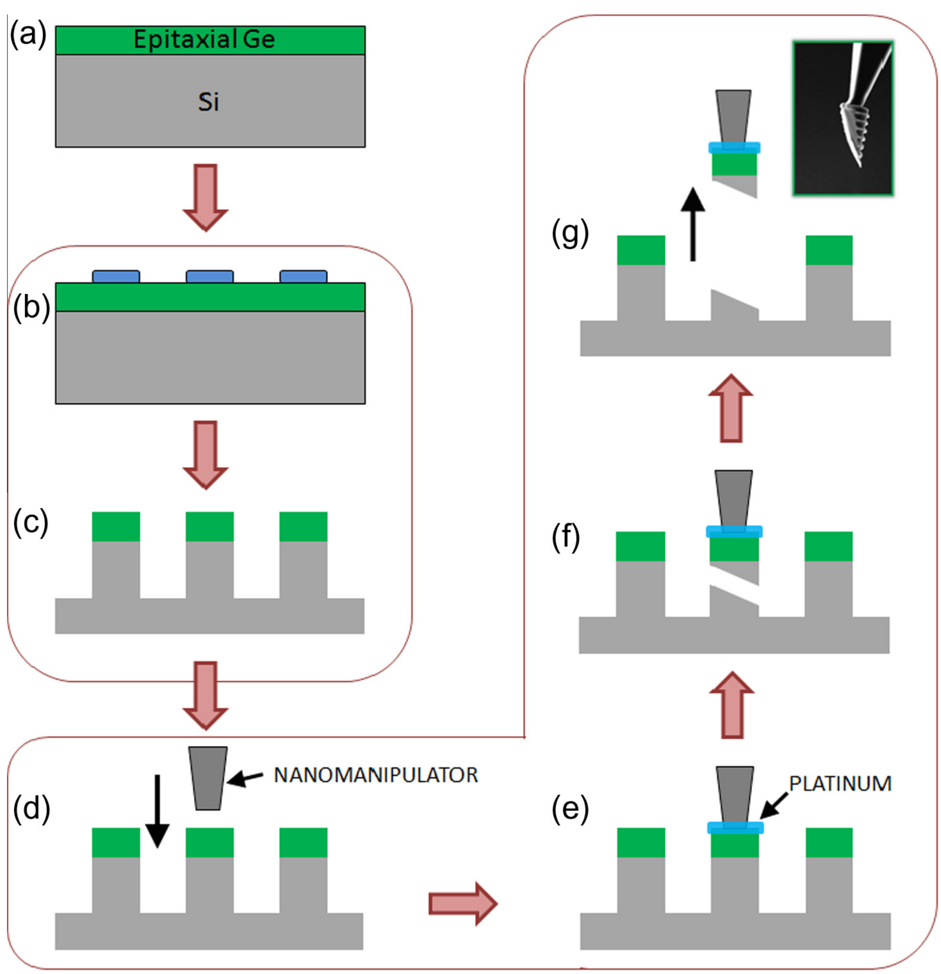

Fig. 1. Schematic sequence of the different steps for the fabrication of free-standing Ge nanostructures. (a) Starting from a continuous epitaxial Ge film grown on Si (001) substrate by LEPECVD, a particular pattern with different geometries is obtained by electron beam lithography (EBL) (b). Inductively coupled plasma (ICP) etching techniques have been employed to realize pillars with high aspect ratio on silicon substrate (c). Then, for each nanostructure, a nanomanipulator is approached on the Ge pillar surface (d) and glued on the Ge pillar top by ion-beam induced deposition (IB-ID) or by electron-beam induced deposition (EB-ID) (e). The free-standing Ge nanostructure is obtained by cutting the pillar base through FIB milling (f) and subsequent lift-out of the structure (g).