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Size dependence of hall mobility and dislocation density in Ge heteroepitaxial layers grown by MBE on a SiO_2 patterned Si template

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Abstract

We have investigated the size dependent properties of the Hall mobility and etch pit dislocations (EPDs) in germanium (Ge) heteroepitaxial layers. Pure Ge thin films were grown by molecular beam epitaxy (MBE) using pattern guided growth at 650 °C and compared with homoepitaxially grown films. The results show enhanced Hall mobility and lower dislocation density as the pattern size decreases. The number of EPDs was decreased by one order of magnitude and the Hall mobility measured with different sizes of van der Pauw patterns was enhanced by two times as the pattern size was decreased from $200 \times 200\,\mu m^2$ to $3 \times 3\,\mu m^2$. Raman spectroscopy was also employed to characterize residual strain and crystalline quality of the epitaxial films with respect to the pattern size. We conclude that nano-scale pattern guided heteroepitaxial growth of Ge may be a plausible method for monolithic integration of Ge optoelectronic or electronic devices onto a Si substrate.

Keywords: Germanium heteroepitaxy; Patterned template; Size dependence; Hall mobility; Etch pit dislocations (EPDs); Molecular beam epitaxy (MBE)

1. Introduction

Heteroepitaxial growth of pure Ge on Si substrates is a key technology for both high performance Ge-based electronic and optoelectronic device applications. Recently, Ge- field effect transistors (FETs) with higher mobility and high quantum efficiency Ge photo detectors in important spectral ranges of 1.3–1.6 μm for optical communications have been demonstrated [1,2]. High quality epitaxial Ge can also provide a good platform for III–V growth on Si substrates in order to provide a better lattice match for III–V with a larger mismatch with Si. However, it needs additional efforts to resolve some important issues such as misfit and threading dislocations because of large lattice mismatch (~4.2%) between Ge (*a*: 5.6461 Å) and Si (*a*: 5.4309 Å). The dislocations make device performance seriously degraded due to rough surface morphology and large leakage currents. It was also found that threading dislocations affect carrier transport, in particular, low temperature mobility significantly decreases when their density exceeds $3 \times 10^8\,cm^{-2}$ [3].

To date, several approaches have been reported to reduce threading dislocation densities for highly applicable quality of heteroepitaxial Ge layers. One is to grow on a relatively thick and linearly graded buffer layer with a typical grading rate of 10%/μm [4]. The second one is to use a congregational layer such as a low temperature buffer for defect gathering and annihilation [5]. However, the resulting threading dislocation density is still too high and very thick buffer layers are needed. Another approach is using a patterned substrate with a finite dimension in vertical and/ or lateral [6]. F. Huang proposed in a theoretical analysis for dislocation generation and elastic strain in lattice mismatched epitaxial layers grown on a finite dimension [7]. Zubia et al. demonstrated the mismatch strain energy can be distributed in three dimensions during epitaxial growth and it is greatly reduced by using nano-heteroepitaxy by taking into account of strain partitioning effects [8].

In previous works [9,10], using a patterned template, the interests were limited to growth of hetero structures on

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mesa islands of finite sizes and there have not been studies on the improvements of electrical and optoelectronic properties of the epitaxial layers. In contrast, in this paper, we focus on the pattern guided heteroepitaxial growth of Ge within a restricted area. The Ge growth was performed on exposed Si surface in different sizes of trench holes which are isolated by oxide walls. This scheme provides a more desirable way for nano-scaled device integration by combining isolation process with epitaxial growth for process simplification. The heteroepitaxially grown Ge thin films were characterized by etch pit dislocations (EPDs) and Hall mobility using van der Pauw measurements for samples with different feature sizes. The size dependent EPD reduction and size dependent mobility enhancement were achieved clarifying the material quality. Raman spectroscopy was also employed to characterize residual strain of the epitaxial films with respect to the pattern sizes.

2. Experimental procedures

To make a template for the pattern guided epitaxial growth, a 300 nm-thick thermal SiO_2 was initially grown on a p-type Si (1 0 0) substrate (2–5 Ω cm) and then printed with a Leica electron beam direct writer to define square-shaped hole arrays along <1 1 0> directions. Each square was designed down to $200 \times 200\,nm^2$ from $200 \times 200\,\mu m^2$ and separated one another by at least 400 nm. Then the oxide was etched by anisotropic reactive ion etching (RIE) to expose Si surface through via holes. Fig. 1 (a) shows a 2D array of the patterns drawn for 200 nm squares and Fig. 1 (b) verifies a complete opening of the via holes showing a flat bottom profile and slightly higher vertical distance (~310 nm) after the RIE etching than the deposited thermal oxide thickness. The patterned template was chemically cleaned in a hot bath (~100 °C) Piranha solution, which consists of 4 parts of sulfuric acid (H_2SO_4) and 1 part of hydrogen peroxide (30 wt% of H_2O_2). Before introducing the template into a load-lock chamber, 5 s dip in 25:1 HF (30%) was performed to remove chemical oxide formed by the Piranha in the via holes. In-situ thermal desorption (900 °C, 5 min) was also performed in an ultra high vacuum growth chamber to remove residual native oxide and other surface contaminants. The substrate temperature was then cooled down to and kept at growth temperature and 1 mono layer (ML) of antimony (Sb) was deposited before Ge growth. The Sb has been known as segregating species and can act as surfactants to inhibit 3D island formation allowing for smooth wetting and continuous film growth [11,12]. During the Ge growth, nominal growth rate was maintained with 0.5 Å/s and substrate temperature was kept at 650 °C. After 300 nm of Ge growth, the substrate temperature was elevated to 700 °C and annealed for 5 min in order to flash-off the Sb segregated on top.

For an etch pit dislocation density measurement, a mixture of glacier acetic acid (CH_3COOH, 44 ml), nitric acid (HNO_3, 40 ml), hydrofluoric acid (30% HF, 20 ml) and 120 mg of iodine (I_2) was used to selectively decorate EPDs [13]. The statistics of EPDs with respect to the pattern sizes were obtained with a Nomarski interference microscope and SEM by inspecting all squares from 200×200 to $200 \times 200\,nm^2$. Raman spectroscopy (Renishaw 1000 μ-Raman system with a Leica microscope and excitation wavelength (λ) of 514.5 nm from an Ar^+ ion laser) and atomic force microscopy (AFM) were also employed to determine strain relaxation and surface morphology of the Ge epitaxial grown layers. For the Hall measurements, metal pads were formed with a 20 nm-thick titanium (Ti) layer and a 500 nm-thick aluminum (Al) using a metal lift-off process and image reversal printing to minimize the processing steps. Variable temperature Hall measurements were performed with a magnetic field of 0.35 T in a cryogenic Dewar (Janis, ST-300) for both heteroepitaxial and homoepitaxial Ge layers grown under identical conditions, where the homoepitaxial Ge was compared as a reference.

3. Results and discussion

3.1. Dislocation density in Ge heteroepitaxial layers

Fig. 2 shows the normalized pit densities obtained from statistics as a function of the pattern area and the

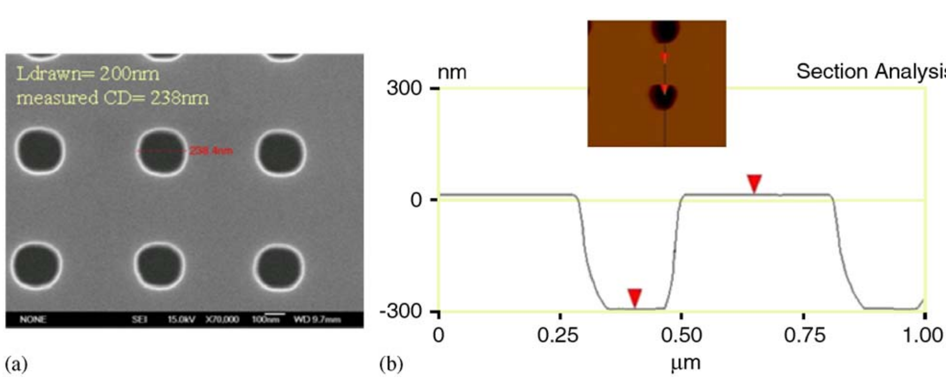

Fig. 1. (a) 2D array of via holes with 238 nm of measured CD and (b) AFM image with a line scan result showing a flat bottom profile and slightly higher vertical distance (~308 nm) than deposited SiO_2 thickness.