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The evolution of Si-capped Ge islands on Si (100) by RF magnetron sputtering and rapid thermal processing: The role of annealing times

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A B S T R A C T

Germanium (Ge) nanocrystals were synthesized by rapid thermal processing (RTP) of radio frequency sputtered Ge on silicon (100) substrate. A Si capping layer of 185 nm thickness was deposited onto the 225 nm Ge layer. The layered samples subsequently underwent annealing during RTP at 900 °C for 30, 45, and 60 s to subsequently evolve into Ge islands. Scanning electron microscopy (SEM) showed that as the annealing time increased, the Ge islands' size increased from 100 nm to 500 nm, and they became more uniform and dense. Atomic force microscopy (AFM) indicated that the increase in annealing time reduced the surface roughness by approximately 50%. Raman spectra showed that good crystalline nanostructures of the Ge peaks were obtained for all samples, with increased annealing time improving the crystallinity. A visible broad band photoluminescence was observed from UV to green with blue shift as nanocrystallite size decreases. High resolution X-ray diffraction (HR-XRD) revealed cubic and tetragonal Ge phases in the samples with low tensile strain around the Ge islands. The results indicated that both annealing ambient temperature and time do significantly influence the formation and evolution of the Ge islands on Si.

1. Introduction

The development of high-frequency heterojunction bipolar transistors has led to considerable interest in the growth of germanium (Ge) on silicon (Si) [1]. One of the major advantages of Ge is its compatibility with conventional Si-integrated technology and its potential for incorporation into low-dimensional self-assembled Ge islands. Because of its high carrier mobility and unique optical properties at the nano scale, Ge has potential for application in photonics and high-speed devices [2]. Fundamentally, the Ge islands' self-organization follows the so-called strain-driven Stranski–Krastanov (S–K) growth mode due to the 4.2% lattice mismatch with Si substrate [3,4]. Interesting morphological investigations show the shape of the Ge nanocrystal transforms from small, low temperature huts into larger and higher-temperature square base pyramids, domes, and superdomes.

Ge nanocrystals or quantum dots are mostly grown on Si substrates, either by molecular beam epitaxy [5,6] or by chemical vapour deposition [7]. Most studies have been performed on Si(001) surfaces, and some have been done on Si(111) [8,9]. The size of the islands grown can be precisely controlled to provide size quantization effects at low and room temperature [10]. Studies have been performed to identify the optimum growth temperature [2] and evolution of the Ge islands with in-situ post growth annealing [11]. However, these methods still rely on costly growth apparatus. Baharin et al. [12] demonstrated an alternate way of growing Ge islands using the low-cost method of thermal evaporation and rapid thermal annealing (RTA). The challenge in this technique lies in controlling the size and position of the Ge islands because of the non-epitaxial nature of the deposited Ge layers. Nonetheless, the non-uniform Ge islands beneath the Ni contact on the Si substrate have been shown to suppress the dark current and enhance the photocurrent of metal–semiconductor–metal photodetectors. Another alternative growth method used by Saha et al. [13] demonstrated that sputtered ion-beam of Si films followed by annealing in nitrogen(N_2) or hydrogen(H_2) ambient (600–900 °C for 30 min) produces microcrystalline films. Later, Carder et al. [14] has shown possible Ge nanostructures formation on Si substrate by post growth electron beam annealing of sputtered ion-beam Ge films.

A good, uniform Ge layer can also be produced using the cost-effective technique of plasma-assisted deposition, a technique

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which is compatible with planar device technology. However, there are limited reports available in the literature on the growth properties of Ge nanostructures on the Si substrate produced using this technique. Das et al. [15] demonstrated the potential growth of Ge islands on Si(001) substrates and in a SiO_2 matrix using the simple radio frequency (RF) magnetron sputtering method. They have shown that the growth of energetically stable domes is favourable in this technique. The Ge islands were grown after subsequent annealing at different temperatures in a N_2 atmosphere for 1 and 2 h. In general, the conversion of amorphous samples to nanocrystals requires annealing the samples from 30 min up to few hours. Prolonged heating however, may not be compatible with the future submicron IC fabrication processes. As an alternative, rapid thermal processing (RTP) has been shown to be very effective in increasing the growth or the recrystallization of Si and Ge in shorter time compared to conventional furnace annealing [16]. Choi et al. [16] showed that Ge nanocrystals embedded in a SiO_2 film were obtained by annealing for 300 s at temperatures of 800 °C and above; they used RF co-sputtering of Ge and SiO_2 followed by RTA for 300 s. The common structural characteristic showed that the Ge quantum dots were randomly distributed in the SiO_2 matrix with non uniform size. However in applications, the dots need to be accurately controlled with the position, size and density. Recent work by Nie et al. [17] has shown the formation of Ge-rich quantum dots embedded in SiO_2 matrix with the position accurately located upon the as-grown quantum dot formed by direct annealing of Si-capped Ge quantum dots in an oxygen atmosphere. This triggered the idea that the use of Si capping layer on top of the Ge deposited layer can promote the formation of uniform Ge islands and help to passivate the Ge island from oxidation during the evolution.

Herein, we used the cost-effective and simple technique of RF magnetron sputtering of the Ge layer on Si(100) substrate followed by Si capping. The samples were annealed at 900 °C for much shorter times of 30, 45, and 60 s to grow self-assembled uniform and dense Ge islands on the Si(100) substrate. In this technique, the substrate was not heated during the RF sputtering. The surface showed no evidence of nanostructure formation immediately after the growth. However, when the samples underwent RTP, significant surface roughening at the nanoscale was observed. The effect of the rapid annealing times used for the formation of the Ge islands on the surface morphology, crystallinity and optical property of the Ge islands were evaluated and the growth mechanism of the Ge islands was proposed.

2. Experimental procedures

The experiment began with standard Radio Corporation of America (RCA) cleaning of several n-type <100>-oriented silicon wafers with a resistivity of 1–10 Ω cm diced into $10 \times 10\ mm^2$ samples. After the cleaning, a Ge target of 99.999% purity (obtained from a commercial source) was sputtered onto the Si substrate using an Edwards A500 RF sputtering unit with RF power of 110 W for 30 min in a vacuum with a background pressure of 2.5×10^{-5} Torr. This was followed by the deposition of the Si capping layer from a commercial Si target of 99.999% purity using the same method at RF power of 150 W for 60 min. The estimated Ge and Si capping layer thicknesses were 250 nm and 150 nm, respectively, as measured by spectroscopic reflectometry (Filmetrics). Next, the samples underwent RTP at 900 °C for 30 s, 45 s, or 60 s in flowing N_2 at 500 sccm using a Jipelec JetFirst 100 rapid thermal processor to grow the Ge islands on the Si substrate. The samples were supported by a 4″ silicon wafer during RTP, and the temperature was monitored using a pyrometer placed underneath the wafer. Structural analyses of the 30 s, 45 s, and 60 s samples were performed using scanning electron microscopy (SEM, JEOL JSM-6460LV), energy dispersive X-ray analysis (EDX), and, for surface analysis, atomic force microscopy (AFM). The optical measurements were conducted using integrated PL-Raman spectroscopy (Jobin Yvon HR800UV) and high-resolution X-ray diffractometry (HR-XRD, PANalytical). All measurements were made at room temperature.

3. Results and discussion

3.1. Surface morphology

Fig. 1 shows a cross-sectional SEM image of the initial (as-grown) Ge and Si capping layers deposited on the Si(100) substrate. The layers showed acceptable uniformity. The measured average thickness of the Si capping layer was 185 nm and that of the Ge layer was 225 nm; these values are comparable with measurements made using spectroscopic reflectometry.

Fig. 2 presents SEM micrographs of samples grown at 900 °C for three different annealing times. With RTP at 900 °C for 30 s, the Ge and Si capping layers rapidly began to transform into very uniform Ge-rich or SiGe nano-islands with estimated average sizes ranging from 100 nm to 200 nm (Fig. 2a).

However, the islands became bigger and non-uniform at the annealing time of 45 s. The tabulated sizes of the islands were between 100 nm and 500 nm (Fig. 2b). A drastic transformation of island growth from non-uniform to more uniform islands occurred at the annealing time of 60 s (Fig. 2c): the island density and the island size increased significantly relative to those at 45 s. The sample was covered with dense islands with an average size of 500 nm. This represents layer-to-island mass transfer upon annealing, which is also evident in the AFM results. In addition, uniform nano-islands of about 50 nm were also observed between the bigger islands; these could have formed from the wetting layer that was left.

The data show that island density increased from around ($2.2 \times 10^8\ cm^{-2}$ to $12.2 \times 10^8\ cm^{-2}$) when annealing time increased from 45 s to 60 s. Because of their size, the big Ge islands were most likely partially or totally relaxed, with a large number of misfit dislocations at the Ge/Si interface. In general, island density was lower and the size of islands was larger in this study compared to density and size data reported elsewhere [18]. In Fig. 2, the respective EDX spectrum of each sample is shown to the right of the SEM micrograph. The EDX was taken at two spots: the white spot (W), which was believed to be the Ge-rich islands, and the black spot (B), which was the area between the islands. The three samples show that as the annealing time increased (from 30 s to

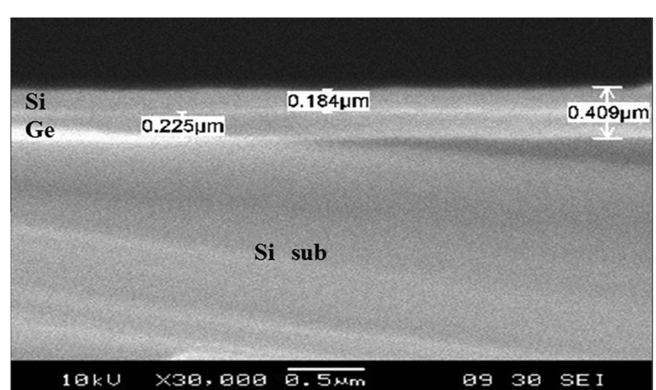

Fig. 1. Cross-sectional SEM image showing initial (as-grown) Ge and Si capping layers on the Si(100) substrate.