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Structural and Optical Characterizations of Ge Nanostructures Fabricated by RF Magnetron Sputtering and Rapid Thermal Processing

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In this work, we use a simple and cost effective technique of sputtering followed by the rapid thermal processing at 900 °C for 30 s to form Ge nanostructures on the Si(100) substrate. A layer of Ge (300 nm) and Si cap layer (100 nm) were deposited using RF magnetron sputtering. Two samples were prepared: Ge layer with Si capping (Si/Ge/Si) and Ge layer without Si capping (Ge/Si). Scanning electron microscopy shows that subsequent annealing in a rapid thermal processing gives uniformed Ge or SiGe islands with an estimated size of 100–500 nm. For the Ge/Si sample, under post growth annealing there had vanished the deposited Ge layer as confirmed by energy dispersive X-ray analysis. Atomic force microscopy shows that good crystalline structures of the Ge and SiGe peaks are produced. High resolution X-ray diffraction reveals cubic and tetragonal Ge phases with estimated average crystallite sizes of 42 nm and 20 nm, respectively. The results showed that it is possible to grow high quality Ge and SiGe nanostructures using a simple technique of sputtering for potential applications in photonics and high speed devices.

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1. Introduction

Ge/Si nanostructure has attracted great attention for its potential application in optoelectronic devices. Germanium is an interesting group IV semiconductor for its high carrier mobility and its display of unique optical properties at the nanoscale which holds potential for the applications in photonics and high speed electronics [1]. A variety of techniques have been employed to grow such structures, the most popular one is the self-assembled growth nanometer islands in a highly strained system using the molecular beam epitaxy (MBE) and low pressure chemical vapor deposition (LPCVD) techniques [2, 3]. However, these techniques require a sophisticated machine and the cost is very high. On the other hand, Baharin and Hashim [4] have shown a possible way of growing Ge islands using low cost methods of thermal evaporator and rapid thermal annealing. The challenge in this technique is to control 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

2. Experimental procedures

Several *n*-type $\langle 100 \rangle$ -oriented silicon wafers with a resistivity of 1–10 Ω cm were cleaned in a wet chemical

been shown to suppress the dark current and enhance the photocurrent of metal-semiconductor-metal photodetector. A good Ge epitaxial layer can be produced using plasma-assisted deposition. Limited reports are available in the literature on the growth properties of Ge nanostructures on the Si substrate using plasma-assisted deposition. Das et al. [5] in their study have demonstrated the potential growth of Ge islands on Si(001)substrates and in SiO₂ matrix using the simple radio frequency (RF) magnetron sputtering method. The islands were grown after subsequent annealing at different temperatures in N_2 atmosphere for about 1 h. In addition, rapid thermal processing (RTP) has shown to be very effective in increasing the growth or the recrystallisation of Si and Ge compared to the conventional furnace annealing [6]. Hence in this work, the cost effective and simple technique of RF magnetron sputtering coupled with subsequent RTP will be utilized to grow uniformed Ge islands on the Si(100) substrate. Structural and optical characterizations of the structures will be discussed.

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etch process, using the RCA cleaning method before the Ge layer was deposited. To elaborate, after cleaning, a Ge with 99.999% purity commercial source was then deposited (estimated thickness ≈ 300 nm) onto the Si substrate by using the Edwards A500 RF sputtering unit with RF power of 110 W for 30 min in a vacuum condition with a background pressure of 2.5×10^{-5} Torr. This is followed by the deposition of the Si capping layer (estimated thickness ≈ 200 nm) by the same method at RF power of 150 W for 60 min. After that, the sample underwent rapid thermal annealing at 900 °C for 30 s in flowing N₂ at 500 sccm using Jipelec JetFirst 100 RTP to form the Ge islands. Three samples were prepared: as deposited Si/Ge/Si layer (as dep), Ge island on the Si substrate (Ge/Si) and Ge islands on the Si substrate with its Si capping layer (Si/Ge/Si). Structural and optical analyses of the samples were performed using JEOL JSM--6460LV scanning electron microscope (SEM), an energy dispersive X-ray analysis (EDX), Jobin Yvon HR800UV Raman spectroscopy and a high-resolution PANalytical X-ray diffractometer (HR-XRD). All measurements were made at room temperature.

3. Results and discussion

Figure 1 shows SEM images of the as deposited, Ge/Si and Si/Ge/Si samples. The as deposited sample showed a smooth surface with substantial Si and Ge weight contents of about 41.15% and 39.05%, respectively, as shown by the EDX spectra on the right of Fig. 1a. A very low oxide content is also found in the structure suggesting the possibility of SiO₂ being formed. The Ge/Si sample in Fig. 1b shows rough and spongy structures emerged after RTP compared to the as deposited sample. While for the Si/Ge/Si sample which was put under rapid annealing of 900 °C for 30 s, the layer slowly emerged into uniform Ge or SiGe islands as can be seen from the Si/Ge/Si sample (Fig. 1c). The estimated Ge islands' size formed, ranged from 100 nm to 500 nm. On the right of each sample there is the respective EDX of the samples. For the Ge/Si and Si/Ge/Si, the EDX was taken at 2 spots which was at the white spot (W) believed to be the islands and black spot (B) which was the area between the islands. For Ge/Si (Fig. 1b) both EDX spectra show only Si contents which confirms that the RTP had made vanished the Ge layer on the Si substrate. However, the Si/Ge/Si sample shows the presence of Ge while the presence of low Ge peak on the black spot suggests the presence of a thin wetting layer between the Ge islands. The presence of oxygen content suggests the possibility of GeO presence in the structures.

The surface morphology of the samples was examined from the atomic force microscopy (AFM) micrograph (Figs. 2a and b). As deposited Si/Ge/Si sample proves, the grains are uniformly distributed within the scanning area (30 μ m ×30 μ m), with individual columnar grains extending upwards. The average grain height is found to be in the range of 8.97 nm and the root mean square



Fig. 1. SEM and EDX graphs of (a) as deposited Si/Ge/Si, (b) Ge/Si, and (c) Si/Ge/Si: W = white area and B = Black area.

(RMS) is equal to 3.4 nm. This indicates that the deposited Si capping and Ge layer using the RF magnetron sputtering have exhibited a very smooth film surface for the subsequent Ge islands formation. The significant increase of the roughness of the surface, and the height of the islands have been observed on the Si/Ge/Si sample (Fig. 2b). Specifically the RMS of the surface roughness of Si/Ge/Si has increased to 52.87 nm which corresponds to 15.55% increase.



Fig. 2. AFM of (a) as deposited and (b) Si/Ge/Si samples at room temperature.

Figure 3 shows the Raman spectrum of the three samples taken at room temperature. In general, the as deposited sample shows no Raman peaks in the scan range indicating the absence of crystalline phases and the structure of the deposited film that could be disordered. However, the inset in Fig. 3 shows a low and broad hump centred at 274.9 cm^{-1} with full width at half maximum (FWHM) of 77.3 $\rm cm^{-1}$ corresponding to the amorphous Ge [6, 7]. As the film was annealed at 900 °C for 30 s (Si/Ge/Si), the appearance of pronounced Raman peak at 292.1 $\rm cm^{-1}$ with asymmetrical broadening over the low frequency side emerged, indicating that a good Ge nanocrystal had been obtained. This peak is attributed to the optical phonon contribution of the Ge-Ge stretching mode [5] and is red-shifted by $\approx 8 \text{ cm}^{-1}$ as compared to that of the bulk Ge (300 cm^{-1}) [8], which is in agreement with the phonon confinement theory pre-



Fig. 3. Raman spectroscopy of as deposited, Ge/Si, and Si/Ge/Si at room temperature. Inset shows the enlarged Ge peak and SiGe peaks.

diction for nanocrystals [9] whereby as the average size decreases the Raman peak broadens and shifts slightly to lower frequencies. Another sharp peak at $405.2 \text{ cm}^$ with FWHM of 16.9 $\rm cm^{-1}$ is also observed which corresponds to the Si–Ge alloy mode [10]. In addition, there exist small peaks at 432.4 cm^{-1} and 484.9 cm^{-1} which belong to Ge-Si and local Si-Si vibration mode [11], indicating the intermixing of Ge-Si. The red-shifted bond of Si–Si at 509.6 $\rm cm^{-1}$ demonstrates a tensile strain in the Si cap layer possibly caused by the difference in lattice parameters of Ge and Si. For the Ge/Si sample, only the Si–Si phonon mode at about 520 $\rm cm^{-1}$ is observed. This is in agreement with the previous EDX spectra which show only the Si content left after subsequent annealing in RTP. This shows that with Si cap layer, the film can adequately transform into uniformly good crystalline Ge and SiGe islands at rather high temperature during the annealing process. The Si/Ge/Si probably consists of relaxed Ge cores surrounded by the SiGe shells whereby the Si cap layer must be alloyed with Ge to form a SiGe shells around the Ge cores. It is possible that the Si cap layer plays a role in relieving stress and acts as a capping factor protecting the Ge core from getting oxidized [12].

Figure 4 shows the measured high resolution X-ray diffraction (HRXRD) of the three samples. The asdeposited and Ge/Si samples spectra reveal the presence of only Si phases with the orthorhombic structure by (220) and (421). Another pronounced peaks at about 69° and 82° correspond to (400) and (440) reflections of the Si cubic structures. In general, the Ge/Si sample shows decreasing intensity compared to as-deposited sample, except at 69° where the peak for Ge/Si is much stronger than the former. This suggests that in high temperature the top deposited Ge layer vanished and there left only the Si substrate beneath it, while the as-deposited sample reveals only the Si peak from the Si cap layer and the Si substrate. The Si/Ge/Si sample reveals Ge cubic phases



Fig. 4. HRXRD of (a) as deposited, (b) Ge/Si, and (c) Si/Ge/Si samples taken at room temperature.

by (111) and (220) and Ge tetragonal phases by (201) and (113), respectively. The estimated average Ge crystallites size using Scherrer's equation ranges from 42 nm (from the Ge cubic) to 20 nm (from the Ge tetragonal) phases. In addition, we can see that the dominant peak at about $2\theta = 69.1^{\circ}$ is the (400) diffraction from the Si substrate [13] and a small peak at 82.1° belong to the cubic Si (440) reflection.

4. Conclusions

We have shown that it is possible to grow Ge and SiGe nanostructures on the Si (100) substrate by using a simple and cost-effective technique of the RF magnetron sputtering and rapid thermal processing. SEM showed that subsequent annealing in rapid thermal processing at 900 °C for 30 s resulted in uniform Ge or SiGe islands with estimated sizes of 100–500 nm. The AFM shows that the surface roughness increased by a factor of 15.55% as the islands were formed. For the Ge/Si sample, under post--growth annealing there vanished the deposited Ge layer as confirmed by EDX, Raman and XRD. The Raman spectrum shows that good crystalline structures of the Ge and SiGe peaks can be produced from the annealed sample with the Si cap layer. HRXRD reveals cubic and tetragonal Ge phases with estimated average crystallites size ranging from 42 nm to 20 nm, respectively, from the Si/Ge/Si sample. The presence of the Si cap layer on top of the Ge layer deposited has shown to prevent the Ge layer from vaporizing and helps to form good Ge and SiGe nanostructures. The results exhibited that it is possible to grow high quality Ge and SiGe nanostructures using conventional techniques for potential applications in photonics and high speed devices.

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