



# Effect of isochronal hydrogen annealing on surface roughness and threading dislocation density of epitaxial Ge films grown on Si

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## ARTICLE INFO

Available online 21 October 2009

### Keywords:

Germanium

Silicon

Hetero-epitaxy

Chemical vapor deposition

Hydrogen annealing

Dislocation

## ABSTRACT

We report the effect of hydrogen annealing on the surface roughness and threading dislocation density (TDD) of germanium (Ge) films grown on silicon (Si) substrates by reduced-pressure chemical vapor deposition (RPCVD). The surface roughness initially decreased with an increase in the annealing temperature. At annealing temperatures greater than 650 °C the film thickness varied owing to surface undulations, leading to an increase in the surface roughness. Although high-temperature annealing at 850 °C is effective for reducing TDD, the surface roughness of a 150-nm-thick Ge film annealed at 650 °C reaches a minimum value (~0.7 nm).

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## 1. Introduction

Germanium (Ge)/silicon (Si) heterostructures are being increasingly used for the fabrication of novel devices that are compatible with Si-based technologies. In particular, Ge is a very promising material as a future channel material for nanoscale metal-oxide-semiconductor field-effect transistors (MOSFETs) due to its high mobility [1], and it finds an important application in the fabrication of photodetectors on Si [2]. Moreover, epitaxial growth of Ge on a Si substrate is an important method for producing suitable substrates for III–V-based devices [3]. For successful fabrication of these high-speed MOSFETs and optical devices, it is necessary to reduce the surface roughness and threading dislocation density (TDD) of Ge films.

Nayfeh et al. demonstrated that hydrogen annealing results in a dramatic improvement in the surface roughness of Ge films [4]. They found that annealing at 825 °C resulted in almost a 90% decrease in the surface roughness, i.e., from 25 nm to 2.9 nm. Further, they developed a novel technique that involved multiple growth and hydrogen annealing steps for depositing high-quality heteroepitaxial Ge films on Si [1]. Several research groups have used a two-step deposition method to grow thick Ge films with a smooth surface on Si [5–9]. In the first step, a 30–50-nm-thick seed layer is deposited in the layer-by-layer growth region at 330–400 °C. In the second step, the growth temperature is increased from 600 to 850 °C for the formation of a high-quality flat Ge film at a high deposition rate. Recently, Choi et al. grew high-quality, pure thick Ge films on Si substrates by using a combination of Nayfeh et al.'s method and the abovementioned two-

step deposition method [10]. They found that minimum values of the TDD and the root-mean-square (RMS) surface roughness ( $R_{\text{RMS}}$ ) of 1–2- $\mu\text{m}$ -thick samples are  $(0.8\text{--}1.0) \times 10^7 \text{ cm}^{-2}$  and 0.4–0.6 nm, respectively. Hydrogen annealing is thought to be the most important process not only in the multiple deposition and annealing steps but also during the second high-temperature growth step in the two-step deposition method; however, the net effect of this annealing on the resultant film remains to be clarified.

In this study, we deposited one 150-nm-thick Ge film on Si substrate by reduced-pressure chemical vapor deposition (RPCVD) and subsequently annealed in a hydrogen ambient. Since the two-step deposition method was not adopted for this purpose, the substrate was maintained at a low temperature (310–350 °C) during deposition. Therefore, this paper only reports the effect of isochronal hydrogen annealing on the reduction in the surface roughness and TDD of the deposited Ge films.

## 2. Experimental

The samples were grown and annealed in an industrial RPCVD system (Applied Materials Epi Centura). Si(100) substrates were first cleaned using a 4:1  $\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$  mixture, and then with a 1:1:4 mixture of HCl,  $\text{H}_2\text{O}_2$ , and high-purity deionized (DI) water; subsequently, the substrates were rinsed with high-purity DI water. The substrates were then dipped into 2% HF for native oxide removal, rinsed with DI water, and immediately loaded into the reactor. After hydrogen annealing at 1100 °C, an epitaxial Si buffer layer was deposited on the Si substrate to reduce oxygen and carbon pile-up at the Ge/Si heterointerface [11]. Epitaxial Ge films (thickness: ~150 nm) were grown at 310 and 350 °C using a  $\text{GeH}_4\text{--H}_2$  gas mixture. Immediately after the epitaxial growth,

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hydrogen annealing was performed for 15 min in the temperature range of 450–850 °C at a pressure of 11 kPa. The surface roughness of the Ge films was determined by tapping-mode atomic force microscopy (AFM). The TDD of the films was determined by plan-view transmission electron microscopy (TEM). The dislocations formed in the films were analyzed by cross-sectional TEM observations.

### 3. Results and discussion

The growth mode of Ge on Si during RPCVD was found to be critically dependent on the substrate temperature ( $T_s$ ). Above 400 °C, large Ge islands were formed on the Si substrate [8,12]. The Ge layers grown below 400 °C were characterized by three different surface morphologies, as shown previously by Olubuyide et al. [9]. At low  $\text{GeH}_4$  partial pressures ( $P_{\text{GeH}_4}$ ), i.e.  $P_{\text{GeH}_4} < 2$  Pa, the growth of small islands with diameters of a few tens of nanometers occurred due to the suppression of adsorption and decomposition of  $\text{GeH}_4$  on Si–H bonds [13]. At  $P_{\text{GeH}_4} > 30$  Pa, the growth of large islands with diameters of a few hundred nanometers was favored. Between the two abovementioned pressure regimes, i.e., at  $P_{\text{GeH}_4} \sim 10$  Pa, a layer-by-layer growth region was found to exist.

Fig. 1(a) shows the  $5 \times 5 \mu\text{m}^2$  AFM scan of an as-deposited Ge film (sample A;  $T_s = 310$  °C;  $P_{\text{GeH}_4} = 32$  Pa). Fig. 1(b–f) show the AFM images of the Ge films after 15 min of isochronal hydrogen annealing in the temperature range of 450–850 °C. The growth of islands in the  $\langle 110 \rangle$  direction can be clearly observed in Fig. 1(a). Islands are formed on the flat film surface and  $R_{\text{RMS}}$  is 18.9 nm. After isochronal annealing at 450 °C, surface pits are formed along with small islands. Above 650 °C, only surface pits with a diameter of  $\sim 1 \mu\text{m}$  exist. Similar pits have often been observed on the surface of Ge films deposited by the previously mentioned two-step deposition method [14].

Fig. 2(a) shows the  $5 \times 5 \mu\text{m}^2$  AFM scan of an as-deposited Ge film (sample B;  $T_s = 350$  °C;  $P_{\text{GeH}_4} = 12$  Pa), while Fig. 2(b–f) show the AFM images of the Ge films after isochronal hydrogen annealing. Since the Ge film is deposited in the layer-by-layer growth region, its surface is very smooth ( $R_{\text{RMS}} = 1.2$  nm), and the film has a thickness of 150 nm (Fig. 2(a)). A small undulation with a wavelength of  $\sim 0.1 \mu\text{m}$  can be seen on the as-deposited Ge surface, and the surface of this film becomes smoother with an increase in the annealing temperature. After annealing at 450 and 550 °C, surface pits with a diameter

of  $\sim 0.5 \mu\text{m}$  appear on this film and the surface pit density of this sample was less than that of sample A. The surface undulations reappear at temperatures above 650 °C. The undulation wavelength increases with the annealing temperature.

Fig. 3(a) shows the cross-sectional TEM images of sample A before and after isochronal hydrogen annealing. Ge islands are formed on the as-deposited film surface, as seen in Fig. 1(a), with V-shaped defects in every island. Furthermore, threading dislocations and stacking faults, as well as misfit dislocations at the Ge/Si heterointerface, can be seen in the film. After annealing at 450 °C, the Ge islands are transformed into pits. Above 650 °C, the stacking faults and V-shaped defects disappear, while the misfit dislocations are mostly confined to the Ge/Si heterointerface and do not thread to the surface. Fig. 3(b) shows the cross-sectional TEM images of sample B before and after hydrogen annealing. No V-shaped defects are seen in the as-deposited film, and the film surface is smooth with no island formation. Above 650 °C, stacking faults are no longer observed, and the misfit dislocations are mostly confined to the Ge/Si heterointerface. In particular, at 850 °C, misfit dislocations are confined to the Ge/Si heterointerface or run parallel to it. A majority of the observed dislocations in the film annealed at 850 °C are 90° full-edge dislocations and the average spacing between the dislocations in Fig. 3(c) and (d) is  $\sim 10$  nm which is consisted with that reported previously [6,15].

Fig. 4 illustrate the dependence of  $R_{\text{RMS}}$  and the Z range (i.e.  $Z_{\text{max}} - Z_{\text{min}}$ ) on the annealing temperature, as determined by  $10 \times 10 \mu\text{m}^2$  AFM scans. The surface roughness of sample A initially decreased with an increase in the annealing temperature, reaching a minimum value at 650–750 °C and increased thereafter. However, Nayfeh et al. [4] had previously reported that  $R_{\text{RMS}}$  decreases monotonically with the annealing temperature (Fig. 4(a)). After annealing,  $R_{\text{RMS}}$  of sample B decreased, reaching a minimum of  $\sim 0.7$  nm at 650 °C. On the other hand, as the annealing temperature increased, the Z range of sample B initially increased because of pit formation and then decreased to a minimum value at 650 °C because of pit deformation (Fig. 2). Above 650 °C, the undulation wavelength increased, causing a corresponding increase in the Z range; this in turn led to an increase in  $R_{\text{RMS}}$ . In the case of sample A, the Z range increased with the annealing temperature because of pit formation, reaching a maximum between 550 and 650 °C, and then decreased monotonically. The reason of

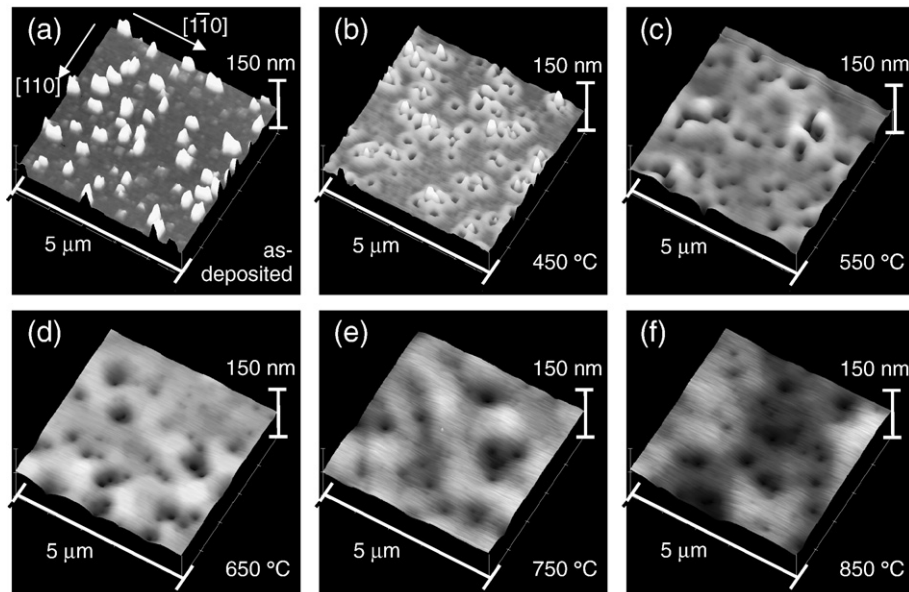


Fig. 1. AFM scans: (a) an as-deposited Ge film (sample A;  $T_s = 310$  °C;  $P_{\text{GeH}_4} = 32$  Pa); (b–f) Ge films after 15 min of isochronal hydrogen annealing at 450–850 °C.

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