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SyunMing Jang, HyounWoo Kim, and Rafael Reif

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Thermal stability of $Si/Si_{1-x}Ge_x/Si$ heterostructures deposited by very low pressure chemical vapor deposition

Syun-Ming Jang and Hyoun-Woo Kim
Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

Rafael Reif
Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

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We have studied the thermal stability of metastable $Si/Si_{1-x}Ge_x/Si$ strained structures deposited by very low pressure chemical vapor deposition at 620 °C. Samples after furnace anneals at 800–1100 °C for 30 min were characterized by double-crystal x-ray diffraction and plan-view transmission electron microscopy to determine residual strain and misfit dislocation structure. It is found that strains in ~1400 Å Si/520–700 Å $Si_{1-x}Ge_x/Si$ structures with Ge contents of 0.09–0.16 relax gradually at temperatures <950 °C but relax rapidly at temperatures >950 °C, showing a transition and different mechanisms in the relaxation process. The process, involving a single-kink dislocation mechanism as well as Si-Ge interdiffusion, has been investigated for $Si_{0.87}Ge_{0.13}$.

The great potential of Si-based heterojunction bipolar transistors has been demonstrated by recent work using $Si_{1-x}Ge_x$ as a base, and a very high cutoff frequency of 75 GHz has been achieved.[1] Since useful $Si_{1-x}Ge_x$ strained films are usually thicker than the equilibrium critical thickness[2,3] and integrated circuit processing routinely involves high temperature steps, the metastable strained layer may relax during elevated temperature exposure and degrade device performance. It is hence of practical interest to investigate strain relaxation in $Si_{1-x}Ge_x$ for maintaining a defect-free film after thermal treatments. In recent years, the thermal stability of $Si_{1-x}Ge_x$ with Si capping layers has been intensively studied.[4–13] The relaxation processes dominated by misfit dislocations are structure dependent, and possible mechanisms including double-kink (paired dislocation), which is frequently observed, and single-kink (single dislocation) models have been proposed to characterize strain relaxation.[5,9,10] Recently, the thermal stability of $Si_{1-x}Ge_x$ (≤1000 Å with various Ge compositional profiles) capped with a relatively thin Si layer of ≤500 Å deposited by ultrahigh vacuum chemical vapor deposition has been reported.[13] In all cases, relaxation due to 30 min furnace anneal at 950 °C accomplished via single-kink dislocation nucleation and glide was mentioned. In this letter, we report the thermal stability of very low pressure chemical vapor deposition (VLPCVD)[14] $Si/Si_{1-x}Ge_x/Si$ heterostructures throughout the temperature range of 800–1100 °C. We concentrated on very thin $Si_{1-x}Ge_x$ (≤700 Å) with boxlike Ge profile capped with a ~1400 Å Si layer, which is suitable for heterojunction bipolar transistors, and found that the single-kink mechanism also became important in these structures during annealing. The quantitatively measured strain and the corresponding misfit dislocation mechanisms for $x=0.13$ as a function of temperature are presented.

The films used in this study were grown at 620 °C by VLPCVD. The structural quality of the as-grown films were characterized by cross-sectional transmission electron microscopy (XTEM) and a dilute Schimmel etch (4 parts of 48% HF, 5 parts 0.3M CrO_3 for 1 min at room temperature). Both techniques showed no misfit dislocation. Rutherford backscattering spectroscopy was performed to determine Ge composition and layer thickness, and the results were confirmed by double-crystal x-ray diffraction (DCD) and XTEM. The as-grown samples were annealed in furnace at temperatures ranging from 800 to 1100 °C for 30 min in flowing N_2. Strain relaxation in $Si_{1-x}Ge_x$ was measured by DCD, and the presence and structure of misfit dislocations were determined by plan-view TEM.

In Fig. 1 we show results of DCD characterization of $Si_{1-x}Ge_x$ films with Ge compositions of 0.09, 0.13, and 0.16, and the thicknesses of $Si_{1-x}Ge_x$, which is buried by a ~1400-Å-thick Si capping layer, are 710, 550, and 520 Å,

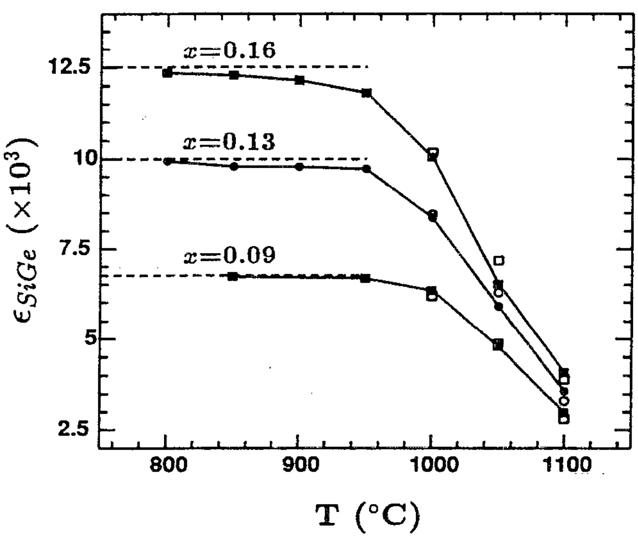

FIG. 1. Measured strain vs annealing tempertures for Ge=0.09, 0.13, and 0.16. The close symbols refer to the measured data, and the open symbols are from Eq. (4). The strains measured from as-grown samples are presented by the dashed lines.

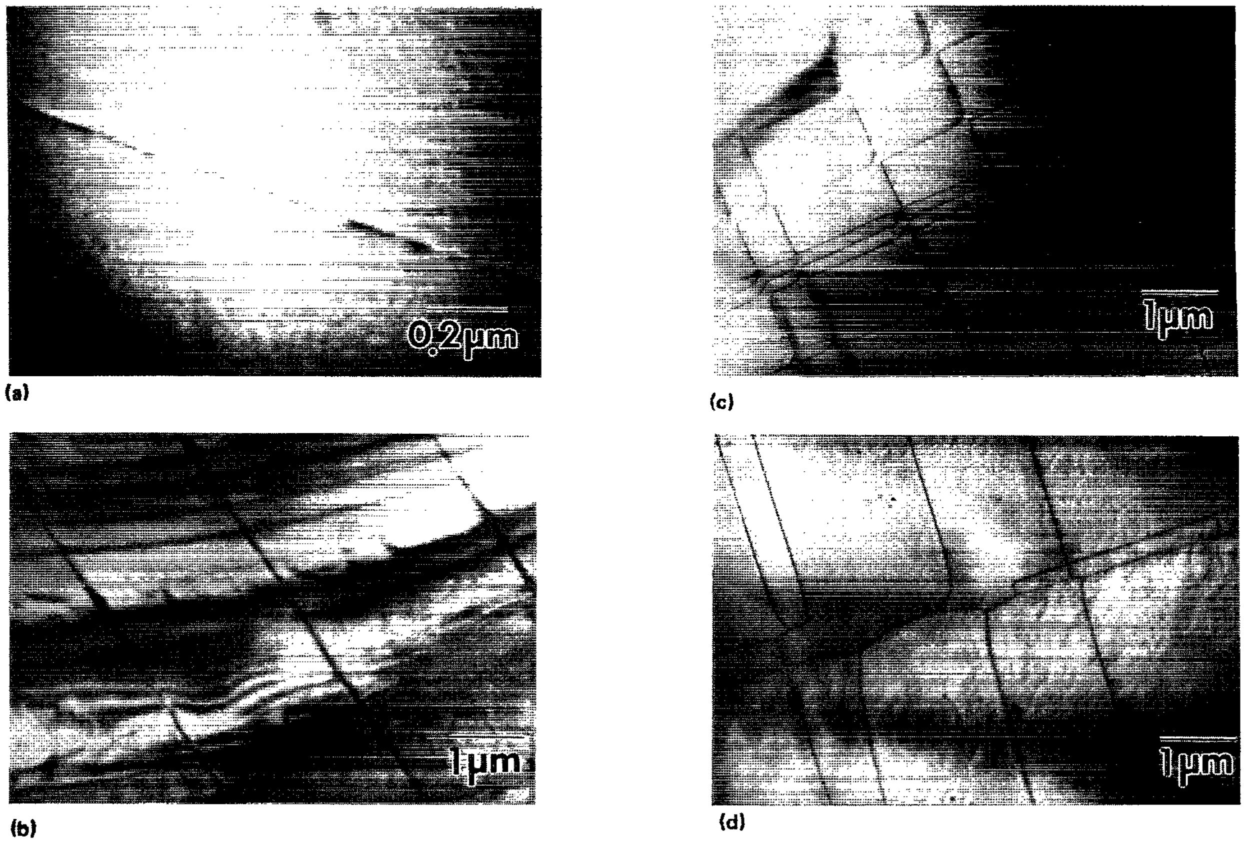

FIG. 2. Plan-view TEM micrographs of 1400 Å Si/550 Å $Si_{0.87}Ge_{0.13}$/Si annealed for 30 min at (a) 800 °C, (b) 900 °C, (c) 1000 °C, and (d) 1100 °C. Relaxation occurs only by single dislocations as $\tau_{\rm eff}^{S}=2.2\tau_{\rm eff}^{P}$.

respectively. The perpendicular lattice parameter of $Si_{1-x}Ge_x$, $a_{\rm SiGe}^{\perp}$, was obtained from (400) rocking curve and the strain $\epsilon_{\rm SiGe}$ is given by

$$\epsilon_{\rm SiGe}=(a_{\rm SiGe}^{\perp}-a_{\rm Si})/a_{\rm Si}, \qquad (1)$$

where $a_{\rm Si}$ is the lattice parameter of Si. Upon relaxation, $Si_{1-x}Ge_x$ film loses its coherency with Si substrate and $a_{\rm SiGe}^{\perp}$ (i.e., $\epsilon_{\rm SiGe}$) decreases as strain relaxation proceeds. From Fig. 1 we observe that relaxation takes place relatively gradually at temperatures <950 °C, but rapidly at $T>950$ °C, showing a transition in the relaxation process. To examine the process, $Si_{0.87}Ge_{0.13}$ films were characterized by plan-view TEM after receiving 30 min annealing at various temperatures, as shown in Figs. 2(a)–2(d). It is apparent that in such structure misfit dislocations retain a single-kink or two-segment configuration with dislocations created only at the interface between $Si_{0.87}Ge_{0.13}$ and the underlying Si, and no misfit dislocation resulted at the Si cap/$Si_{0.87}Ge_{0.13}$ interface. To realize the dislocation mechanism, following Houghton,[9] we compare the unbalanced force, i.e., effective stress $\tau_{\rm eff}$, driving 60° type misfit dislocation for single-kink (single dislocation, $\tau_{\rm eff}^{S}$) and double-kink (paired dislocation, $\tau_{\rm eff}^{P}$) mechanisms in strained $Si_{1-x}Ge_x$ buried layer using

$$\tau_{\rm eff}^{S}=2\mu\cos\psi\,\frac{1+\nu}{1-\nu}\left(\frac{h}{h+H}f\cos\lambda-\frac{b(1-\nu\cos^2\theta)}{8\pi(1+\nu)(h+H)}\ln\frac{4(h+H)}{b}\right) \qquad (2)$$

and

$$\tau_{\rm eff}^{P}=2\mu\cos\psi\,\frac{1+\nu}{1-\nu}\left[f\cos\lambda-\frac{b(1-\nu\cos^2\theta)}{8\pi(1+\nu)h}\times\left(\ln\frac{4(h+H)}{b}+\ln\frac{4H}{b}+2\ln\frac{h}{h+H}\right)\right], \qquad (3)$$

where, for 60° mixed $a/2\langle 110\rangle$ dislocations, $\psi=35°$ is the angle between the strained interface normal and the slip plane, $\theta=60°$ is the angle between the Burgers vector ($b=4$ Å) and the dislocation line, $\lambda=60°$ is the angle between the Burgers vector and the direction in the interface, normal to the dislocation line, $\nu=0.28$ is the Poisson's ratio, $f=0.042x$ is the lattice mismatch between Si and $Si_{1-x}Ge_x$, h is the $Si_{1-x}Ge_x$ layer thickness, H the thickness of Si capping layer, and μ the shear modulus of $Si_{1-x}Ge_x$. The results from Eqs. (2) and (3) with $x=0.13$, $h=550$ Å, and $H=1400$ Å give $\tau_{\rm eff}^{S}=2.2\tau_{\rm eff}^{D}$. Therefore, it appears that single-kink propagation is the dominant mechanism in this case.

Looking at Fig. 2, we also see different dislocation structures formed at different annealing temperatures. In

Fig. 2(a), discrete dislocation segments are observed after 800 °C annealing, indicating that homogeneous nucleation of dislocation half-loops may operate in the early stage of relaxation and the observed segments correspond to individual half-loops. At 900 °C, the sample exhibits more defects and the misfit dislocation lines extend up to at least 100 μm running in only one direction, while at 1000 °C the interactions of two orthogonal misfit dislocations with the same Burgers vector are evident. No strong evidence shows the complete Hagen–Strunk mechanism for dislocation multiplication,[15] although the required dislocation intersections appear and the Hagen–Strunk sources have been observed in our previous study using similar but uncapped $Si_{1-x}Ge_x$ films (600 Å $Si_{0.87}Ge_{0.13}$).[16] As temperature was increased to 1100 °C, misfit dislocations in distinct arrangements were formed, and the presence of curved dislocations was thought to be a result of dislocation climb activated at high temperature.

In Figs. 2(a)–2(d), the observed dislocations lead to strain relief at 800–1100 °C by the amount of $\sim b/2p$, where p is the dislocation spacing, and in this study we find that $b/2p$ is on the order of 10^{-4} or less and is consistent with the observed sluggish relaxation by DCD at $T < 950$ °C. However, since strain relief is much greater than 10^{-4} at $T > 950$ °C, the formation of dislocation is not sufficient to explain the significant relaxation at higher temperatures. Moreover, excess relaxation $[a^{\perp}_{SiGe} < a^{\text{total relax}}_{SiGe} = x a_{Ge} + (1-x) a_{Si}]$ was measured at 1000–1100 °C. This suggests that a new mechanism is in operation as annealing temperature increases. Such mechanism can be attributed to the interdiffusion of Si and Ge which has been reported to be a competing process compared to dislocation generation at high temperatures for relieving the misfit stress at the interface and is particularly significant for thin layers.[17] Since dislocation-induced relaxation is negligible at high temperatures, the strain is determined by Si-Ge interdiffusion and ϵ_{SiGe} can be estimated by

$$\epsilon_{SiGe} = \frac{1+\nu}{1-\nu}\bar{f} = \frac{1+\nu}{1-\nu}\frac{0.042}{h_{eff}}\int_{-h_{eff}/2}^{h_{eff}/2} x(z)dz \sim \frac{1+\nu}{1-\nu}\frac{0.042 x_0 h}{h_{eff}}, \quad (4)$$

where $\bar{f}$ is the average mismatch, $x(z)$ is the Ge profile modulated by diffusion, and h_{eff} the effective layer thickness of $Si_{1-x}Ge_x$ with Ge confined in the region $-h_{eff}/2 < z < h_{eff}/2$. Prior to annealing h_{eff} is the original $Si_{1-x}Ge_x$ thickness h and x_0 the original Ge composition. $\bar{f}$ can be written as $0.042 x_0 h/h_{eff}$ if Ge atoms at $z > h_{eff}/2$ and $z < -h_{eff}/2$ are negligible. To estimate h_{eff} we consider the finite-thickness solution to the Fick's second law for Ge diffusion into Si, $x(z) = x_0/2 \times \{\text{erf}[(z+h/2)/2\sqrt{Dt}] - \text{erf}[(z-h/2)/2\sqrt{Dt}]\}$, and use the Si-Ge interdiffusion coefficients for $x=0.15$ measured by Green *et al.*[18] Since $\sqrt{Dt}$ at 950 °C=23 Å, diffusion phenomenon can be considered negligible at temperatures below 950 °C. Assuming that $\sim$95% Ge atoms are within h_{eff}, we calculate ϵ_{SiGe} using Eq. (4) and find the calculated and measured values are in good agreement as indicated in Fig. 1. Clearly, the dominant mechanism at $T > 950$ °C is with Si-Ge interdiffusion and Ge profile controls the misfit strain.

In summary, strain relaxation in annealed $\sim$1400 Å Si/520–700 Å $Si_{1-x}Ge_x$/Si heterostructures ($x=0.09$–0.16) have been quantitatively measured and the corresponding misfit dislocation structures for $x=0.13$ are identified with respect to single-kink dislocation mechanism, which is explained in terms of τ_{eff} for single and paired misfit dislocation propagation. A transition from partially relaxation to fully and excess relaxation is revealed by DCD. Our experimental observations and calculations indicate that in these structures the sluggish relaxation observed at $T < 950$ °C is dominated by misfit dislocations, and the rapid relaxation at $T > 950$ °C is associated with Si-Ge interdiffusion.

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