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Hydrogen evolution from strained Si_xGe_{1-x}(100)2×1:H surfaces

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The evolution of hydrogen from a series of strained $Si_xGe_{1-x}(100)2\times1$:H surfaces were studied with angle resolved ultraviolet photoemission spectroscopy (ARUPS). A series of strained Si_xGe_{1-x} alloys were grown on Si(100) wafers using electron beam evaporation in an ultra-high vacuum molecular beam epitaxy chamber. The growth was followed by an *in situ* H-plasma exposure. After the *in situ* H-plasma exposure, a diffuse double domain 2×1 reconstructed surface was obtained, which indicates a Si(Ge)–H monohydride surface termination. ARUPS spectra of the series of H-terminated Si_xGe_{1-x} alloys were obtained as a function of annealing temperature. Hydrogen induced surface states/resonances were observed from the H-terminated surfaces of all samples, and as the annealing temperature was increased the states were gradually extinguished. The ARUPS spectra of the H-terminated alloy surfaces indicated that the monohydride started to dissociate at annealing temperatures ≤ 250 °C. The results show that, for all H-terminated Si_xGe_{1-x} alloys and Ge, the surface state attributed to the dangling bond was visible after annealing at ~ 250 °C. In contrast, annealing to >400 °C was required to desorb H from a pure Si surface. It is proposed that the hydrogen starts to desorb preferentially from the Ge sites on the strained Si_xGe_{1-x} alloys surfaces. © 1996 American Institute of Physics. [S0021-8979(96)08420-4]

I. INTRODUCTION

In situ low temperature processing is important for next generation integrated circuit fabrication. Hydrogen plasma exposure is capable of not only removing the carbon and oxygen contaminants from a Si surface but also passivating the surface with atomic hydrogen.^{1–3} It has also been reported that H-passivation has a significant effect in retarding the recontamination of the surface with oxygen and carbon.^{4,5} Furthermore, a clean surface can be easily established since hydrogen can be desorbed by annealing the surface at low temperatures.^{6–8}

The kinetics of hydrogen desorption from the Si(100) surface has been investigated extensively.⁹⁻¹¹ Recent work demonstrates that the desorption of molecular hydrogen from the monohydride phase of Si(100) surfaces follows firstorder kinetics.^{12,13} Furthermore, hydrogen desorption from the monohydride phase of Ge(100) surfaces was also found to be first order.¹⁴ In contrast, the traditional model¹⁵ of adatom "hopping" on a surface, followed by recombination and desorption, should give rise to second-order desorption kinetics. Sinniah et al. first proposed a two-step model for the desorption process.¹² Hydrogen adatoms that are chemisorbed in the ground state on Si(100) are thermally excited irreversibly into a two dimensional, delocalized band state and this excited, delocalized adatom then reacts with a localized hydrogen adatom to produce H2 which desorbs. A second model was proposed by Wise et al.,¹³ in which a reaction occurs between two H atoms that have previously paired at a site. Such a preferential pairing was indeed observed in a scanning tunnel microscopy (STM) study of Si(100)2×1:H surfaces¹⁶ and provides a natural explanation for the observed first-order desorption kinetics.13,16,17 The rate of hydrogen desorption from (100) surfaces of both silicon and germanium has been found to be strongly temperature dependent. $^{12-14,18,19}$

Currently Si_rGe_{1-r} alloys are receiving considerable attention, and the materials are being investigated for applications including optoelectronic devices and high speed heterojunction bipolar transistors.²⁰⁻²⁶ Silicon and germanium are completely miscible over the entire compositional range and give rise to alloys with the diamond crystal structure. Due to the lattice mismatch (4.17%) between silicon and germanium, the epitaxy of lattice mismatched materials results either in a strained (pseudomorphic) layer, if the layers are sufficiently thin, or in a layer relaxed by misfit dislocations. The interest in strained Si_rGe_{1-r} alloys is not only because of the lack of interfacial misfit dislocations but also because of a large variation in the band gap. The band gap of the Si_rGe_{1-r} alloys decreases as the Ge content increases, and a strained $Si_x Ge_{1-x}$ alloy exhibits a greater reduction in the band gap than an unstrained Si_rGe_{1-r} alloy.²⁷

In this study, ARUPS was employed to investigate the stability of hydrogen on strained $Si_xGe_{1-x}(100)2\times1$:H surfaces (x=0.40, 0.60, and 0.80). This technique allows the identification of the H-induced surface state/resonance on monohydride surfaces and the surface state associated with the dangling bond on clean surfaces. The hydrogen evolution from the strained $Si_xGe_{1-x}(100)2\times1$:H surfaces was also studied in comparison with $Si(100)2\times1$:H and $Ge(100)2\times1$:H surfaces. The Ge(100) surface was also prepared by MBE growth on a Si substrate, and this film was relaxed by the formation of misfit dislocations at the interface.

II. EXPERIMENTAL PROCEDURES

The experiments presented in this article were carried out in an integrated film growth/characterization system which is a combination of several ultra-high vacuum (UHV) chambers connected by a UHV transfer system. The details

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of the system are described elsewhere.^{28,29} The Si_xGe_{1-x} film growth, H-plasma exposure, and the surface measurements were performed in UHV compatible processing systems without exposure to ambient.

The substrates used in this study were 25 mm diam *n*-type Si(100) wafers doped with phosphorous with a resistivity of 0.8–1.2 Ω cm. The *ex situ* surface cleaning included UV/ozone exposure for 5 min followed by an HF based spin etch (HF:H₂O:ethanol=1:1:10). The UV/ozone exposure is known to remove hydrocarbons^{4,30-32} and the HF based spin etch removes the oxide leaving a hydrogen passivated surface.^{4,33,34} Following the *ex situ* chemical cleaning the wafer is mounted with tantalum wire on a molybdenum sample holder. The wafer is then introduced into UHV through a loadlock and transferred to the UHV MBE chamber which has a base pressure of $<1 \times 10^{-10}$ Torr. Following an in situ thermal desorption at 850 °C for 10 min in the UHV MBE chamber, the substrate temperature is reduced to 550 °C for all depositions. A 200 Å homoepitaxial silicon buffer layer is deposited on the clean Si(100) substrate to further ensure a contamination free interface. A series of strained $Si_x Ge_{1-x}$ alloys were epitaxially grown on the silicon buffer layers using electron beam evaporation in the UHV MBE chamber. A 300 Å Si or Ge layer was deposited for the pure Si and the pure Ge samples. To prepare strained Si_{0.8}Ge_{0.2}, Si_{0.6}Ge_{0.4}, and Si_{0.4}Ge_{0.6} alloy films, 200, 120, and 40 Å were deposited, respectively. To ensure a uniformly strained $Si_x Ge_{1-x}$ epilayer, the thicknesses of the strained Si_xGe_{1-x} alloys are less than the respective critical thicknesses.³⁵ Auger electron spectroscopy (AES) was also performed to verify relative surface atomic composition of the $Si_x Ge_{1-x}$ alloy samples. We note that the pure Ge film thickness is far above the critical thickness for strain relaxation, and this film is then relaxed by the formation of misfit dislocations at the interface.

To obtain H-terminated surfaces, the samples were transferred to the plasma chamber. The base pressure in the H-plasma chamber was $<2\times10^{-9}$ Torr. The samples were located 40 cm downstream from the center of the plasma tube. The H-plasma was generated by exciting the hydrogen gas in a quartz tube with 13.56 MHz rf radiation. The process pressure was controlled with a throttle valve and a turbo pump, and the flow rate of hydrogen gas was controlled with a mass flow controller. The samples were exposed to the H-plasma under the following conditions: process pressure=15 mTorr, rf power=20 W, flow rate of H₂ gas=80 sccm, and exposure time=1 min. To obtain a hydrogen terminated, 2×1 reconstructed surface the sample temperature was maintained at 400 °C for pure Si and Si_{0.8}Ge_{0.2}, while 180 °C was used for Si_{0.6}Ge_{0.4}, Si_{0.4}Ge_{0.6}, and pure Ge. For the higher Ge composition sample (Ge content $\geq 40\%$), no features due to hydrogen bonding were identified from the alloy surfaces for hydrogen plasma exposure at a substrate temperature of 400 °C.8

The low energy electron diffraction (LEED) patterns of the H-terminated samples showed a diffuse double domain 2×1 pattern. The diffuse spots indicate small domains on the surfaces.³⁶ In contrast, a sharp double domain 2×1 reconstructed LEED pattern was observed after the final step of *in* *situ* annealing for the H-terminated samples. AES data indicated no evidence of oxygen and carbon prior to or after ARUPS scans.

ARUPS was performed to examine the stability of the hydrogen passivated $Si_xGe_{1-x}(100)2\times1$ surfaces and to observe the surface states of the clean and H-terminated surfaces. The base pressure of the ARUPS chamber was $<2\times10^{-10}$ Torr with an operating pressure of $<1\times10^{-9}$ Torr. The sample was mounted on a heating stage which allows a sample temperature of up to ~1000 °C. The annealing process involves 50 and 500 °C/min ramp up and down rates, and the sample was held at the designated temperature for ~1 min.

The ARUPS spectra were obtained with a differentially pumped Ne discharge lamp which generated NeI (16.85 eV) radiation. The uv light is incident on the sample at $\sim 45^{\circ}$ from the surface normal in the analyzer rotation plane and at $\sim 15^{\circ}$ from the surface in the plane perpendicular to the analyzer rotation plane. The photoemitted electrons were analyzed with a 50 mm mean radius hemispherical analyzer (VSW HA 50) with an energy resolution of 0.25 eV and an angular resolution of 2°. The analyzer is mounted on a two stage goniometer which allows angle dependent measurement and can be rotated in the plane which is perpendicular to the surface of the sample. All ARUPS experiments presented in this article were performed at an emission angle of 15° along the [010] crystal direction. We note that the [010] direction was chosen in order to eliminate the ambiguity due to the superposition of the two surface Brillouin zones. The selection of angle for comparison in the spectra was chosen to emphasize features associated with the H-induced states and the surface states of Si, (relaxed) Ge, or strained Si_xGe_{1-x} alloys. ARUPS spectra of the series of H-terminated Si_rGe_{1-r} alloys were obtained as a function of annealing temperature. Each spectrum was acquired using a 0.005 eV step size and an integration time of ~ 1 s at each energy. To improve the signal-to-noise ratio, each sample was scanned five times and the five spectra were summed. After summing, the spectra were subjected to a five point smooth to further distinguish the data from the random noise. The position of the Fermi level was determined by measuring either a spectrum of a thick metal layer on the semiconductor or a spectrum of the metallic (Mo) sample holder. Both techniques yielded the same values.

III. RESULTS

LEED measurements were used to characterize the clean and H-terminated surfaces of the strained Si_xGe_{1-x} alloys. The LEED patterns of the H-terminated and clean surfaces are shown Fig. 1. Both LEED patterns indicate double domain 2×1 reconstructed surfaces. The diffraction pattern of the monohydride surface (before *in situ* annealing) is somewhat diffuse when compared to the 2×1 LEED pattern obtained from a clean surface with no bonded H (after *in situ* annealing). For the clean (100) surface, the direction of the dimer orientation is rotated by 90% between two terraces separated by a step of a monolayer height. This results in a double domain 2×1 LEED pattern. Hence, the sharp 2×1 [Fig. 1(a)] LEED pattern indicates well ordered dimer for-





(b)



Energy relative to the Fermi level (eV)

FIG. 1. The LEED patterns of the clean and the H-terminated $(100)2\times 1$ surfaces: (a) the sharp 2×1 pattern observed after the *in situ* annealing process, and (b) the diffuse 2×1 pattern with streaks observed after the H-plasma treatment.

FIG. 2. The ARUPS spectra of the H-terminated $Si(100)2\times 1$ surface obtained at sequential annealing steps. The surface state due to the dangling bond and the H-induced surface state/resonance are labeled *S* and *M*, respectively.

mation on relatively large terraces. However, after H-plasma exposure the diffuse spots and streaks in the LEED pattern [Fig. 1(b)] indicate small domains on the surface³⁶ or an incoherence of the dimer domains.³⁷ Since the sharp double domain 2×1 pattern is obtained after a low temperature (≤ 450 °C for alloys and ≤ 550 °C for pure Si) anneal to desorb the H, it is suggested that the diffuse peaks in the H-terminated surface indicate disorder or incoherence of the H-termination rather than a change of the terrace structure.

Figures 2 and 3 show the ARUPS spectra of Si(100)2 \times 1:H and (relaxed) Ge(100)2 \times 1:H surfaces obtained at sequential steps of the annealing process. While both desorption and diffusion processes will assist in the desorption, the short annealing times employed in these experiments were chosen to emphasize the thermally activated desorption rather than surface diffusion processes. From the monohydride Si(100) and (relaxed) Ge(100) surfaces, the H-induced surface state/resonance was identified and a reduction in the amplitudes of the H-induced peak was observed as the annealing temperature increased. During the annealing process, the hydride formed on Si(100) and Ge(100) surfaces begins to dissociate at annealing temperatures of ~ 450 and \sim 200 °C, respectively, and the surface state attributed to the dangling bond becomes evident after annealing at 450 and 250 °C, respectively. These results are consistent with the previously reported data that the Si–H monohydride begins to desorb at an annealing temperature of \sim 460 °C,^{6,7} while the Ge–H monohydride begins to desorb at an annealing temperature of \sim 190 °C.⁸

Figures 4, 5, and 6 show the ARUPS spectra of the strained $Si_xGe_{1-x}(100)2\times1$:H surfaces obtained as a function of annealing temperature. The H-induced surface state/ resonance labeled M was observed from all 2×1 reconstructed monohydride $Si_xGe_{1-x}(100)$ surfaces. The surface state associated with the dangling bond, which was not evident after hydrogen passivation, exhibited its full value after annealing at 400, 350, and 300 °C for the strained $Si_{0.8}Ge_{0.2}$, $Si_{0.6}Ge_{0.4}$, and $Si_{0.4}Ge_{0.6}$ (100) surfaces, respectively. It was also found that the feature associated with Si(Ge)-H bonding was not evident after the surface was annealed at ~450, ~350, and ~300 °C for the strained $Si_{0.8}Ge_{0.2}$, $Si_{0.6}Ge_{0.4}$, and $Si_{0.4}Ge_{0.6}$ (100)2×1:H surfaces, respectively.

The normalized peak intensities versus annealing temperature for the surface state associated with the dangling bond and the H-induced surface state/resonance are shown in Figs. 7 and 8, respectively. The peak intensities were measured from the difference spectrum obtained by directly subtracting the spectrum of either the clean surface or the H-terminated surface from the relevant UPS spectra. The peak intensities were normalized with respect to the clean





FIG. 3. The ARUPS spectra of the H-terminated (relaxed) $Ge(100)2\times 1$ surface obtained at sequential annealing steps. The surface state due to the dangling bond and the H-induced surface state/resonance are labeled *S* and *M*, respectively.

surface state peak height after *in situ* annealing process or to the H-induced peak height after *in situ* hydrogen exposure.

IV. DISCUSSION

The ARUPS spectra revealed that the desorption of hydrogen is affected by the presence of Ge on the surface. Figures 7 and 8 show almost an inverse dependence for the presence of the dangling bond states and the hydrogen induced surface state/resonance versus annealing temperature.

Consider first the electronic states of the H-terminated surface. In Figs. 2 and 3, the H-induced peak related to the Si–H and Ge–H bonds appears at -5.76 and -5.35 eV relative to the Fermi level, respectively. For all of the alloy samples, a single peak was observed at an energy between -5.50 and -5.60 eV relative to the Fermi level. The observation of a single peak suggests that the neighboring sites are coupled into a well defined electronic band rather than separate Si–H and Ge–H molecular states. The different electronic state energies of the Si–H and Ge–H peaks also reflect the fact that the respective bonding energy of the chemisorbed Si–H is larger than that of the chemisorbed Ge–H. The Ge–H bond is about 15% weaker than the Si–H bond (Ge–H=80 kcal/mol, Si–H=94 kcal/mol).³⁸

We consider next the initial desorption from the H-terminated surfaces. Figure 7 shows that, for the series of

FIG. 4. The ARUPS spectra of the H-terminated strained $Si_{0.8}Ge_{0.2}(100)2 \times 1$ surface obtained at sequential annealing steps. The surface state due to the dangling bond and the H-induced surface state/ resonance are labeled *S* and *M*, respectively.

the strained Si_xGe_{1-x} alloy samples, the surface state related to the dangling bond can be identified after the surfaces are annealed at 250 °C. At this annealing temperature, the surface state due to the dangling bond of the Ge(100) surface can also be observed, while that of the Si(100) surface is not visible until the surface is annealed at 450 °C. Figure 8 also shows that the hydrogen starts to desorb from the Si(100)surface at an annealing temperature of ~450 °C, while, for the Ge and alloy samples, the H dissociation is initiated at temperatures ≤ 250 °C. This is consistent with the work of Greenlief et al.³⁹ who suggested that the preadsorption of Ge strongly alters the desorption of H_2 , shifting most of the desorption into a lower temperature state most strongly influenced by Ge. Hence, it can be suggested that the hydrogen preferentially starts to desorb from Ge sites on the $Si_rGe_{1-r}(100)2 \times 1$:H surfaces.

We consider now the role of surface diffusion. If there is no H diffusion on the surface, then the hydrogen desorption from the Si_xGe_{1-x} alloy surface would be a combination of the hydrogen desorption from pure Si and Ge surfaces. Then the H-induced peak for the Si_xGe_{1-x} alloys should decrease in the 150–250 °C and 400–550 °C ranges while remaining relatively unchanged in the 250–400 °C range. However, the results show that hydrogen desorption from the Si_xGe_{1-x} alloy surface does not consist of components that are associ-





FIG. 5. The ARUPS spectra of the H-terminated strained $Si_{0.6}Ge_{0.4}(100)2 \times 1$ surface obtained at sequential annealing steps. The surface state due to the dangling bond and the H-induced surface state/ resonance are labeled *S* and *M*, respectively.

ated with hydrogen desorption from pure Si and pure Ge surfaces.

For the series of the monohydride $Si_x Ge_{1-x}(100)2 \times 1$ surfaces, it can be assumed that there are a mixture of Si-H bonds and Ge-H bonds on the surfaces. For the Si_{0.4}Ge_{0.6}, $Si_{0.6}Ge_{0.4}$, and $Si_{0.8}Ge_{0.2}$ (100)2×1:H surfaces, the peak associated with the H-induced surface state/resonance was not detectable after annealing at 300, 350, and 450 °C, respectively, while the peak height of the H-induced surface state/ resonance for Si(100)2 \times 1:H surface remains unchanged up to an annealing temperature of 400 °C. It should be also noted that the peaks attributed to the dangling bond surface states for the $Si_{0.4}Ge_{0.6}$, $Si_{0.6}Ge_{0.4}$, and $Si_{0.8}Ge_{0.2}$ (100)2×1 surfaces exhibit their full heights after annealing at 300, 350, and 400 °C, respectively, which implies an essentially hydrogen free surface. However, there is no evidence of the surface state due to the dangling bonds for the Si(100) surface until this surface was annealed at 450 °C.

This is consistent with the report of Sharp *et al.*⁴⁰ who studied the kinetics of hydrogen desorption in surfacelimited thin-film growth of Si_xGe_{1-x} alloys. They found that the hydrogen desorption process from the Si_xGe_{1-x} alloy surface consists of two components, i.e., fast and slow components, and that the activation energies of the two components are not identical to the hydrogen desorption energy from pure silicon or that from pure germanium surfaces.

FIG. 6. The ARUPS spectra of the H-terminated strained $Si_{0.4}Ge_{0.6}(100)2 \times 1$ surface obtained at sequential annealing steps. The surface state due to the dangling bond and the H-induced surface state/resonance are labeled *S* and *M*, respectively.

In one picture for the H_2 recombinative desorption process, H atoms approach each other through a thermal diffusion mechanism.^{17,41–43} An indication of hydrogen diffusion on the surface can be found from several published



FIG. 7. The surface state peak intensity associated with the dangling bond as a function of annealing temperature. The peak intensities have been normalized to the value obtained from the clean surface.



FIG. 8. The H-induced peak intensity as a function of annealing temperature. The peak intensities have been normalized to the value obtained from the initial H-terminated surface.

results.^{16,44–46} Recently, Vittadini *et al.* presented local density approximation (LDA) calculations of the potential energy surface for the diffusion of a single hydrogen atom on Si(100)2×1.⁴⁷ Their results indicate that H should move along a surface dimer row and that diffusion can be considered a fast process compared to H₂ recombinative desorption. Therefore, it is plausible to assume that the hydrogen desorption from the strained Si_xGe_{1-x}(100)2×1:H surfaces is related to the diffusion of hydrogen on the surface. It was suggested above that the desorption of hydrogen occurs from the Ge sites. As the annealing temperature increases, the hydrogen can diffuse more readily along the surface, and as the hydrogen diffuses to a Ge site the desorption can occur.

We note from Figs. 7 and 8 that the H concentration versus temperature for the alloy surfaces appears to exhibit two components. This effect is represented as shoulders in the curves (Figs. 7 and 8) for the $Si_{0.6}Ge_{0.4}$ and $Si_{0.8}Ge_{0.2}$ alloy samples. As noted above, if the H desorption involves Si sites, it would be expected that the H concentration would remain constant in the 250–400 °C temperature range. We suggest then that the diffusion of H on the surface is likely to be responsible for the higher temperature shoulder in the measurements. This analysis suggests that the H desorption is essentially totally mitigated by the Ge on the surface and the surface diffusion of the H. There is then little, if any, desorption from the Si sites.

We should note that additional research is necessary to explore the details of the actual desorption process. We may expect that the Si_xGe_{1-x} alloy surface will include a random distribution of Si and Ge at the different sites. This will then include Si–Si, Si–Ge, and Ge–Ge dimers, and the efficacy of the different dimer structures may actually determine the evolution of the surfaces.

V. CONCLUSIONS

The hydrogen evolution from a series of strained $Si_xGe_{1-x}(100)2 \times 1$:H surfaces was studied using the

ARUPS technique. To avoid strain relaxation, the H adsorption and desorption were completed at temperatures less than the growth temperature of the strained $Si_rGe_{1-r}(100)$ films. The surfaces exhibited a sharp double domain 2×1 reconstruction for the series of clean Si_rGe_{1-r} alloys and a diffuse double domain 2×1 reconstruction after hydrogen passivation of the surface. The diffuse diffraction was attributed to disorder or a loss of coherence of the H bonding rather than changes of the terrace structure. The ARUPS spectra of H-terminated Si_rGe_{1-r} alloy surfaces showed a single hydrogen-induced surface state/resonance corresponding to the Si(Ge)-H bond. After in situ annealing, the surface state due to the dangling bond could be identified from ARUPS spectra of all alloys. The experimental results indicate that the hydrogen desorption is initiated from Ge sites and proceeds via a combination of rapid surface diffusion and desorption from the Ge sites. It was suggested that the Si sites may not directly participate in the desorption process, but further studies are necessary to determine the roles of specific dimer structures.

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