

Low-temperature silicon homoepitaxy by ultrahigh vacuum electron cyclotron resonance chemical vapor deposition

Heung-Sik Tae,^{a)} Seok-Hee Hwang,^{a)} Sang-June Park,^{b)} Euijoon Yoon,^{b)} and Ki-Woong Whang^{a)}

Inter-university Semiconductor Research Center (ISRC), Seoul National University, Seoul 151-742, Korea

(Received 3 August 1993; accepted for publication 2 December 1993)

High quality silicon homoepitaxial layers are successfully grown at 560 °C by ultrahigh vacuum electron cyclotron resonance chemical vapor deposition (UHV-ECRCVD) using a SiH₄/H₂ plasma. The effects of substrate dc bias on the *in situ* hydrogen plasma clean and the subsequent silicon epitaxial growth are examined by the reflection high-energy electron diffraction (RHEED), secondary ion mass spectroscopy (SIMS), and cross-section transmission electron microscopy (XTEM). It is observed that the substrate dc bias plays a significant role in obtaining a damage-free, clean Si substrate prior to epitaxial growth. Severe damage in the Si surface is observed by XTEM, though RHEED shows a streaky pattern, when the substrate is electrically floating, but the damage can be suppressed with +10 V dc bias to the substrate. Substrate dc bias during plasma deposition drastically changes the crystal structure from polycrystalline at -50 V to high quality epitaxial silicon at substrate biases greater than +50 V. Precise control of the ion energy during *in situ* cleaning and plasma deposition is very important in low-temperature Si epitaxy by UHV-ECRCVD and it is possible by proper control of the substrate dc bias.

Low-temperature Si epitaxy has become one of the most essential processes for the successful realization of Si-based heterostructure devices. It depends heavily on the ultrahigh vacuum environment,¹ the cleanliness of the epilayer/substrate interface, and tight optimization of process parameters.² Epitaxy temperature can be further lowered by providing extra energy from other than thermal energy. Plasmas are one of the most frequently used means of adding extra energy for low temperature epitaxy, and even room-temperature Si epitaxy without substrate heating was reported.³ The use of plasma increases the growth rate at low temperatures, and generates reactive ions and neutrals for efficient *in situ* cleaning of substrates prior to epitaxy. The combination of a ultrahigh vacuum chamber and a plasma source looks very promising. Care, however, must be taken to avoid damage to the substrate during *in situ* cleaning and epitaxial layers during deposition, since ion energies often exceed the binding energy of the substrate constituents, and extended defects may result in the substrate and in the epitaxial layers.

The most frequently used plasmas for low-temperature Si epitaxy in ultrahigh vacuum systems are electron cyclotron resonance (ECR)³⁻⁵ and radio frequency (rf) plasmas.^{6,7} ECR plasmas are believed to be more promising than rf plasmas, since the operating pressure and the plasma potential are lower in ECR, and the chances of obtaining purer and less damaged Si epitaxial layers at low temperatures are higher for the ECR plasma enhanced process. Remote plasma chemical vapor deposition (RPCVD)^{6,7} utilizes an rf plasma, and confines the intense glow away from the substrate to minimize the ion damage due to higher plasma potential of the rf plasma. No substrate bias is applied in

RPCVD. ECR plasma enhanced CVD processes operate normally at the pressure ranges from 10⁻⁴ to 10⁻³ Torr, where the diverging downstream glows are in touch with the substrate, but the energy of the impinging ions across the plasma/substrate boundary may be high enough to put permanent damage to the substrate as well as the epilayers.

The effect of the substrate bias is often neglected and the provision for it is missing, assuming that the plasma potential is small in the ECR plasma, and usually other process parameters such as plasma power, substrate temperature, flow rates, etc., are optimized for good epitaxy.^{3,5} A positive bias to the substrate is expected to reduce the energy of the energetic ions from the plasma.⁸ However, to obtain Si homoepitaxy at low temperatures, there is a contradictory report indicating that the optimum ion energy for Si homoepitaxy at 400 °C is 250 eV by applying a *negative* bias to the substrate.⁴ Our experimental result shows that such ion energy is too high to get epitaxy at 560 °C, where the dynamic defect annihilation is even faster than 400 °C. In our work, homoepitaxial growth of silicon is investigated using a SiH₄/H₂ plasma by ultrahigh vacuum electron cyclotron resonance chemical vapor deposition (UHV-ECRCVD) at 560 °C. The effects of the substrate dc bias are examined by the reflection high-energy electron diffraction (RHEED), secondary ion mass spectroscopy (SIMS), and cross-section transmission electron microscopy (XTEM). We found that the dc substrate bias in the ECR plasma plays a significant role in both *in situ* hydrogen plasma cleaning and low temperature Si epitaxy at temperatures below 560 °C by plasma deposition of SiH₄/H₂. In both cases, positive 10 V, 50 V substrate biases are necessary for damage-free *in situ* hydrogen plasma cleaning and defect-free homoepitaxial growth at 560 °C, respectively.

Figure 1 shows the schematic diagram of the UHV-ECRCVD system employed in this research. The main growth chamber consists of a turbomolecular pump and an

^{a)}Also with the Department of Electrical Engineering.

^{b)}Also with the Department of Inorganic Materials Engineering.

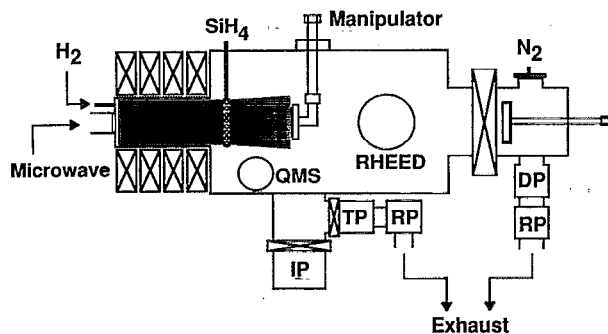


FIG. 1. Schematic diagram of the UHV-ECRCVD system.

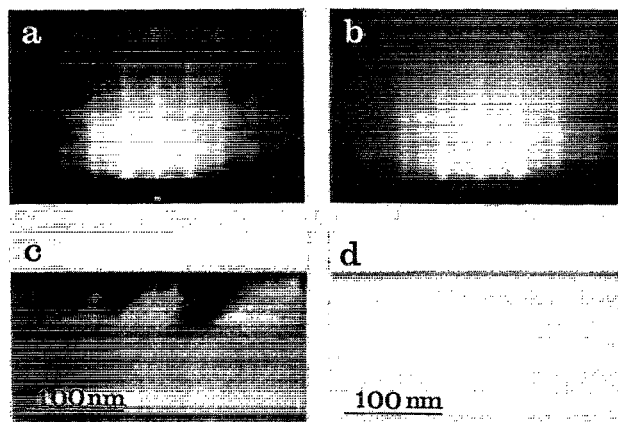


FIG. 2. The effect of the substrate dc bias on *in situ* hydrogen plasma cleaning. Microwave power 100 W, magnet current 50 A, H₂ flow rate 74 sccm, 2 min plasma exposure. (a) and (b) are RHEED patterns for floating and +10 V dc bias conditions, respectively. (c) and (d) are corresponding XTEM photographs. Severe ion damage is clearly shown in (c) even after 2 min hydrogen plasma exposure.

ion pump, and its base pressure is 1×10^{-9} Torr. It has a quadruple mass spectrometer and a RHEED to study the surface before and after the epitaxial growth. The substrate can be heated to 900 °C and the dc bias can be applied to control the ion energy impinging on the substrate. H₂ (99.9999%) is introduced to the ECR cavity and SiH₄ (99.999%) is injected into the growth chamber through a gas dispersal ring located between the ECR cavity and the substrate. The location of the ECR layer is adjustable by changing the current input to the four magnets around the ECR cavity. The surface temperature of the Si wafer is calibrated using infrared laser interferometric thermometry⁹ and a pyrometer.

The substrates used for the study are 4-in.-diam, *n*-type (100) silicon wafer (resistivity, 10–20 Ω cm). Wafers are dipped into a 4:1 H₂SO₄:H₂O solution for 6 min and rinsed with de-ionized water for 6 min, and subsequently dipped into a 10:1 HF solution for 30 s for the complete removal of surface oxide and hydrogen termination of silicon surface.¹⁰ The wafers are loaded into the growth chamber of the UHV-ECRCVD system through a nitrogen-purged load-lock chamber immediately after the final HF dip.

It is important to obtain a clean, defect-free silicon substrate surface prior to epitaxial growth,^{2,6} since contaminants and defects on the starting substrates may result in the epitaxial films with high defect density. In our work, the effects of the substrate dc bias on the *in situ* hydrogen plasma cleaning are examined by RHEED, SIMS, and XTEM. The results of RHEED and XTEM, after the 2-min *in situ* H₂ plasma clean at 560 °C on the Si substrates are shown in Fig. 2. In UHV-ECRCVD, the ECR plasma produces highly reactive hydrogen atoms and ions. As shown in the RHEED patterns of Figs. 2(a) and 2(b), both 2-min hydrogen cleans with a floated substrate and a +10 V biased substrate at 560 °C, with microwave power of 100 W, transformed the 1×1 Si surface (not shown) into a 2×1 surface. However, it is observed that even the 2-min hydrogen plasma cleans at 560 °C with a floated substrate results in the damage in the Si substrate, as shown in the XTEM image of Fig. 2(c). At ground potential the similar damage is observed in the substrate. The damage production can be suppressed by applying biases greater than +10 V to the substrate and the damage-free Si substrate is shown in Fig. 2(d).

Figure 3 shows the SIMS profile for the epilayer grown for 285 min at 560 °C after a damage-free *in situ* H₂ plasma clean at 560 °C, +10 V bias condition. The SIMS analysis

shows no pile up of oxygen and carbon at the interface as indicated by an arrow. Similarly, no oxygen and carbon pileup is detected by SIMS in the plasma cleaned samples at floating condition. Figures 2 and 3 clearly demonstrate the fact that the hydrogen plasma cleaning is very effective in removing the surface contaminants, however, care should be taken not to damage the substrate. We have obtained highly defective epilayers on the damaged substrates.

Figure 4 shows the RHEED patterns for the Si epilayers grown for 30 min at various dc bias conditions at the microwave power of 50 W, substrate temperature 560 °C, SiH₄ flow rate of 2.5 sccm, H₂ flow-rate of 100 sccm, after a damage-free hydrogen plasma cleaning for two minutes at +10 V dc bias. The RHEED pattern of Fig. 4(a) shows a ring

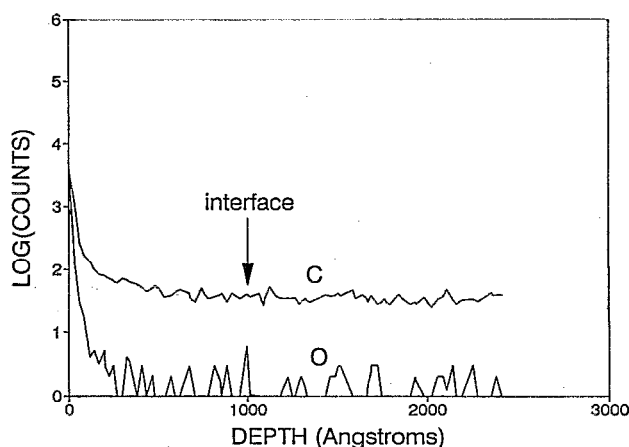


FIG. 3. SIMS profile for the epilayer grown for 285 min after *in situ* hydrogen plasma cleaning shown in Fig. 2(b). Epilayer/substrate interface is indicated by an arrow. Growth conditions are substrate temperature 560 °C, microwave power 50 W, magnet current 40 A, H₂ flow rate 100 sccm, SiH₄ flow rate 2.5 sccm, total pressure 1.5×10^{-3} Torr, deposition time 285 min.

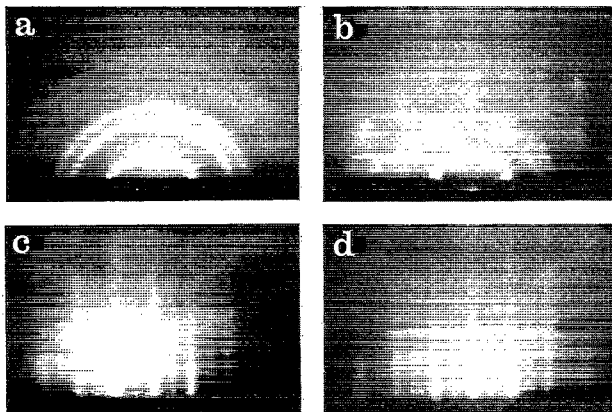


FIG. 4. Changes in RHEED patterns of Si epilayers grown at various substrate dc bias. (a) -50 V, (b) floating, (c) $+10$ V, (d) $+50$ V. Other deposition conditions are substrate temperature 560 °C, microwave power 50 W, magnet current 40 A, H_2 flow rate 100 sccm, SiH_4 flow rate 2.5 sccm, total pressure 1.5×10^{-3} Torr, deposition time 30 min.

pattern indicating that the Si layer grown at -50 V dc bias is a polycrystalline silicon. The RHEED pattern of Fig. 4(b) for the Si layer grown at floating condition shows a diffraction pattern of a single crystal in which each spot is split. It is presumed that the splitting of each spot is caused by the double diffraction from the high density of twins in the epilayer.¹¹ On the other hand, the RHEED patterns of Figs. 4(c) and 4(d) show the clear half-order streaks, indicating that the Si layers grown at the $+10$ V, and $+50$ V dc bias are high quality, single crystalline silicon epilayers with smooth surfaces. The improvement in crystallinity with the positive substrate dc bias implies that even the plasma potential in the ECR plasma (10 – 20 eV), often regarded small, is high enough to damage the Si single crystal, and consequently the ion energy should be reduced below a certain level (~ 10 eV) in low-temperature Si epitaxy by UHV-ECRCVD. Details of

the low temperature Si epitaxial growth and the effects of various process parameters at substrate temperatures at and below 560 °C will be reported elsewhere.

In summary, Si epilayers are successfully grown by UHV-ECRCVD at 560 °C with a SiH_4/H_2 plasma on the Si substrates plasma-cleaned *in situ*. It is observed that the substrate dc bias during *in situ* H_2 plasma cleaning plays a significant role in obtaining a damage-free Si substrate prior to epitaxial growth. A 2-min hydrogen plasma cleaning with a floated substrate at 100 W, 560 °C damages the Si substrate, as determined by XTEM, though the RHEED for the surface shows a well-defined streaky 2×1 reconstruction. Damage production is suppressed by applying dc biases greater than $+10$ V to the substrate. Positive substrate dc bias (>50 V) should also be applied to suppress the energetic ion bombardment towards the growing epilayers, in order to get high quality Si epilayers at 560 °C.

This work is sponsored by the Korea Telecommunication Research Center and by the SNU Development Fund under project number 92-15-2111.

- ¹ B. S. Meyerson, Appl. Phys. Lett. **48**, 797 (1986).
- ² T. J. Donahue and R. Reif, Appl. Phys. Lett. **57**, 2757 (1985).
- ³ K. Fukuda, J. Murota, S. Ono, T. Matsuura, H. Uetake, and T. Ohmi, Appl. Phys. Lett. **59**, 2853 (1991).
- ⁴ H. Yamada and Y. Torii, J. Appl. Phys. **64**, 702 (1988).
- ⁵ D. S. L. Mui, S. F. Fang, and H. Morkoç, Appl. Phys. Lett. **59**, 1887 (1991).
- ⁶ L. Breaux, B. Anthony, T. Hsu, S. Banerjee, and A. Tasch, Appl. Phys. Lett. **55**, 1885 (1989).
- ⁷ S. V. Hattangady, J. B. Postill, G. G. Fountain, R. A. Rudder, M. J. Mantini, and R. J. Markunas, Appl. Phys. Lett. **59**, 339 (1991).
- ⁸ J. Ramm, E. Beck, and A. Zuger, Mater. Res. Soc. Symp. Proc. **220**, 15 (1991).
- ⁹ V. M. Donnelly and J. A. Macaulley, J. Vac. Sci. Technol. A **8**, 84 (1990).
- ¹⁰ D. B. Fenner, D. K. Biegelsen, and R. D. Bringans, J. Appl. Phys. **66**, 419 (1989).
- ¹¹ D. W. Pashley and M. J. Stowell, Philos. Mag. **8**, 1605 (1963).