Noncontact minority carrier lifetime measurement of Si and SiGe epilayers prepared by ultrahigh vacuum electron cyclotron resonance chemical vapor deposition

Seok-Hee Hwang, Yun-Phil Eo,[a] Jung-Hyun Seo, and Ki-Woong Whang
School of Electrical Engineering & Inter-university Semiconductor Research Center (ISRC), Seoul National University, Seoul 151-742, Korea

Euijoon Yoon
School of Materials Science and Engineering and ISRC, Seoul National University, Seoul 151-742, Korea

Heung-Sik Tae
School of Electronic and Electrical Engineering, Kyungpook National University, Taegu 702-701, Korea

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A 70 MHz inductively coupled rf bridge probe is used to measure the minority carrier lifetime of the Si and SiGe epilayers grown by ultrahigh vacuum electron cyclotron resonance chemical vapor deposition (UHV-ECRCVD) at temperatures below 560 °C. Proper surface treatments before HF immersion are required for the accurate measurement of the bulk minority carrier lifetime. The effects of the process parameters such as the substrate dc bias, the distance of the ECR layer from the substrate, and the substrate temperature, including *in situ* surface cleaning, on the minority carrier lifetime of the Si and SiGe epilayers are examined by the rf bridge probe. It is confirmed that the rf bridge probe can monitor the epitaxial quality of low temperature Si and SiGe epilayers, making it an indispensable tool for the high quality device fabrication with Si and SiGe epitaxial layers grown by low temperature UHV-ECRCVD.

I. INTRODUCTION

A long minority carrier lifetime of epilayers is desirable for the efficient operation of semiconductor devices. The minority carrier lifetime depends largely on both the foreign impurities and crystalline defects that can act as recombination centers. While the crystalline defects can be detected by transmission electron microscopy (TEM), some nondestructive methods directly measuring minority carrier lifetimes are desirable to determine the electrical quality of the epilayers.

We have reported that dislocation-free Si and SiGe epilayers were successfully grown at temperatures below 560 °C by ultrahigh vacuum electron cyclotron resonance chemical vapor deposition (UHV-ECRCVD).[1–4] Using plan-view, cross-section, and high-resolution TEM as well as large-area optical microscopy, it was confirmed that Si and SiGe epilayers were dislocation free. The fabrication of a SiGe heterojunction bipolar transistor for high speed devices is currently underway using the Si and SiGe epilayers grown by this system. For the device fabrication, fast, reliable, and nondestructive process monitoring techniques are greatly needed. For this purpose, an inductively coupled rf bridge probe[5] was made and the minority carrier lifetimes of the Si and SiGe epilayers were measured.

The effects of the various chemical treatments of the substrate surface on the bulk minority carrier lifetime were examined. The effects of hydrogen plasma cleaning before epitaxial growth on the minority carrier lifetime of the Si and SiGe epilayers were also examined. In particular, the changes in the minority carrier lifetimes of the Si and SiGe epilayers grown with various process parameters such as the substrate dc bias, the distance of the ECR layer from the substrate, and substrate temperature were carefully examined by the inductively coupled rf bridge probe.

II. EXPERIMENT

The schematic diagram of the 70 MHz noncontact inductively coupled rf bridge probe developed in this work is shown in Fig. 1. The samples immersed in a HF bath are placed near the rf coil, and illuminated with light pulses from a strobe lamp (beam intensity of 11×10^6 cd/m^2, pulse duration $\sim 3\ \mu$s). Decay of the injected photocarriers is monitored by the reflected signal from the rf coil after the photocarrier injection. The reflected signal is proportional to the degree of impedance imbalance in the resonance circuit, and it scales with the photocarrier concentration in the sample. The reflected signal is amplified and monitored as a voltage output by a digital oscilloscope. The transient wave form is obtained by averaging 20 cycles to reduce the noise of the rf bridge probe. An exponential decay is assumed for the decay of the photocarrier to extract the lifetimes. The Si and SiGe epilayers were grown on N-type phosphorus-doped (100) Czochralski-grown silicon wafers (10–20 Ω cm) at temperatures below 560 °C by UHV-ECRCVD. A detailed description of the low temperature Si and SiGe epitaxial growth by UHV-ECRCVD is reported elsewhere.[3,4]

Proper substrate surface pretreatment is very important for the accurate bulk minority carrier lifetime measurement. Table I shows the various pretreatment methods tested in this

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[a] Present address: Samsung Display Devices, Kiheung, Kyungki-do 449-900, Korea.

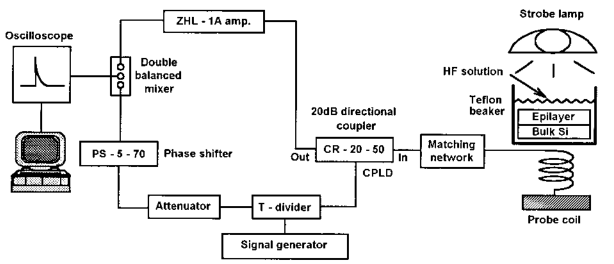

FIG. 1. A schematic diagram of a 70 MHz inductively coupled rf bridge probe.

work. For the bulk Si wafers a combination of degreasing with acetone and native oxide strip with 10% HF gives the longest lifetime compared with nonpretreatment. Weinberger *et al.* reported that KOH etch results in a poorly passivated surface.[6] However, our result shows that a modified KOH etch[7] (300 g KOH+300 ml propanol+12 g $K_2Cr_2O_7$+1200 ml H_2O) results in an improvement of the lifetime of Si epilayers. It is presumed that the slight removal of the contaminated surface suppresses the surface recombination. Another result shows that the 10:1 HF dip treatment before HF immersion affects measured lifetime values. It is thought that the removal of native oxide by a 10:1 HF dip enables a good surface passivation.[8]

All samples measured in this study have the same size of 25×25 mm^2. The pretreatment of SiGe epilayers is identical to those of bulk Si wafers. Figure 2 shows a lifetime decay curve for a typical Czochralski-grown bare silicon wafer. The minority carrier lifetime of the silicon wafer reached 550 μs, which compares well with the measurement by a similar rf bridge probe method.[9] To verify repeatability of the lifetime measurement by our system, the lifetime of a standard silicon wafer is always measured and compared with the previous values before the lifetime measurement of Si and SiGe epilayers.

III. RESULT AND DISCUSSION

The effects of *in situ* surface cleaning on the minority carrier lifetime of Si epilayers are examined by the rf bridge probe. It is well known that the quality of low temperature Si and SiGe epitaxial layers depends strongly on *in situ* surface cleaning before epitaxial growth.[8] We previously reported that defect-free Si epilayers are grown after ECR hydrogen plasma cleaning, otherwise defective epilayers are grown.[2] It is interesting to see if the rf bridge probe is sensitive enough to tell the differences in epilayer quality with different Si surface preparation. Figure 3 shows the changes in minority carrier lifetimes of the Si epilayers grown under the same growth condition except for the *in situ* cleaning step. Sample (a) is cleaned by a HF dip [with no-deionized (DI) water rinse] and is subsequently exposed to air for 30 min, then

TABLE I. The changes in minority carrier lifetimes of bulk Si wafers and Si epilayers with various chemical treatments.

No.	Sample	Step 1	Step 2	Lifetime (μs)
1	Si wafer	⋯	⋯	100
2	Si wafer	⋯	10% HF dip	190
3	Si wafer	Acetone	10% HF dip	550
4	Si wafer	4:1 H_2SO_4:H_2O_2	10% HF dip	510
5	Si epilayer	Acetone	10% HF dip	33
6	Si epilayer	Modified KOH etch+acetone	10% HF dip	66

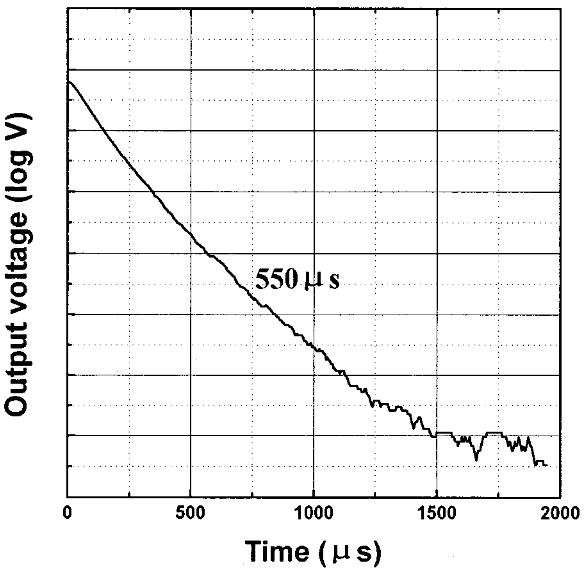

FIG. 2. The lifetime decay curve of a Czochralski-grown bare Si wafer.

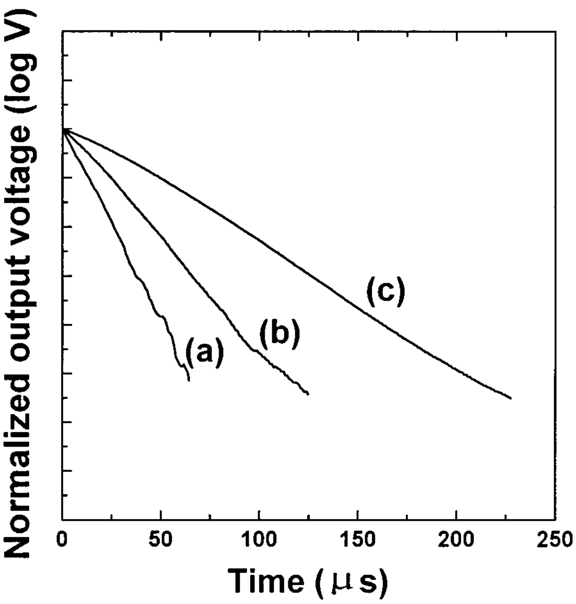

FIG. 3. The changes in minority carrier lifetime of the Si epitaxial layers grown under the same growth condition after three different Si surface cleaning treatments: (a) 30 min of air exposure after a HF dip, and no hydrogen plasma cleaning; (b) immediate loading after the HF dip, and no hydrogen plasma cleaning; (c) immediate loading after the HF dip, and *in situ* hydrogen plasma cleaning.

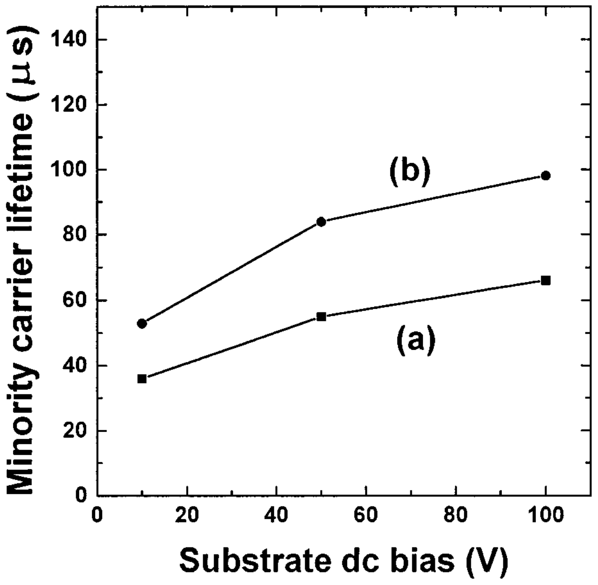

FIG. 4. The changes in minority carrier lifetimes of the Si and SiGe epilayers with substrate dc bias. (a) Si epilayer, (b) SiGe epilayer.

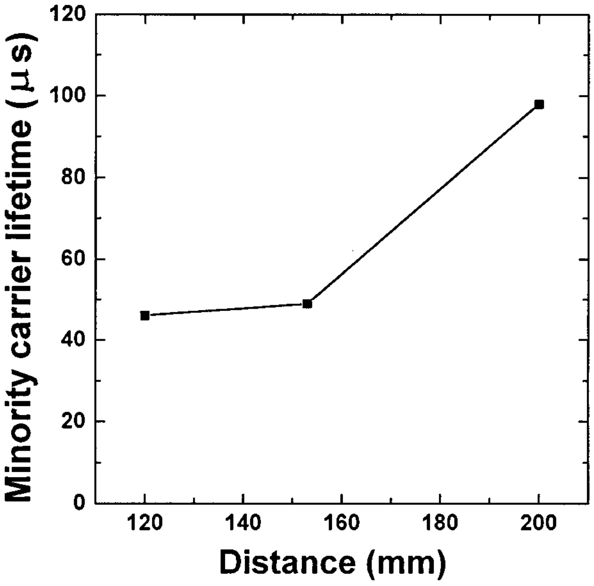

FIG. 5. The changes in minority carrier lifetimes of the SiGe epilayers with the distance of the ECR layer from the substrate.

loaded into the growth chamber. Sample (b) is cleaned by a HF dip (with no DI water rinse) and immediately loaded into the growth chamber. Sample (c) is cleaned by a HF dip and cleaned *in situ* by hydrogen plasma for 2 min. The measurement for each sample is repeated and the data are superimposed in Fig. 3. The lifetime of the epilayer grown after 30 min of air exposure ranges from 37 to 39 μs, and that of the fast-loaded sample after the HF dip ranges from 42 to 45 μs. On the other hand, the lifetime of the epilayer grown after *in situ* hydrogen plasma cleaning [sample (c)] ranges from 65 to 66 μs. Cleaning and hydrogen passivation by a HF dip has been widely used in the Si epitaxial growth. However, when the hydrogen passivation is not perfect, an appreciable concentration of impurities can be absorbed on the Si surface and they lead to defective epitaxial layers. This improvement in lifetimes confirms the effectiveness of *in situ* hydrogen plasma cleaning, which was shown by our previous study.[2]

The effects of the process parameters, such as the substrate dc bias, the distance of the ECR layer from the substrate, and substrate temperature, on the minority carrier lifetime of Si and SiGe epilayers are examined by the rf bridge probe. We have reported the effects of the process parameters on the low temperature Si epitaxy. In particular, the positive substrate dc bias was crucial in determining the crystallinity of the Si epilayers grown by UHV-ECRCVD.[3] Figure 4 shows the minority carrier lifetime of the Si and SiGe epilayers grown at different positive substrate dc biases. The deposition conditions for Si epilayers are as follows: substrate temperature—560 °C; microwave power—50 W; distance of the ECR layer from the substrate—200 mm; H_2 flow rate—100 sccm; SiH_4 flow rate—2.5 sccm; total pressure—3 mTorr. The deposition conditions for SiGe epilayers are as follow: substrate temperature—440 °C; microwave power—50 W; distance of the ECR layer from the substrate—200 mm; H_2 flow rate—100 sccm; SiH_4 flow rate—1 sccm; GeH_4 (5% in H_2) flow rate—5 sccm; total pressure—3.3 mTorr. All Si epilayers grown at positive dc biases greater than 10 V were dislocation free by using destructive techniques such as cross-section TEM, plan-view TEM, and high-resolution TEM.[3] But, as shown in Fig. 4, the lifetimes of Si and SiGe epilayers increase monotonically as the substrate dc bias changes from +10 to +100 V. This kind of improvement in crystalline quality cannot be distinguished by TEM analysis when the dislocation density in the epilayer falls below $10^5/cm^3$ or a small quantity of defects such as the interstitials and vacancies are present. This clearly shows that the rf bridge probe can monitor the epitaxial quality of low temperature Si epilayers that cannot be quantified by TEM analysis.

Changes in minority carrier lifetimes of the SiGe epilayers with the distance of the ECR layer from the substrate are shown in Fig. 5. The other deposition conditions are identical: microwave power—50 W; H_2 flow rate—100 sccm; SiH_4 flow rate—1 sccm; GeH_4 (5% in H_2) flow rate—5 sccm; substrate dc bias—100 V; and total pressure—3.3 mTorr. As shown in Fig. 5, the lifetimes of the SiGe epilayer increase monotonically as the ECR layer is moved away from the substrate. It was observed that *in situ* reflection high energy electron diffraction patterns for the SiGe epilayers change from one with spotty streaks to a 2×1 pattern with clear half-order streaks as the ECR layer is moved away from the substrate. The maximum plasma density occurs at the ECR layer of 875 G in the ECR plasma. Electrons and ions produced at the ECR layer move downstream toward the substrate with an ion energy of about kT_e. The energy and the flux of the reactant precursors, including an ion decrease from collisions and charge exchanges as the ECR layer is moved away from the substrate, result in the improved crystal quality of the SiGe epilayers.

Figure 6 shows the changes in minority carrier lifetimes

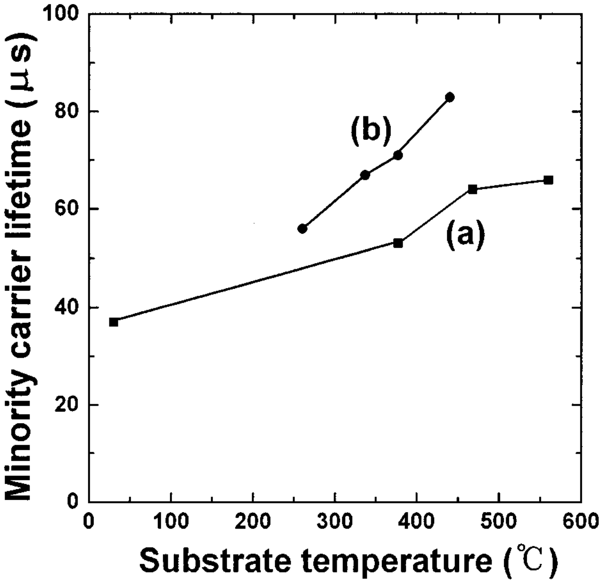

FIG. 6. The changes in minority carrier lifetimes of the Si and SiGe epilayers with substrate temperature. (a) Si epilayer, (b) SiGe epilayer.

of the Si and SiGe epilayers with substrate temperature. Deposition conditions for Si are as follows: microwave power—50 W; substrate dc bias—100 V; distance of the ECR layer from the substrate—200 mm; H_2 flow rate—100 sccm; SiH_4 flow rate—2.5 sccm; total pressure—3 mTorr. Deposition conditions for SiGe are as follows: microwave power—50 W; distance of the ECR layer from the substrate 200 mm; H_2 flow rate—100 sccm; SiH_4 flow rate—2 sccm; GeH_4 (5% in H_2) flow rate—10 sccm; total pressure—3.6 mTorr. As shown in Fig. 6, the lifetime of the Si and SiGe epilayers decreases as the substrate temperature decreases. By plan-view TEM and dilute Schimmel etch analysis, it was also observed that a high density of defects in Si and SiGe epilayers are produced as the substrate temperature decreases. This decrease in lifetimes may be due to the insufficient adatom migration to grow defect-free epilayers as the substrate temperature decreases.

The effects of *in situ* boron doping on the Si and SiGe epitaxial growth were examined by the lifetime measurement. It is observed that the lifetimes of *in situ* B-doped Si and SiGe epilayers, whose dopant concentrations are about 10^{17}–10^{19}/cm^3, are 30–40 μs, although the lifetimes of undoped Si and SiGe epilayers are longer than 50 μs. Kapoor *et al.* reported that excess carrier lifetime decreases rapidly with increasing dopant concentration.[10] In our study, however, the decrease in lifetime of boron-doped Si and SiGe epilayers in proportion to boron-doping concentration is not observed.

IV. CONCLUSION

A noncontact 70 MHz rf inductively coupled bridge probe was built for a rapid, nondestructive evaluation of Si and SiGe epilayers grown by UHV-ECRCVD. The proper chemical pretreatment of the sample surface is required to measure the bulk minority carrier lifetime. Lifetime measurement with various Si surface cleaning conditions shows that *in situ* hydrogen plasma cleaning is very effective in obtaining good electrical quality Si and SiGe epilayers. The effects of various process parameters, such as the substrate dc bias, the distance of ECR layer from the substrate, and the substrate temperature on the minority carrier lifetime of Si and SiGe epilayers grown by UHV-ECRCVD, are examined and are found to coincide well with the results of TEM analysis.

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