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Surface and Interface Characterization of Sequentially Plasma Activated Silicon, Silicon dioxide and Germanium Wafers for Low Temperature Bonding Applications

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This article reports the sequentially plasma activated bonding (SPAB) of *n*–Ge with *p*–Si and SiO_2 at low temperature. Surface activation resulted in highest hydrophilicity of Ge compared with Si and SiO_2 counterparts. The highest hydrophilicity of Si, Ge and SiO_2 induced by O_2 RIE plasma was combined with their highest reactivity induced by MW N_2 radicals while maintaining smooth surface roughness. Weak bonding strength of Si/Ge and SiO_2/Ge in the SPAB at room temperature was improved after heating at 200°C, but they were still lower than that of Si/Si in the SPAB at room temperature, which is due to the unique reactivity of Ge. The deviation of the reverse bias behavior from a typical *p–n* junction is due to the low doping concentration in Ge. The degradation of current in the sequential heating resulted mainly from the oxidized surfaces of Ge and Si as well as the bonded interface.

Introduction

Germanium (Ge) has recently drawn considerable interest because of its large absorption coefficient at near infrared frequency range, and high carrier mobility [1]. On the other hand, silicon (Si) and silicon dioxide (SiO_2) offer a low cost solution for microelectronic and micro-electromechanical systems. Integration of Ge with Si and SiO_2 can find important applications in high performance photodetectors, solar cells, and Ge on insulator (SiO_2) (GeOI) structures. However, epitaxial growth of Ge on Si is not preferable because of high temperature (~700-900°C), high cost and large lattice mismatch (4.2 %). To address these issues, low temperature bonding of Ge/Si and Ge/SiO_2 has been proposed. Currently, direct bonding of Ge/Si and Ge/SiO_2 require high temperature (~300-400°C) at prolonged annealing (~48 hours) [2, 3]. The high bonding temperature creates thermal stress, interfacial voids and hence is not preferable for delicate structures and materials with large differences in thermal expansion coefficients. In addition to these problems, device throughput can be reduced by prolonged annealing, hence a low temperature, fast and spontaneous bonding technique for Ge/Si and Ge/SiO_2 is needed for practical applications.

One of the innovative techniques to improve the surface adhesion required for fast and spontaneous direct bonding is sequential plasma activation. This process maintains both flatness and smoothness of the activated surface. The sequential plasma consists of oxygen, nitrogen or argon (O_2/N_2/Ar) reactive ion etching (RIE) plasma followed by microwave (MW) oxygen or nitrogen (O_2/N_2) radicals. While RIE plasma removes contaminants and native oxides (i.e., SiO_2 in the case of Si) from the surface, MW radicals create a highly reactive surface [4]. It is well known that the chemical nature of

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$O_2/N_2/Ar$ gases is quite different. For example, O_2 RIE plasma and MW N_2 radicals, respectively induce hydroxyl and oxynitride layers [4], in addition to their physical sputtering capability. In contrast, the inert nature of Ar gas in the RIE plasma may lead to unprecedented phenomena. This discrepancy further leads to anomalous surface characteristics while treating with different types of MW radicals.

To gain insight the role of the RIE plasma and MW radicals and the best combination required for the sequential plasma activated bonding, the contact angle and surface roughness measurements have been performed as a function of gas in the RIE plasma and MW radicals. The contact angle between a drop of water and the wafer surface allows an understanding of the surface energy in activated wafers prior to bonding. The lower the contact angle, the higher the hydrophilicity and the easier the spontaneous bonding at room temperature. Recently, the hydrophilicity of Si and germanium (Ge) wafer surfaces were investigated through contact angle measurements after O_2 and N_2 RIE plasma activation [5]. Although the authors [5] recommended wafer bonding immediately after plasma activation, the contact angle measurements were done within two hours of the plasma activation. In fact, the surface hydrophilicity of wafers is an indispensable property to be investigated through contact angle measurements with a quick transition period following plasma activation. This allows understanding the applicability of SPAB, especially in bonding and packaging environments where a few minutes may be required for handling and alignment after surface activation.

The surface smoothness is one of the important criteria for direct wafer bonding which can be understood through surface roughness measurements. While the sequentially treated smooth surfaces of Si wafers have been bonded at room temperature [4, 6], the sequentially plasma activated wafer bonding of Ge/Si and Ge/SiO_2 was not successful (i.e., weak bonding) at room temperature due to weak Van der Waals forces. In order to achieve a strong bonding, the OH^- molecules on both surfaces need to react with one another. The problem arises when excess OH^- molecules are left intermittent on the Ge reacting surface due to low contact angle. Bonding, therefore, is hindered through the inability of these excess OH- molecules to form reacting pairs with the activated Si or SiO_2 surfaces, resulting in water creation at the bonding interface. Hence, to increase the bonding strength, the bonded Ge/Si and Ge/SiO_2 pairs were heated at lower temperature for a shorter duration compared with the reported works [3]. Heating steps are required in order for the transfer of thin layers of Ge in Smart Cut® technology [7] as well as to increase bonding strength. Therefore, the heating effect on *p–n* junction behavior of Si/Ge needs to be assessed for high efficiency solar cell applications.

Experimental

Commercially available Czochralski (CZ) grown one-side polished 2-in. p-type (100) silicon (Si) wafers, 2-in p-type (100) Si wafers with 50 nm thermal oxide (SiO_2) and 2-in (100) n-type germanium (Ge) wafers were used for the experiments. The thicknesses of the Si and Ge wafers were 275±25 μm and 400±25 μm, respectively. The resistivity of the Si and Ge wafers were 0.001-0.006 Ω-cm and >30 Ω-cm, respectively.

The wafer level hybrid plasma bonding (HPB) system, as shown in Figure 1, was used for the bonding experiments. The details of the HPB system can be found elsewhere

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[8]. The surfaces of Si, SiO_2 and Ge were activated without dipping in wet chemical and/or water. The wafers were activated using radio frequency (RF) reactive ion etching (RIE) plasma with a frequency of 13.56 MHz and/or microwave (MW) radicals with a frequency of 2.45 GHz. The RIE plasma and MW radicals were generated from O_2, N_2 or Ar gases, with a self-bias voltage of -227 V at the substrate electrode in the RIE plasma. For contact angle and surface roughness measurements, two separate sets of specimens of Si, SiO_2 and Ge were prepared using different plasma parameters. After plasma activation, the contact angle was measured using the sessile drop method with a deionized (DI) water droplet (9 µL) by the Kruss Drop Shape Analysis system DSA100. A contact angle below 2° cannot be detected using the equipment. An atomic force

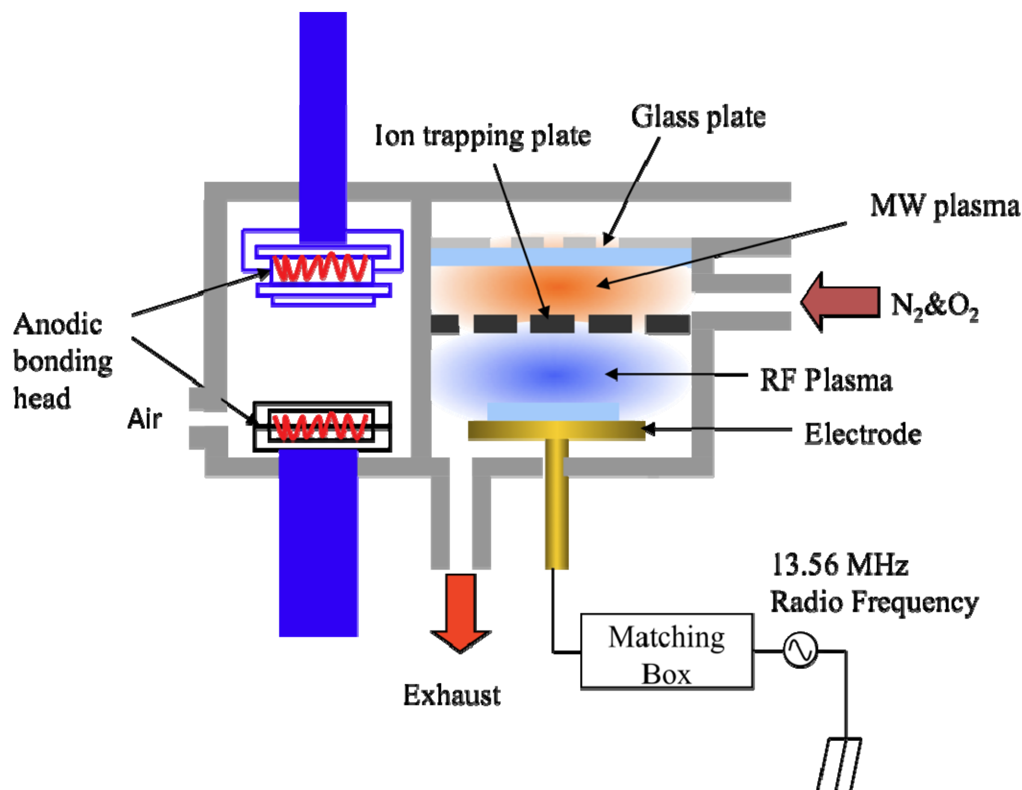

Figure 1. Hybrid plasma bonding system.

microscope (AFM) from Veeco (ICON) was used for the surface roughness measurement. Si tip in tapping mode was utilized over a scanning area of 2µm×2µm.

For the bonding experiments, the specimens were contacted under hand-applied pressure outside the vacuum chamber in a class 10,000 clean room with ambient conditions after surface activation. Then, the contacted specimens were cold rolled under rollers and compressed with 0.1 MPa of pressure to remove air trapped across the interface. Finally, the bonded specimens were heated in the anodic bonding chamber with no applied anodic voltage and a pressure of 0.08 MPa at 200°C for 4 h. Subsequent heating was done at 150°C for 4 h. After the bonded specimens were cooled down to room temperature, they were diced into 10 mm×10 mm squares and glued with copper jigs for the bonding strength measurement using a tensile pulling tester from Instron. In order to investigate the electrical characteristics of the bonded interfaces, highly

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conductive silver paste (typical resistivity ~ 3×10^{-5} Ω-cm) was used to make electrode contacts on the both sides of the bonded specimens. The HP-4145B semiconductor parameter analyzer was used for current-voltage (*I*-*V*) measurements.

Results and Discussion

Surface Hydrophilicity

In order to choose the appropriate gas for plasma activation, the contact angle measurements were performed within three minutes after surface activation. Table 1 shows the contact angles of a drop of DI water on Si, SiO_2 and Ge wafer surfaces compared with their surface roughness before and after surface activation. Before surface activation, the contact angle of Si, SiO_2 and Ge was 33.4, 52.0 and 68.0°, respectively. After RIE plasma treatment, the contact angles of Si surfaces were increased except in the O_2 RIE (29.1°) plasma. This lowest contact angle indicates that the O_2 RIE offers highest hydrophilicity among the RIE activations. After MW plasma treatment, the contact angle of Si was higher in the MW O_2 radicals than that in the MW N_2 radicals. The lower contact angles of Si in the MW N_2 radicals indicate higher reactivity. This is due to Si oxynitride (SiO_xN_y) and Si-N bonds [4]. Therefore, the sequential activation using O_2

Table I. Water contact angles and root mean square (RMS) surface roughness of Si, SiO_2 and Ge wafer surface before and after activated using different gases in RIE and MW plasma conditions. The RIE plasma parameters include O_2, N_2 and Ar gases with flow rate of 50 sccm, power 200 W, chamber pressure 60 Pa, and plasma treatment time 30 s. The MW N_2 radicals include O_2 and N_2 gases with a flow rate of 50 sccm, power 2000 W, chamber pressure 60 Pa, and plasma treatment time 30 s.

Surface Activation	Specimen	Contact Angle [°]	RMS Surface Roughness [nm]	AFM Images
Before activation	Si	33.4	0.17	fig 3(a)
	SiO_2	52.0	0.14	fig 3(d)
	Ge	68.0	0.25	fig 3(g)
O_2 RIE	Si	29.1	0.13	fig 3(b)
	SiO_2	38.5	0.17	
	Ge	<2.0	0.23	
N_2 RIE	Si	43.8	0.18	
	SiO_2	41.2	0.17	
	Ge	<2.0	0.81	fig 3(h)
Ar RIE	Si	51.8	16.2	fig 3(c)
	SiO_2	49.2	4.78	fig 3(e)
	Ge	27.0	0.41	fig 3(i)
O_2 MW	Si	27.4	0.20	
	SiO_2	37.8	0.15	
	Ge	<2.0	0.24	
N_2 MW	Si	16.1	0.18	
	SiO_2	29.5	0.10	fig 3(f)
	Ge	<2.0	0.23	

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RIE plasma followed by MW N_2 radicals provides surfaces with the highest hydrophilicity and reactivity required for the fast and spontaneous bonding.

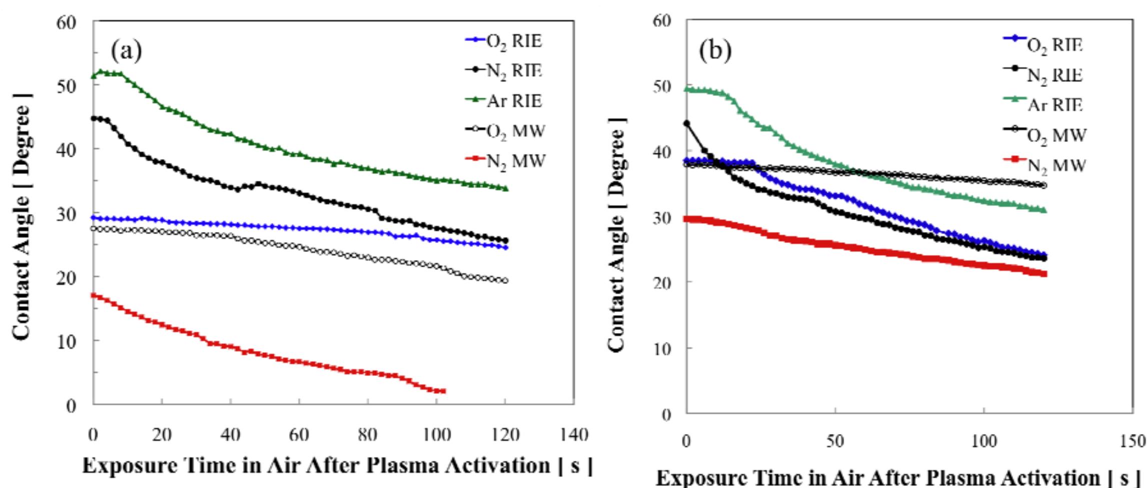

Figure 2. Elapsed time dependence of contact angles of a droplet of DI water on (a) Si and (b) SiO_2 wafer surface activated using different gases in the RIE plasma and MW plasma conditions. Contact angles were measured every 3 s starting from three minutes after plasma conditions.

In contrast to Si, the contact angles of SiO_2 were decreased after surface activation. The contact angles of the activated SiO_2 surfaces were 38.5, 41.2 and 49.2° in the O_2, N_2 and Ar RIE plasma, respectively. This increasing trend in the contact angles as a function of gas is similar to that of the activated Si surface in the RIE plasma. For MW plasma, the contact angle of SiO_2 was also lower in MW N_2 radicals compared with the MW O_2 radicals. This behavior is similar to that in the Si wafer.

As previously shown, the contact angle of Ge (68.0°) was remarkably high before activation when compared to Si and SiO_2. This highest contact angle of Ge dropped to less than 2.0° after activation regardless of the gases and plasma type except in the Ar RIE plasma. The sudden change of the contact angle attributes to the large number of free dangling sites generated by surface activation [5]. Immediately after plasma activation, the wafers were removed from the vacuum chamber that resulted in termination of large numbers of dangling bonds because of the reaction with the components of the surrounding class 10,000 clean room. The atmospheric components that resulted in the termination included water, impurities or even other dangling bonds. This effect can be investigated through measuring the contact angle of the Si and SiO_2 activated surfaces after leaving them in the atmosphere. But this effect on the Ge surface was not feasible because of very low contact angle after activation. The contact angle of a single water droplet was measured every three seconds for Si and SiO_2 surfaces, and no evaporation was observed during the 120 s process. Figure 2 shows the contact angles of (a) Si and (b) SiO_2 versus exposure time in air after plasma activation with different gases, respectively. The changing rate of contact angle development can be interpreted in terms of the surface reactivity. In both Si and SiO_2, the reactivity in the O_2 RIE plasma is lower than that in the N_2 and Ar RIE plasma, which have similar reactivities. The reactivity of

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SiO_2 in the O_2 RIE plasma is higher than that of Si. Both for Si and SiO_2, the reactivity of MW N_2 radicals is higher than MW O_2 radicals.

In the previous study of silicon bonding [4], O_2 RIE followed by MW N_2 radicals provided the highest reactive surfaces for strong bonding at room temperature. Based on this fact, similar reactivity behavior of the Si and SiO_2 surfaces in the RIE and MW radicals with different gases should provide the surface reactivity when combining MW N_2 radicals with O_2 RIE plasma that is required for spontaneous bonding at room temperature. The considerable change in the contact angle of Ge after surface activation and its reactivity with Si and SiO_2 determine the effectiveness of spontaneous bonding involving Ge wafer. In fact, the change in contact angle with RIE gases is presumably due to the different etching behavior and chemical reactivity, which can be correlated with the surface roughness and morphology.

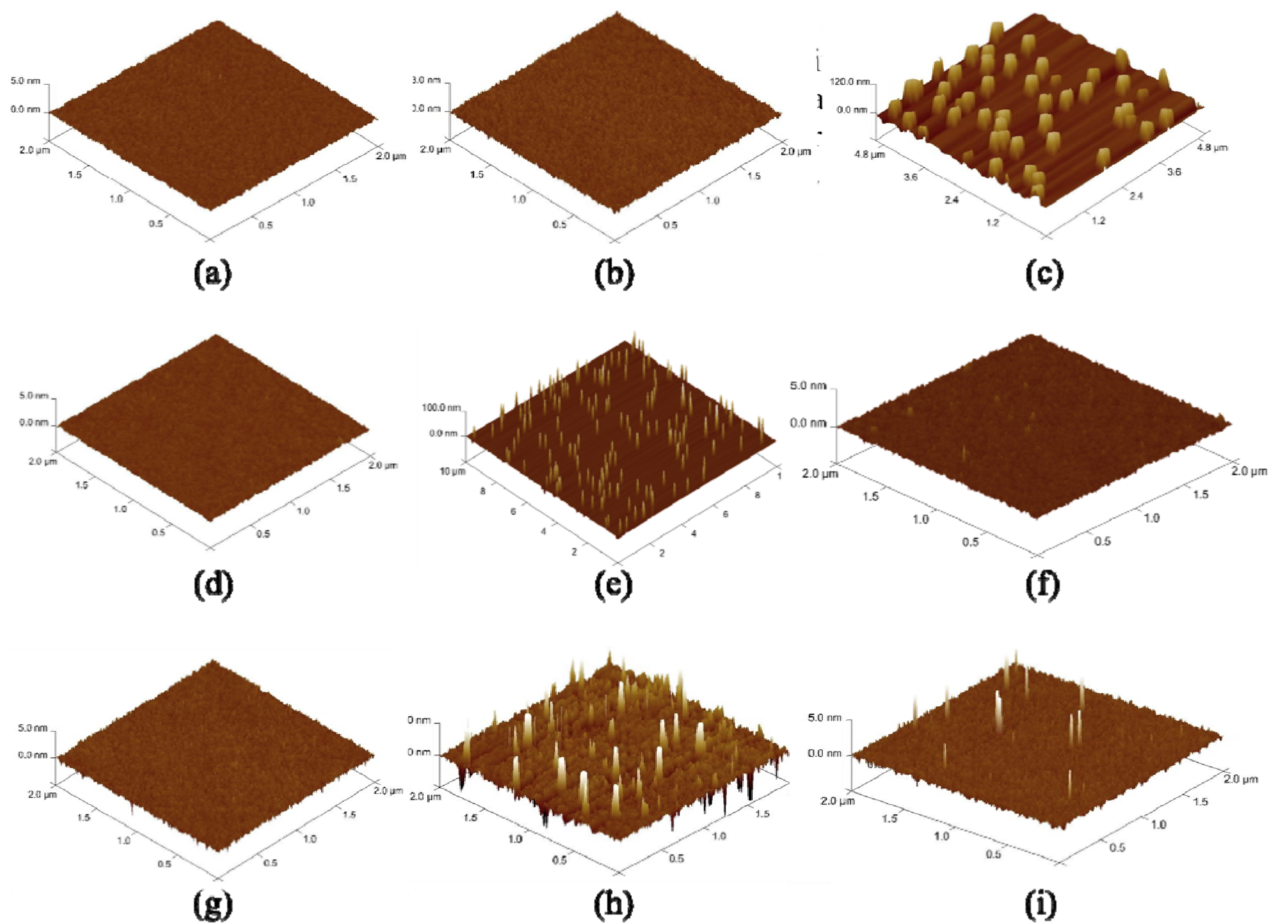

Figure 3. AFM images of Si (a, b, c), SiO_2 (d, e, f) and Ge (g, h, i) wafer surfaces. For Si, the included images are (a) before activation, and after activation with (b) O_2 RIE plasma and (c) Ar RIE plasma. For SiO_2, they are (d) before activation, and after activation with (e) Ar RIE plasma and (f) MW N_2 radicals. For Ge, the surfaces are (g) before activation, and after activation with (h) N_2 RIE plasma and (i) Ar RIE plasma.

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Surface Roughness

The root mean square (RMS) surface roughness of Si, SiO_2 and Ge before surface activation was 0.17, 0.14 and 0.25 nm, respectively. After surface activation, the surface roughness and contact angle were randomly varied with different plasma types and gases. A correlation presented itself as the higher the surface roughness observed, the higher the contact angle, except for Ge. In the case of Ge under N_2 RIE, while the surface roughness increased to 0.81 nm, the contact angle decreased to less than 2°. The surface roughness of Si, SiO_2 and Ge was increased in the Ar RIE, but the surface roughness of Ge was still lower than that in the N_2 RIE. Furthermore, no considerable change in the surface roughness was observed in the MW plasma other than the contact angle. This is due to the chemical reactivity and insignificant physical sputtering behavior (because MW plasma has no biasing voltage) of the MW plasma radicals [4]. In the case of rough surface, air traps between the water droplet and the wafer surface and hinders spreading the water droplet. This results in a higher contact angle [Table I]. In the Ar RIE plasma, an anomalous surface roughness was observed for all the wafers. Figure 3 shows the surface morphologies of Si, SiO_2 and Ge wafer surface using AFM. For Si, (a) before activation and after activation with (b) O_2 RIE, and (c) Ar RIE, for SiO_2, (d) before activation and after activation with (e) Ar RIE plasma and (f) MW N_2 radicals, for Ge, (g) before activation and after activation with (h) N_2 RIE plasma and (i) Ar RIE plasma. The surface roughness of Si, SiO_2 and Ge over an area of 2×2 μm^2 was 16.2, 4.78 and 0.41 nm, respectively. The anomalous surface in the Ar RIE plasma activation contained nanoneedle like structures with heights of about 100 nm [Fig 3c and e]. The diameter of the nanoneedles on Si was bigger than that on SiO_2. Further research is required to find insight into the formation of the nanoneedles in the Ar RIE plasma. However, the sequentially treated Si surfaces using O_2 RIE plasma followed by MW N_2 radicals provided the smoother surfaces with higher hydrophilicity as compared to the individual treatments [4, 8]. Therefore, this study indicates that O_2 RIE plasma combined with MW N_2 radicals offers the lowest surface roughness of Si, SiO_2 and Ge.

Tensile Strength

Based on the best combination achieved through the contact angle and surface roughness investigations, the Si/Ge and SiO_2/Ge were bonded at room temperature. However, the bonding strength of Si/Ge and SiO_2/Ge was low, and debonded during dicing. To increase the bonding strength, the bonded wafers were heated at 200°C and subsequently annealed at 150°C. Figure 4 shows the bonding strength of the sequentially plasma activated Si/Ge and SiO_2/Ge wafers after annealing at 200°C, as well as 200°C followed by 150°C in air for 4 h at each step. At 200°C, while the bonding strength of Si/Ge was about 1 MPa, it was about 5 MPa for SiO_2/Ge. This difference in the bonding strength is due to the unique reactivity of Ge and its discrepant interactions with Si and SiO_2. Therefore, in terms of the reactivity, the higher reactivity difference of SiO_2/Ge than that of Si/Ge was responsible for higher bonding strength of SiO_2/Ge wafers. Further annealing at 150°C, the bonding strength of Si/Ge was increased, but decreased in the case of SiO_2/Ge bonding. As previously mentioned, the reactions between Si/Ge and SiO_2/Ge produce water across the interface after heating at 200°C. In order to enhance bonding strength, the water needs to be diffused out of the interface. The bonding strength of SiO_2/Ge was higher compared to the Si/Ge, which was due to the absorption

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of water by the SiO_2 layer through the annealing process at 200°C [6]. Further annealing at 150°C desorbed the water from SiO_2 and initiated a reaction at the interface of SiO_2/Ge, causing a brittle interfacial layer and reduced bonding strength of SiO_2/Ge. Contrastingly, after the annealing of Si/Ge in identical conditions, the remaining interfacial water was diffused through the plasma-induced oxide and reacted with the bulk of silicon and Ge to form respective oxides. The bonding strength of Si/Ge and SiO_2/Ge was relatively low compared to that of Si/Si in the SPAB at room temperature

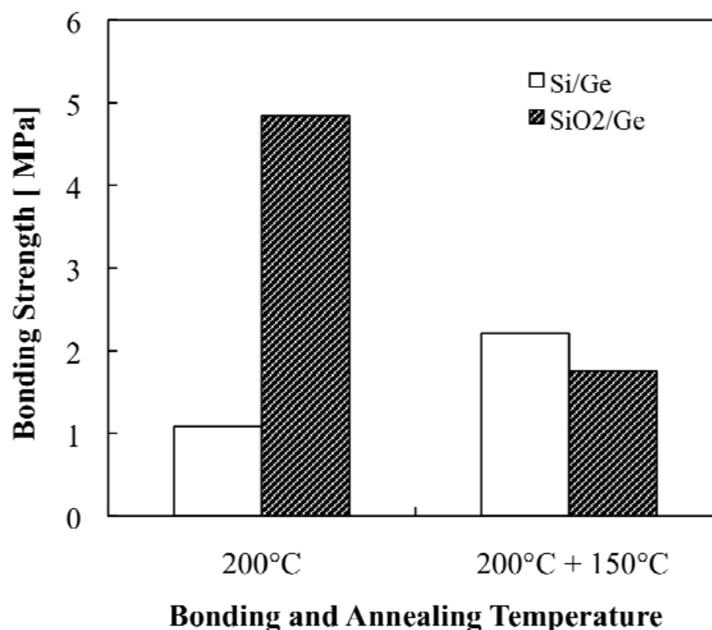

Figure 4. Bonding strength of the sequentially bonded Si/Ge and SiO_2/Ge wafers. After contacting at room temperature, the bonded wafers were annealed in air for 4 h at each step.

[6], and Si/glass in the HPB at 200°C under 1 kV [9]. The relatively low bonding strength in the Si/Ge and SiO_2/Ge again can be attributed to the high reactivity of Ge wafer after surface activation. It is well known that Si and Ge surfaces have native oxides. The bond length of Ge-O (oxygen) is higher than that of Si-O [10]. Therefore, the O_2 RIE plasma breaks Ge-O bonds and MW N_2 radicals generate Ge oxynitride relatively easier than Si-O that results in a large number of free dangling sites on Ge surface. These sites occupy higher number of OH^- molecules on the activated Ge surface (as observed by the low contact angle of Ge after plasma activation) when exposed to the clean room ambient, which results in weak bonding at room temperature. In order to improve the bonding strength, it was essential to remove the large number of OH^- molecules cross the interface. The heating at 200°C was not sufficient to achieve covalent bonding between Si/Ge. Further research is underway at higher temperature to achieve higher bonding strength of Si/Ge.

Current-Voltage Behavior of Bonded Interface

Figure 5 shows the current density of *p–n* junction of Si/Ge, and SiO_2/Ge bonded wafers as function of applied voltage. The insets of the figure show the logarithmic

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current densities of the interfaces. The *p–n* junction current density increased as the applied voltage increased. The *p–n* junction of Si/Ge in the forward bias condition follows the typical behavior due to low impurity of Ge [11]. However, the logarithmic current of Si/Ge shows deviation from the ideal behavior of a *p–n* junction due to the presence of plasma induced oxides as well as trapping centers and defects [12]. The electrical current

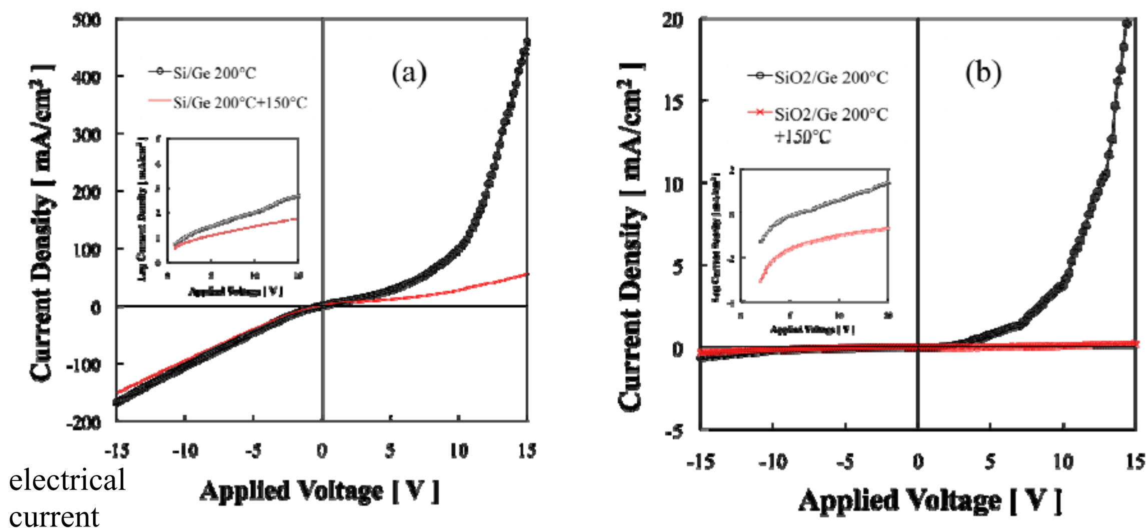

Figure 5. Interfacial current density of *p–n* junction of (a) Si/Ge, and (b) SiO_2/Ge bonded wafers as a function of applied voltage. Due to low impurity of Ge, the *p–n* junction of Si/Ge follows only at the forward bias condition. Insets include logarithmic current density vs. applied voltage.

density decreased after sequential annealing both in the Si/Ge and SiO_2/Ge. This was caused by inclusion of the annealing induced Si and Ge surface oxides [13] and interface oxide [14] in the current measurements. The influence of the annealing induced Si surface oxide on the current-voltage behavior of the surface activated Si/Si bonding in UHV has been reported [13]. However, the estimated area resistance of Si/Ge interface is higher than that in the reported work for solar cell application [14]. This relatively higher value is due to higher resistance and nonohmic contact (due to silver pasted electrodes) used in our study. In the case of SiO_2/Ge bonding, lower current density was observed compared to that of Si/Ge. While the current density in Si/Ge was 460 mA/cm^2 at 15 V, it was 24 mA/cm^2 for SiO_2/Ge. In fact, the bonding structure used for the measurement of interfacial current contains Si-SiO_2 (50 nm thick)/Ge. This reduced current density in the SiO_2/Ge indicates that it can be used for electrical isolation required for the germanium photodetectors integrated on silicon waveguides [2].

Conclusions

The surfaces of Si, SiO_2 and Ge wafers have been investigated through contact angle and surface morphology observations in order to develop the sequentially plasma activated bonding (SPAB) of *p*–Si/*n*–Ge and SiO_2/Ge at low temperature. Through surface activation, the hydrophilicity of Ge changes most dramatically from lowest to highest in comparison to its Si and SiO_2 counterparts. Argon RIE plasma generates anomalous surface behavior because of its nonreactivity with the surface. The highest

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hydrophilicity of Si, Ge and SiO_2 induced by O_2 RIE plasma was combined with their highest reactivity induced by MW N_2 radicals while maintaining smooth surface roughness. Weak bonding strength of Si/Ge and SiO_2/Ge in the SPAB at room temperature was improved after heating at 200°C, but they were still lower than that of Si/Si in the SPAB at room temperature, which is due to the unique reactivity of Ge. The deviation of the reverse bias behavior from a typical *p-n* junction is due to the low doping concentration in Ge. The degradation of current in the sequential heating resulted mainly from the oxidized surfaces of Ge and Si as well as the bonded interface. The SPAB of *p*–Si/*n*–Ge and SiO_2/Ge demonstrates the potentials in solar cell and photodetector applications at low temperature.

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