Analysis of Au/Si Schottky Barrier Type AC Surface Photovoltage in Silicon Wafer Surface Dipped into Au Aqueous Solution

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SUMMARY

The state of a gold (Au)-contaminated silicon (Si) surface dipped in an aqueous solution, with a standard Au solution, was investigated using the alternating current surface photovoltage (AC SPV) method [1, 2]. On a Au-contaminated n-type Si wafer surface, frequency-dependent AC SPV was observed and it was shown that the n-type Si surface was in a depleted or weakly inverted state. The cutoff frequency was found from the AC SPV frequency dependence curve of the Au-contaminated n-type Si wafer and an analysis of the Si surface potential was performed. With those results, the band diagram of the Au/n-type Si Schottky contact was found and a Schottky barrier height Φ_B was calculated as 0.73 to 0.76 eV. This value is close to the reported value $\Phi_B = 0.79$ eV and supports the formation of a Au/n-type Si Schottky barrier. From these results, a model for AC SPV occurrence is proposed in which the n-type Si surface is weakly inverted with the formation of a Au/n-type Si Schottky barrier. Meanwhile, in a Au-contaminated p-type Si wafer, the measured voltage was the noise level of the measurement device and indicates a low surface potential. © 2007 Wiley Periodicals, Inc. Electron Comm Jpn Pt 2, 90(6): 27–33, 2007; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/ecjb.20308

Key words: silicon wafer; surface photovoltage; Au; Schottky barrier.

1. Introduction

In the alternating current surface photovoltage (AC SPV) method, a semiconductor surface in a depleted or inverted state is irradiated with chopped light and the resulting change in the surface potential is measured as the alternating current voltage [1–5]. The AC SPV occurring on the semiconductor surface can be measured with a capacitive coupling system through the gap between the electrode and the sample. Consequently, the AC SPV method is nondestructive and noncontact, and makes it possible to measure the oxide charge or surface state of a semiconductor surface without the application of a high electric field to an oxide film on the semiconductor surface.

Metal impurities remaining in a Si wafer surface are known to induce charges by bonding with Si and/or oxygen (O) when an oxide film is formed on the wafer surface [6–10]. Ilner [11] and Eisenman [12] have proposed a model in which, when trivalent aluminum (Al) replaces tetravalent Si in SiO_2, networks of $(SiOAl_4)^-$ and/or $(AlOSi)^-$ are formed and negative charges are induced. When negative charges are present in the oxide film on an n-type Si surface, a depletion layer forms in the n-type Si surface and an inversion layer is formed when there is a large number of negative charges in the oxide film. Likewise, if positive charges are present in the oxide film on a p-type Si surface, a depletion layer or inversion layer is formed in the p-type Si surface depending on the quantity of positive charges. AC SPV occurs when chopped light is

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irradiated on a Si surface which is depleted or inverted and the surface potential has changed.

Meanwhile, it is reported that, when a Si wafer is dipped in an aqueous solution including copper (Cu), an oxidation reduction reaction due to the injection and acceptance of electrons on the Si surface $Cu^{2+} + 2e^- \rightarrow Cu$ occurs and a Cu/Si Schottky contact is formed, a depletion layer and/or inversion layer is formed in the Si surface, and the potential of the Si surface changes [13].

In order to analyze such a change in the surface state due to metal impurities, the authors have made a systematic study of the change in potential of Si surfaces contaminated with various metals, or the oxide charges induced by the metals, using the AC SPV method. This paper focuses on Au believed to form a Schottky barrier in a Si surface and the AC SPV frequency dependence in Au-contaminated n-type and p-type Si(001) surfaces was measured. The Si surface potential was analyzed from the AC SPV frequency dependence measured and a model for AC SPV occurrence due to the formation of a Au/n-type Si Schottky barrier was proposed.

2. AC SPV Device

Figure 1 shows a schematic diagram of the AC SPV device used in this research. AC SPV is the alternating current component of the voltage occurring on a Si wafer surface when irradiated by chopped light. In this device, a blue light emitting diode (wavelength: λ = 470 nm) was used as the AC SPV excitation light source, and operated by a square wave (frequency f = 2 Hz to 80 kHz) generated with a function generator. The chopped light passes through
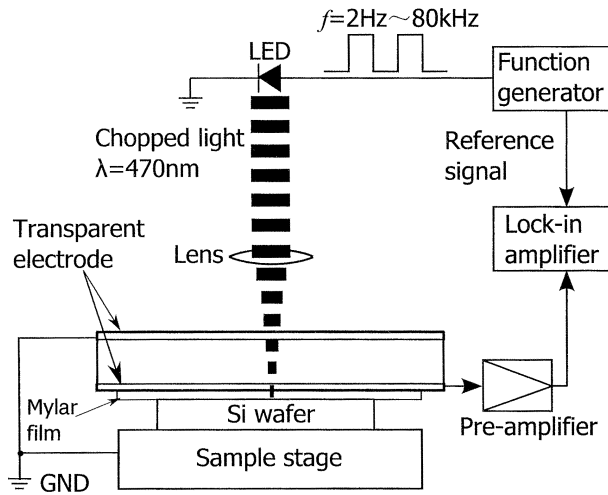

Fig. 1. Schematic diagram of measurement device of AC SPV.

a lens and irradiates the Si wafer surface. AC SPV is detected with capacitive coupling via a capacitor formed by the Si wafer and transparent electrode [ITO (indium tin oxide) film (film thickness 1250 Å, sheet resistance 130 Ω/□)] placed opposite to it. The transparent electrodes are formed on the upper and lower surfaces of a 20-mm-thick quartz panel. The transparent electrode on the upper surface is grounded and serves as an electric shield. The transparent electrode on the lower surface becomes the signal detector for AC SPV on the wafer surface. The Si wafer is placed on a stainless-steel sample stage, and a Mylar film which is an insulating film (thickness: 16 μm) is placed on the Si wafer surface. The lower transparent electrode is pressed thereon with a constant load. The AC SPV signal detected by the lower transparent electrode is passed through a preamplifier and amplified 100 times, and is then measured with a lock-in amp. Because the AC SPV values are extremely small (generally, 1 μV to 1 mV), the insulation between the lower transparent electrode and ground is very important. In this device, a resistance and a capacitance between the lower transparent electrode and ground is larger than 100 MΩ and less than 220 pF, respectively. This device is placed in a black box to avoid the interference of stray light from outside [14].

3. Analysis of Surface Potential in AC SPV Characteristics

When the surface of the Si wafer is in a depleted or inverted state, the surface impedance z is expressed as a parallel connection of the majority carrier conductance g_{mj}, interface trap conductance g_{it}, depletion layer capacitance c_{dp}, and interface trap capacitance c_{it}:

$$z = \frac{1}{(g_{mj} + g_{it}) + j\omega(c_{it} + c_{dp})} \quad (1)$$

At this time, the alternating current surface photovoltage V_{ph} caused by incident light is expressed as

$$V_{ph} = \frac{I_{ph}}{S} |z| \eta_r \eta_d \quad (2)$$

I_{ph} is the surface photocurrent, S is the wafer surface area, η_r is the photovoltage reduction factor, and η_d is the surface photovoltage dividing factor due to the capacitance between the sample and the lower transparent electrode and the lock-in amp input capacitance. Consequently, the characteristics of the AC SPV can be calculated by finding the absolute value |z| of the surface impedance of various frequencies and the surface photocurrent.

The influence of interface traps on the AC SPV characteristics appears in a limited frequency range near the cutoff frequency f_c; but when the interface trap density is

negligibly small, the absolute value of the surface impedance z becomes as follows:

$$|z| = \frac{1}{\sqrt{g_{mj}^2 + \omega^2 c_{dp}^2}} \qquad (3)$$

From this equation, the cutoff frequency f_c in the AC SPV characteristics can be expressed as

$$f_c = \frac{g_{mj}}{2\pi c_{dp}} \qquad (4)$$

g_{mj} and c_{dp} are functions of the Fermi potential Φ_F and the surface potential Φ_S; the surface potential Φ_S can be found from the cutoff frequency f_c [15].

4. Experiment

Samples were prepared from commercially available Si(100) wafers, 125 mm in diameter. The wafers were n-type and p-type with a resistivity of $10 \pm 2\Omega \cdot$cm and thickness of $625 \pm 20\,\mu$m.

The wafers were first washed in an ultrasonic bath of ultrapure water (resistivity 18 Mohm-cm) for 5 minutes, then dipped in a 1% aqueous solution of HF to remove the native oxide film for 5 minutes. Thereafter, the wafers were rinsed for 5 minutes in ultrapure water. Next, the Si wafers were dipped for 20 minutes in an aqueous solution containing $10\,\mu$g/ml Au, and then rinsed for 10 minutes in ultrapure water. For the aqueous Au solution, a standard Au solution (1 mol Au/liter hydrochloric acid solution, Au density 1000 ppm) was used.

Table 1 shows the results of the analysis of the metal impurity concentrations in the Au-contaminated Si surface from the above-mentioned procedure. These measurements were made using the atomic absorption spectroscopy (AAS) method by Renesas Kodaira Semiconductor, Inc. "Au" indicates samples processed under the above-mentioned conditions. "HF" refers to samples which were processed as follows. The samples were washed in an ultrasonic bath of ultrapure water for 5 minutes, and then dipped in a 1% HF aqueous solution for 5 minutes to remove the native oxide film. Thereafter, the wafers were rinsed for 5 minutes in ultrapure water, and then dipped in ultrapure water for 20 minutes. The surface Au concentration of the "Au" samples was 2.0×10^{15} atoms/cm^2. On the surface of Au-contaminated samples, concentrations of iron (Fe) and Cu on the order of 10^{11} atoms/cm^2 were present, but at these levels, the influence of these surface impurities on the AC SPV properties shown in this paper can be ignored [13, 14].

Also, X-ray photoelectron spectroscopy (XPS) was used to examine the Au bonding state on the silicon surface. The XPS measurements used MgKα ($h\nu$ = 1253.6 eV) X-rays at an output of 300 W (15 kV, 20 mA) and were performed using a D-CMA (Perkin Elmer 15-255G). The take-off angle was set to 78 degrees.

5. Experimental Results and Discussion

Figure 2 shows the Si2p and Au4f XPS spectra of Au-contaminated n-type Si wafers which were exposed to air for 15 minutes and 8 days. For the Au4f spectra of the sample surfaces exposed to air for 15 minutes and 8 days, the Au4f$_{7/2}$ binding energy was 84.2 eV for both. This is close to the reported Au4f$_{7/2}$ binding energy of 84 eV from Au [16]. According to Aita and Tran [17] and Dickinson and colleagues [18], the Au4f$_{7/2}$ and Au4f$_{5/2}$ binding energies from Au$_2$O$_3$ were 86.3 and 89.6 eV, respectively; the Au4f$_{7/2}$ and Au4f$_{5/2}$ binding energies from Au(OH)$_3$ were 87.7 and 91.4 eV, respectively. In this research, the Si wafers were dipped in an aqueous Au solution, and it was thought that Au atoms, if present individually, would bond with O or OH. However, peaks caused by Au$_2$O$_3$ or Au(OH)$_3$ could not be confirmed from the measured Au4f spectra. From a comparison of the measured Au4f binding energies and these reported binding energies, it was inferred that Au was not present in atomic form and formed clusters instead. For the Si2p spectra, the Si(Bulk) binding energy was 99.1 eV for both the samples exposed to air for 15 minutes and for 8 days. A reduction in Si(Bulk) peak intensity and an increase in Si oxide peak intensity due to the increased exposure time could be confirmed and it was understood that an oxide film had formed. Usually, the film thickness of SiO$_2$ formed on a Si surface is calculated based on the measured Si(Bulk) and Si oxide Si2p integrated intensity. In this experiment, however, Au is present on the Si surface and it is difficult to correctly calculate the SiO$_2$ film thickness because the depth profile of the Au is unknown. Accordingly, as a rough estimate, the SiO$_2$ film thicknesses from 15 minutes' and 8 days' exposure to air were 2.7 nm and 3.1 nm when calculated from the Si2p integrated inten-

Table 1. Metal concentration at Si wafer surface deliberately contaminated in Au aqueous solution

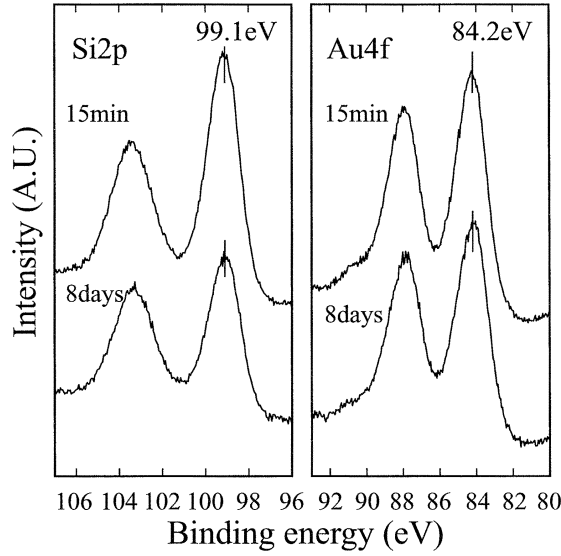|	Concentration ($\times 10^{11}$ atoms/cm^2)							
	Al	Fe	Cu	Ni	Cr	Zn	Co	Au
"HF"	18	<5.9	<2.2	<12	5.0	<4.5	<0.36	<5.6
"Au"	<15	6.7	7.2	<12	<4.3	<4.5	<0.36	2.0×10^4

Fig. 2. Si2p and Au4f spectra from the surface of n-type Si wafer contaminated by Au in Au aqueous solution.

sity of the Si(Bulk) and Si oxide. From the Au4f and Si2p spectra, it is thought that the Au did not bond with O and was still present in clusters in the native oxide growth due to exposure to air.

Figure 3 shows the frequency dependence of AC SPV in a Au-contaminated n-type Si wafer. For comparison, the frequency dependence of the AC SPV characteristics of an
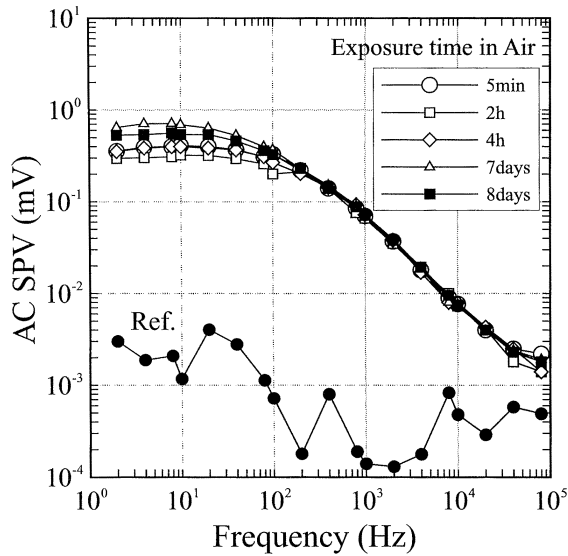
n-type Si wafer dipped into an aqueous solution of 1 mol/liter HCl diluted with ultrapure water is also shown ("Ref." in the drawing). AC SPV occurred immediately after the n-type Si wafer was dipped in the aqueous Au solution. The AC SPV values were approximately constant in the lower frequency range, below frequencies of 40 Hz, and were inversely proportional to the frequencies in the high-frequency range. According to the theory of AC SPV [4, 5], the AC SPV frequency dependence shows that the n-type Si wafer surface is in a depleted or weakly inverted state. Meanwhile, the properties of the reference sample (Ref.) were at the noise level of the device and AC SPV was below the detection limit. Consequently, the appearance of AC SPV in an n-type Si wafer dipped in an aqueous Au solution is clearly caused by the presence of the Au. The change in AC SPV frequency dependence due to the air exposure time is very small. When an n-type Si wafer is dipped in an aqueous Au solution, it is likely that the Au ions are reduced to Au by electron capture ($Au^{3+} + 3e^- \rightarrow Au$) in the Si surface because of the difference in the oxidation reduction potential, resulting in formation of a Au/Si Schottky barrier. Therefore, AC SPV occurs due to the formation of this Au/n-Si Schottky barrier causing a depletion and/or weak inversion layer at the Si surface.

Figure 4 shows a plot of only the AC SPV characteristics for the sample after 5 minutes of air exposure in Fig. 3. The cutoff frequency f_c was found to be 180 Hz from the intersection of the lines extrapolated from the AC SPV values in the low-frequency range and the AC SPV characteristics which are inversely proportional to frequencies in

Fig. 3. Frequency-dependent AC SPV from the surface of n-type Si wafer contaminated by Au in Au aqueous solution.
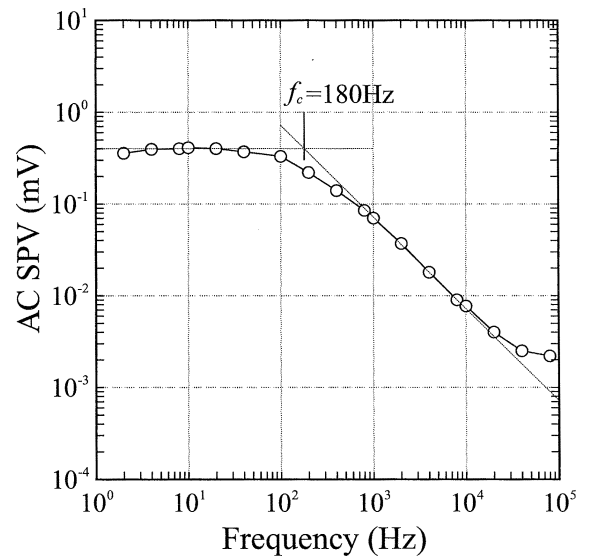

Fig. 4. Cutoff frequency of AC SPV characteristic from the surface of n-type Si wafer contaminated by Au in Au aqueous solution (exposure time in air: 5 min).

the high-frequency range. As a result of an analysis using the HSJ model proposed by Munakata and colleagues [4, 5], the surface potential (Φ_S) was calculated from this cutoff frequency f_c to be –0.44 eV. Also, the Fermi potential (Φ_F) was found to be –0.27 eV from the resistivity of the n-type Si wafers (10 $\Omega\cdot$cm). From these values of Φ_S and Φ_F, this Si surface is in a weakly inverted state. Likewise, for the measured AC SPV frequency dependence for all of the air exposure times, f_c was found to vary in the range of 100 to 240 Hz without dependence on the air exposure time; according to the analysis, Φ_S was calculated to be –0.44 to –0.47 eV. The band bending in the Si surface causes a change in the Si2p binding energy. The binding energies of the Si2p spectra for the 15-minute and 8-day air exposure as shown in Fig. 2 are both 99.1 eV which is 0.2 eV lower than the reported value (99.3 eV) for bulk silicon [16]. This 0.2 eV difference in binding energies reflects the band bending at the silicon surface due to Au contamination. However, there is a difference of 0.24 to 0.27 eV from the Φ_S value found from AC SPV. This is because the Si2p photoelectrons are emitted from the Si surface which is in a depleted or weakly inverted state, meaning from the region where the bands bend continuously toward the surface; this can be interpreted as indicating a small value relative to the surface potential Φ_S calculated from AC SPV characteristics.

Figure 5 shows a band model of the Au/n-Si Schottky contact based on Φ_S calculated from the cutoff frequency f_c = 180 Hz in the AC SPV frequency dependence after 5 minutes of air exposure. The height Φ_B of the Au/n-Si Schottky barrier was calculated as 0.73 eV from the energy gap E_g (1.11 eV) of Si, Fermi potential Φ_F and surface potential Φ_S found in this research. This value is close to the reported Schottky barrier height Φ_B = 0.79 eV of the Au/n-Si Schottky contact and supports the formation of the
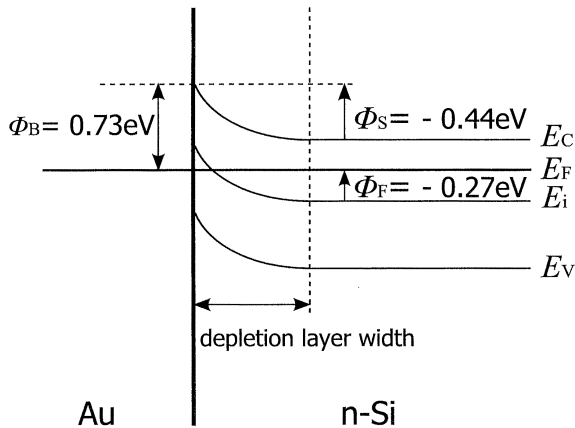

Fig. 5. Schematic energy band diagram of Au/n-type Si Schottky contact.

Au/n-Si Schottky contact. From the measured AC SPV frequency dependence for all of the air exposure times, Φ_B = 0.73 to 0.76 eV was calculated and the change due to air exposure is within the margin of error of the measurements.

Hiraki [20] and Okuno and colleagues [21] studied the Au-Si reaction with respect to the thickness of Au film deposited on a Si(111)-(7 × 7) surface at a slow vapor deposition rate at room temperature by using Auger electron spectroscopy (AES), energy loss spectroscopy (ELS), and low-energy electron diffraction (LEED). According to those results, 1 to 2 ML of Au induces no reaction even when the sample temperature is raised to 800 °C; however, more than 4 ML of Au immediately reacts with the Si and an amorphous Au-Si alloy is formed. Cros and colleagues [22] deposited 4 ML of Au on an Si(111) surface cleaned in ultrahigh vacuum, resulting in a Au-Si alloy formation, then examined the oxide growth on the Au-Si alloy surface by exposure of molecular oxygen (O_2) at room temperature using AES and ELS. They reported that an oxide film is formed on the Au-Si alloy surface as a result of the Si atoms passing through the Au-Si alloy from the Si substrate to the surface and reacting with the O_2. According to the results of the chemical analysis in this research, the quantity of Au was approximately 3 ML. When the Si wafer was dipped in the aqueous Au solution, it is expected that an oxide film is formed at the same time as the Au contamination of the Si surface and it is not likely that the Au covers the entire Si surface. Also, for the sample which was exposed to air for 15 minutes after being dipped in the aqueous Au solution, the oxide film on the surface was etched using an HF solution and AES measurements of the surface were made. As a result, Si LVV peaks at 90 eV and 94 to 95 eV [20–22], showing the presence of a Au-Si alloy, were not observed. When the experimental results of Hiraki [20] and Cros and colleagues [22] are considered together, the Au clusters depositing on the Si surface in this research are less than 4 ML and are supposed to partially cover the Si surface. Presupposing this situation, the quantity of Au on the Si surface is insufficient for the quantity of Au measured by the chemical analysis, suggesting that Au is present in the SiO_2 and on the SiO_2 surface as well.

Figure 6 shows the AC SPV measurement results for an Au-contaminated p-type Si wafer. The voltages measured in the p-type Si wafer are low and all indicate the noise level of the AC SPV device. The surface potential Φ_S which can be measured with this AC SPV device is estimated to be approximately 0.35 eV from the noise level of the device. Consequently, the surface potential Φ_S in an Au-contaminated p-type Si wafer was below the detection limit of 0.35 eV.
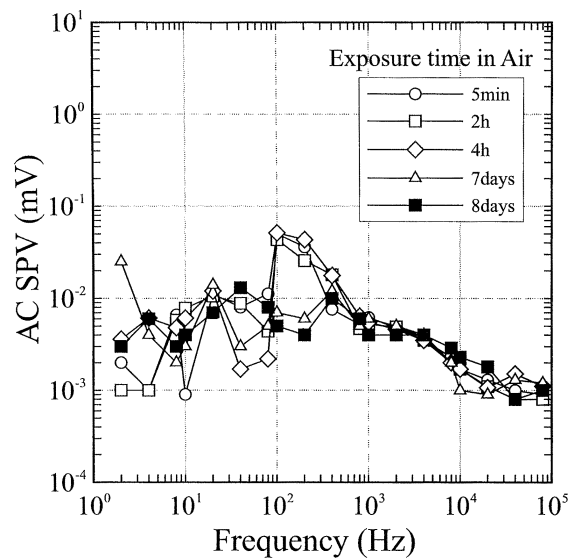
Fig. 6.   Frequency-dependent AC SPV from the surface of p-type Si wafer contaminated by Au in Au aqueous solution.

6.   Conclusions

The state of a Si wafer surface contaminated with Au from being dipped in an aqueous Au solution was examined with an AC SPV method. The AC SPV frequency characteristics of the Au-contaminated n-type Si wafer were constant for frequencies of 40 Hz and below, and were inversely proportional to the frequencies at higher frequencies. The Si surface was shown to be in a depleted or weakly inverted state. From an analysis of the cutoff frequency f_c of the AC SPV frequency characteristics, the Si surface potential Φ_S was found to be –0.44 to –0.47 eV and the Schottky barrier height Φ_B due to the Au/n-type Si Schottky contact was found to be 0.73 to 0.76 eV. This value is close to the reported value $\Phi_B = 0.79$ eV and supports the formation of a Au/n-type Si Schottky barrier. Based on these results, a model for AC SPV occurrence, due to the formation of a Au/n-type Si Schottky barrier, is proposed. Also, the thickness of the native oxide formed on the Si surface is increased by an increased air exposure time, but the AC SPV characteristics do not change. In the Au4f XPS spectral measurement results as well, the Au4f$_{7/2}$ binding energy was not changed by exposure to air and a change in the Au bonding state was not observed. These results reveal that the Au/n-type Si Schottky barrier is present in a stable form.

Meanwhile, in an Au-contaminated p-type Si wafer, the measured voltages indicated the noise level of the device. Even if AC SPV did occur, it was very slight and the surface potential was understood to be small.

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