Annealing Temperature on Contact Properties between Nickel Film and Hydrogen-Terminated Single Crystal Diamond

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Abstract: Ohmic contact of nickel on hydrogen-terminated single-crystal diamond film was investigated with an annealing temperature ranging from room temperature to 750 °C in hydrogen atmosphere. Nickel film was deposited on a hydrogen-terminated single-crystal diamond surface with gold film in order to protect it from oxidation. Contact properties between nickel and hydrogen-terminated single crystal diamond were measured by a circular transmission line model. The lowest specific contact resistivity was 7.82×10⁻⁵ Ω cm² at annealing temperature of 750 °C, indicating good ohmic contact, which reveals improved thermal stability by increasing temperature.

Keywords: H-diamond; I-V; Ohmic contact; CTLM; specific contact resistance

1. Introduction

Diamond has many attractive properties as a semiconductor, such as wide band gap (5.47 eV), large breakdown field (>10 MV cm⁻¹), high saturation velocities (1.5×10⁷ cm s⁻¹ for electrons and 1.05×10⁷ cm s⁻¹ for holes), high carrier mobility (4500 cm² V⁻¹ s⁻¹ for electrons, 3800 cm² V⁻¹ s⁻¹ for holes), and highest thermal conductivity (22 W cm⁻¹ K⁻¹), making it a promising semiconductor for future high-frequency, high-power, and high-temperature electronic devices, such as metal-oxide-semiconductor field effect transistors (MOSFETs), which have been studied extensively [1–17]. Nevertheless, the activation energies are too large to utilize the conventional doping technology for diamond dopants (370 meV for boron and 570 meV for phosphorus) as it is difficult for them to be activated at room temperature (RT). In order to solve this problem, δ-doping technology was used, but this technology had great limitations in device fabrication and cannot be widely used due to its complex process and low carrier mobility [18–20]. Fortunately, a new technology breaks this deadlock by treating diamond with hydrogen plasma to form hydrogen termination on a diamond (H-diamond) surface, which generates two-dimensional hole gas (2DHG) (density: 10¹³ cm⁻²; mobility: 50–150 cm² V⁻¹ s⁻¹). This 2DHG layer resides several nanometers below the diamond surface which possesses many characteristics such as an almost-constant current density within a temperature range from 20 to 300K and ease of fabrication [9,12]. Stable ohmic contact with low resistance is crucial to ensure future applications in low-dimensional devices. For example, high-quality ohmic contact with a low specific contact resistance is required to limit electrical losses in power electronic devices operation [6]. It is necessary to find electrode materials to achieve good ohmic contact between the electrode layer and the H-diamond surface, by which superior diamond electronic devices could be
were fabricated on H-diamond surface with photolithography, electron beam evaporation (EB) and lift-off techniques, in which the spaces between the inner circular electrodes were 100 μm in diameter and the outer electrodes varied from 5 to 30 μm. The CTLM electrodes were composed by a 20-nm-thick Ni layer protected with a 200-nm-thick Au layer. The fabrication process is shown in Figure 1. After that, the sample was annealed in hydrogen atmosphere for 20 min from RT to 750 °C. Finally, the current-voltage (I-V) properties of Ni/H-diamond were measured by Agilent B1505A parameter analyzer after annealing at each temperature.

2. Materials and Methods

In this experiment, 3 × 3 × 0.5 mm³ dimension CVD-synthesized (001) single-crystal diamond was used as the substrate. First, the diamond substrate was cleaned in a mixture acid (H₂SO₄:HNO₃:HCLO₄ = 31.2:36:11.4) at 250 °C for 1h and then treated with mixed alkali (NH₄OH:H₂O₂:H₂O = 4:3:9) at 80 °C for 10 min to remove the non-diamond phase. After that, a 300 nm homoeptaxial diamond epilayer with hydrogen termination was deposited on the diamond substrate by commercial microwave plasma CVD system (AX5200 Seki Technotron Corp., Tokyo, Japan). During diamond growth, the pressure and temperature were 100 Torr, 900 °C and 1 kW, respectively. The ratio of CH₄/H₂ was 1% with a 500 sccm total flow rate of the reaction gas. The H-diamond surface was characterized by Atomic Force Microscope (AFM, INNOVA Bruker Corp., Billerica, MA, USA), X-Ray Diffraction (XRD, PANalytical The Analytical X-ray Company, Almelo, The Netherlands) and Raman spectra. The CTLM structures were fabricated on H-diamond surface with photolithography, electron beam evaporation (EB) and lift-off techniques, in which the spaces between the inner circular electrodes were 100 μm in diameter and the outer electrodes varied from 5 to 30 μm. The CTLM electrodes were composed by a 20-nm-thick Ni layer protected with a 200-nm-thick Au layer. The fabrication process is shown in Figure 1. After that, the sample was annealed in hydrogen atmosphere for 20 min from RT to 750 °C. Finally, the current-voltage (I-V) properties of Ni/H-diamond were measured by Agilent B1505A parameter analyzer after annealing at each temperature.

3. Results and Discussion

In order to characterize the quality of diamond substrate, AFM, Raman and XRD techniques were used. Full-width-at-half-maximum (FWHM) of XRD rocking curve was 0.0256 with a sharp peak centered at 59.174° (Standerr: 59.761°) which was operated in four-bounce Ge (2 2 0)-monochromated Cu-Kα with a 1-mm slit on the detector arm, as shown in Figure 2a. On the other hand, Figure 2b shows Raman spectra with 532 nm excitation laser and 0.4 cm⁻¹/pixel resolution in 20× objective.
lens; the narrow symmetrical sharp peak can be seen at 1331.4 cm$^{-1}$ (Stander: 1332 cm$^{-1}$), and the FWHM was measured as 4.48 cm$^{-1}$. Along with that, the Raman spectra also illustrates the existence of nitrogen impurities, which could reduce the current density [24,25]. Moreover, the root–mean–square (RMS) roughness of H-diamond was 0.651 nm within an area of 10 × 10 μm$^2$, showing a smooth surface. All data illustrate a high-quality diamond layer.

![Figure 2](image-url)

**Figure 2.** (a) XRD rocking curve of H-diamond. (b) Raman spectra of H-diamond. (c) Atomic force microscope (AFM) image of the diamond surface with an area of 10 × 10 μm$^2$.

Figure 3a,b show the I-V characteristics of Ni/H-diamond contact measured at as-deposited and different annealing temperatures. In Figure 3a, the I–V curves were non-linear, indicating that ohmic contact was not formed from RT to 600 °C. Nevertheless, when temperature is higher than 600 °C, linear I–V curves appear, indicating an obvious ohmic contact between Ni/H-diamond, shown in Figure 3b. Consequently, as shown in the graph, the absolute value of current increases as the annealing temperature increases. The data plotted in Figure 3a,b come from one circular ring with a spacing of 20 μm. The I-V characteristics of other circular rings also exhibit the same results, which were not shown here.

As an important parameter of electrical properties to evaluate ohmic contact, the specific contact resistance ($\rho_c$) can be calculated by Equation (1)

$$\rho_c = R_s \times L_T^2$$  \hspace{1cm} (1)

where $R_s$ is the sheet resistance of the H-diamond, $L_T$ is the transfer length. $R_s$ and $L_T$ can be obtained from the contact resistance ($R_c$) vs. the circular ring spacing curve in which $R_c$ is obtained from I–V properties [26].

Linear fitting of all the CTLM patterns about the average contact resistance versus the spacing $d$ after annealing in H$_2$ at 650, 700, 750 °C for 20 min is investigated (see Figure 3c). The linearity of contact resistance at the same temperature is very good and $R_c$ gradually declines as annealing temperature rises, which illustrates obvious promotion of the ohmic behavior. Average specific contact resistance...
(\(\rho_c\)) is exhibited in Figure 3d by measuring CTLM configurations at different annealing temperatures with a downtrend of \(\rho_c\), presenting good ohmic contact. After annealing at 600 °C, I–V curve began to become linear, which could be ascribed to the improvement in the interface performance by the annealing process. Besides, the formation of carbide might also be the reason for the formation of ohmic contact at higher temperature between Ni and H-diamond film [27]. Nickel carbide has good electrical conductivity. The increase in the proportion of nickel carbide will reduce the potential barrier between diamond and nickel, thus reducing the contact resistance. As temperature increases, \(\rho_c\) of ohmic contact shows a gradual decrease, which could be due to the formation of electrical active defects in carbide [6]. Consequently, the minimum \(\rho_c\) obtained was 1.19 \(\times\) 10\(^{-4}\) and 7.82 \(\times\) 10\(^{-5}\) Ω cm\(^2\), respectively, at 750 °C.

\[\begin{align*}
\text{(a)} & \quad \text{Current (A)} \\
\text{20 µm circular ring gap} & \quad \text{Voltage (V)} \\
\text{(b)} & \quad \text{Current (A)} \\
\text{20 µm circular ring gap} & \quad \text{Voltage (V)} \\
\text{(c)} & \quad \text{specific contact resistance (}\rho_c\text{)} \\
\text{anneal-650°C} & \quad \text{anneal-700°C} \\
\text{anneal-750°C} & \quad \text{vs. the circular ring spacing curve in which } \rho_c \text{ is obtained is exhibited in Figure 3d by measuring CTLM configurations at different annealing temperatures.} \\
\text{(d)} & \quad \text{specific contact resistance (}\rho_c\text{)} \\
\text{Temperature (°C)} & \quad \text{specific contact resistance (}\rho_c\text{)} \\
\end{align*}\]

**Figure 3.** I–V characteristics of as-deposited and annealed Ni/H-diamond contact of CTLM configuration: (a) before and after annealing in H\(_2\) from 100 to 600 °C for 20 min; (b) before and after annealing in H\(_2\) at 650, 700, 750 °C for 20 min. (c) Linear fitting of the average contact resistance after annealing in H\(_2\) at 650, 700, 750 °C for 20 min. (d) Relationship between temperature and average specific contact resistance (\(\bar{\rho}_c\)) with annealing temperature from 650 to 750 °C in H\(_2\) for 20 min at each temperature.

4. Conclusions

Ohmic contact of nickel on hydrogen-terminated single-crystal diamond film was investigated with an annealing temperature ranging from room temperature to 750 °C in hydrogen atmosphere. After annealing at 650 °C in the H\(_2\) ambient for 20 min, I–V curves appear, indicating linear behavior, which showed a conversion from Schottky to Ohmic contact. The \(\bar{\rho}_c\) of ohmic contact decreases with the increase in temperature. The measurement results of CTLM at 750 °C indicate a best ohmic contact value of 7.82 \(\times\) 10\(^{-5}\) Ω cm\(^2\) and the minimum \(\bar{\rho}_c\) obtained was 1.19 \(\times\) 10\(^{-4}\) Ω cm\(^2\), exhibiting an excellent ohmic contact. Our results contribute to the low specific contact resistance Ohmic contact of Ni with H-diamond, showing great potential for the fabrication of Diamond-based power devices.
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