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ABSTRACT

n-Type (phosphorus-doped) diamond is a promising material for diamond-based electronic devices. However, realizing good ohmic contacts for phosphorus-doped diamonds limits their applications. Thus, the search for non-conventional ohmic contacts has become a hot topic for many researchers. In this work, nanocarbon ohmic electrodes with enhanced carrier collection efficiency were deposited by coaxial arc plasma deposition. The fabricated nanocarbon ohmic electrodes were extensively examined in terms of specific contact resistance and corrosion resistance. The circular transmission line model theory was used to estimate the charge collection efficiency of the nanocarbon ohmic electrodes in terms of specific contact resistance at a specific voltage range (5–10 V); they exhibited a specific contact resistance of $1 \times 10^{-3} \Omega\text{cm}^2$. The result revealed one order reduction in the specific contact resistance and, consequently, a potential drop at the diamond/electrode interface compared to the conventional Ti electrodes. Moreover, the fabricated nanocarbon electrodes exhibited high mechanical adhesion and chemical inertness over repeated acid treatments. In device applications, the nanocarbon electrodes were evaluated for Ni/n-type diamond Schottky diodes, and they exhibited nearly one order enhancement in the rectification ratio and a fast charge collection at lower biasing voltages.

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Diamond exhibits matchless material properties on thermal conductivity, hardness, breakdown voltage, and carrier mobilities, which makes it a potential candidate for high-temperature power electronics, quantum sensing, vacuum switches, etc.^{1–3} The efficient n-/p-type doping mechanisms and surface reconstruction using H radicals have expanded the versatility to advanced device realizations such as diamond FETs, cold cathode emitters, bipolar

devices, and magnetic sensors.^{4–6} The formation of stable ohmic contacts is an essential aspect of diamond-based device fabrication.⁷ Indeed, the ohmic electrode should be mechanically adhesive, thermally stable, and resistant to corrosive environments for its maximal potential.^{8,9} For p-type (boron-doped) diamonds, carbide-forming Ti-based ohmic electrodes have achieved a specific contact resistance of $\sim 10^{-7} \Omega\text{cm}^2$.¹⁰ On the contrary, the realization of

n-type (phosphorus-doped) diamond-based devices is facing challenges due to the unavailability of practical ohmic contacts that exhibit a comparable specific contact resistance.^{11,12} The Fermi level pinning around 4 eV from the conduction band at the n-type diamond/metal interfaces rules out the candidate metallic ohmic contact choices based on the Schottky barrier height engineering.¹³ Annealed Ti-based ohmic electrodes are widely used for diamond-based electronics due to carbide formation at the interface. Apart from the metal/diamond interfaces, forming ohmic electrodes on shallow electrically active states at the phosphorus-doped diamond film surface induced by ion implantation and thermal graphitization is extensively studied.^{14–16} Even though they exhibit better ohmic behavior than the conventional Ti-based ohmic electrodes, they are not widely implemented in device fabrication owing to the surface lattice deformation, asymmetry of the defect layer, and the surface roughening induced during the contact formation.

This work demonstrates an efficient, mechanically stable, and corrosion-resistant ohmic contact formation with less specific contact resistance on phosphorus-doped diamond films through nanocarbon electrodes fabricated by coaxial arc plasma deposition (CAPD). The comparative electrical characterizations of the nanocarbon electrodes with conventional Ti electrodes were performed using the circular transmission line model (cTLM) theory.

Heavily phosphorus-doped ($\sim 2 \times 10^{20} \text{ cm}^{-3}$) diamond films were homoepitaxially grown on high-pressure high temperature (HPHT) synthetic type 1-b (111) $2 \times 2 \text{ mm}^2$ diamond substrates (Sumimoto Electric Industries) by using a microwave plasma-enhanced chemical vapor deposition system (Seki-ASTeX), and the impurity concentration was characterized by secondary ion mass spectroscopy.^{17,18} From temperature-dependent I–V measurements and processing through the cTLM theory, a hoping regime activation energy of 57 meV is yielded for the diamond film, which is in reasonable agreement with the literature.¹⁷ A pre-electrode deposition surface cleaning of the diamond film has been performed in an $\text{H}_2\text{SO}_4 + \text{HNO}_3$ (3:1) solution at 250°C for 50 min. A homogeneous nanocarbon layer ($\sim 230 \text{ nm}$) was directly fabricated on the diamond surface through CAPD at 550°C . A coaxial arc plasma apparatus ejects highly energetic and dense carbon species onto the substrate. The system employs the cathodic discharge of pure-graphite targets (99.99% pure) coaxially mounted by an anodic cylinder. The base pressure inside the chamber was maintained below 10^{-4} Pa throughout the deposition. The pulsed cathodic arc discharge was realized by an arc plasma gun with a $720 \mu\text{F}$ bank capacitor. The pulse frequency and pulse trigger voltage are calibrated to be 5 Hz and 100 V, respectively. More details about CAPD including schematic and target–substrate distance can be found in the literature.^{19,20} The deposition was carried out at a substrate temperature of 550°C to promote adhesion at the interface since the room temperature deposition shows reduced adhesion. The resistivity of the nanocarbon film is found to be highly dependent on the substrate temperature. A nanocarbon film fabricated at a substrate temperature of 300°C showed a nearly three order increase in the bulk resistivity than that of the same fabricated at 550°C , which is not practically suitable for ohmic contact formation. The low resistivity of the nanocarbon film at higher substrate temperatures is expected to be from the thermal-assisted sp^2 phase formation.²¹

The bulk resistivity of the nanocarbon film is estimated to be $4 \times 10^{-3} \Omega\text{cm}$ through Hall-effect measurements. In addition to the advantage of ohmic contact formation, the nanocarbon film exhibited exceptional corrosion resistance (acidic inertness). The interfacial stability and corrosion resistance of the nanocarbon/n+ diamond structure was examined against the $\text{H}_2\text{SO}_4 + \text{HNO}_3$ (3:1) acid treatment at 250°C . Even though the surface morphology changed, the nanocarbon electrodes were electrically durable for more than ten acid treatment sessions (a total of 500 min) with a modest change in the response current and the linearity of the I–V curve. The complete removal of the nanocarbon electrodes was achieved by 13 acid cleaning sessions. A more deep investigation of the nanocarbon's nature was acquired through Raman spectroscopic analysis.

In Fig. 1, the Raman spectra divulge two prominent peaks located at 1350 and 1570 cm^{-1} ; they are corresponding to D and G bands, respectively, which are generally observed for nanocarbon films.²² The G band is corresponding to the in-plane stretching mode of sp^2 bonded carbon atoms in the film, and the D band originates from the disorder-induced optical phonons in the sp^2 matrix, suggesting that the composite structure of the nanocarbon ohmic electrode contains a large amount of disoriented sp^2 carbon matrix, which is expected as the reason for the ohmic property of nanocarbon electrodes as in reference with the typical graphitic electrodes.

For a qualitative comparison of the charge collection efficiency of nanocarbon electrodes with conventional Ti electrodes, circular electrodes with varying inter-electrode distances ($4\text{--}28 \mu\text{m}$) were fabricated using the standard photolithography lift-off technique and inductively coupled plasma-reactive ion etching (ICP-RIE). The conventional Ti/Mo/Au electrodes were deposited using e-beam evaporation and annealed at 450°C for 15 min.

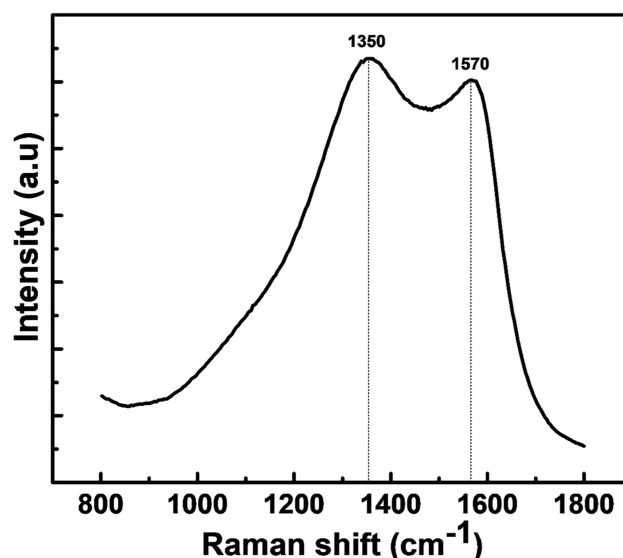


FIG. 1. Visible Raman spectrum of nanocarbon film (excitation wavelength = 532 nm).

According to Fig. 2(a), the n+ diamond film showed a clear inter-electrode spacing (d) dependent ohmic behavior through nanocarbon ohmic electrodes with response current changing respective to d . The linearity of the response current has improved with active region current supplementation for nanocarbon electrodes compared with the conventional Ti electrodes as shown in Fig. 2(b). According to the cTLM theory, the total resistance R_T between the electrodes and the inter-electrode distance d finds a linear relationship (when $r_1 + r_2 = \text{constant}$) according to the following equation:¹⁵

$$R_T = \frac{R_s}{2\pi} \left[L_T \left(\frac{1}{r_1} + \frac{1}{r_2} \right) + \ln \left(\frac{r_2}{r_1} \right) \right] \cong \frac{(2L_T + d)}{2\pi r_0} R_s, \quad (1)$$

where r_1 and r_2 are the circular electrode radii ($r_1 < r_2$), with $r_0 = r_1 + d/2 = r_2 - d/2$; R_s is the sheet resistance of the n+ diamond film; and L_T is the transfer length of the electrode. As Transfer length is the minimum pathway of the carriers through the diamond/electrode interface before the complete interface crossing, and it is estimated from the d-intercept ($R_T = 0$) of inter-electrode total resistance (R_T) vs inter-electrode spacing (d) graph.²³ The specific contact resistance ρ_c (Ωcm^2) can be expressed in terms of sheet resistance R_s and L_T as²³

$$\rho_c = R_s \times L_T^2. \quad (2)$$

The inter-electrode resistance changes from 5 to 36 k Ω for nanocarbon electrodes and from 8 to 40 k Ω for conventional Ti/Mo/Au electrodes as shown in Fig. 3. R_s was obtained to be 1.42 and 1.45 M Ω by using nanocarbon and Ti/Mo/Au electrodes, respectively. This increased sheet resistance, when compared with the previous reports, can be attributed to the lesser thickness of the phosphorus-doped layer (300 nm).^{15,24} The resistivity of the n+ diamond film was calculated to be 50 Ωcm from the cTLM data, which, indeed, shows good agreement with heavily phosphorus-doped films.¹⁷ The room temperature specific contact resistance ρ_c has shown an active reduction to the value

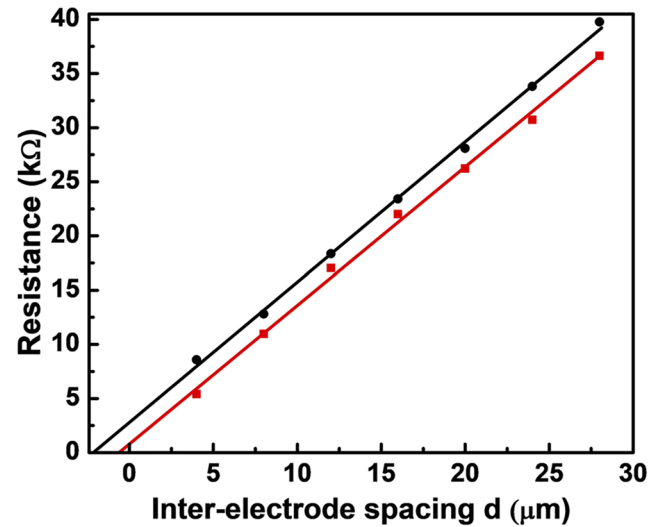


FIG. 3. Dependence of inter-electrode resistance on inter-electrode spacing for nanocarbon (red) and Ti (black).

$1 \times 10^{-3} \Omega\text{cm}^2$ for nanocarbon electrodes to that of Ti/Mo/Au electrodes ($1 \times 10^{-2} \Omega\text{cm}^2$) at the active region (5–10 V). This declination in ρ_c shows an increased carrier collection efficiency for nanocarbon electrodes. For Ti electrodes, the specific contact resistance obtained in this study shows less correlation with the previous report as the active voltage region for differential resistance extraction has been limited below 10 V.^{17,18,24} This discrepancy is explained by using a constant current model in the cTLM theory.

The constant current cTLM theory analyses the n+ diamond/ohmic electrode interfaces based on the double Schottky barriers formed at the reverse and forward regions during the current flow through the electrodes.¹⁵ According to the double Schottky structure at the interface, the carrier crossing the interface can be due to a combined mechanism of thermionic emission interface crossing and field emission interface crossing. The thermionic emission current (I_{TE}) indicates the carrier crossing by nullifying the barrier after attaining certain energy, i.e., the carriers jumping over the potential barrier. However, due to the narrow Schottky barriers at the interface, the tunneling probability through the barrier will change accordingly with the potential drop V_e at the interface. Due to this changing V_e , the barrier tunneling current (I_{BT}) will also increase for a higher applied voltage at the electrodes. The total applied voltage (V_c) at the electrodes mentioned here will be distributed at the interface as V_e and at the diamond bulk as V_{bulk} ($V_c = V_e + V_{\text{bulk}}$).

For a current I ($I = I_{TE} + I_{BT}$) to flow between the electrodes, the thermionic emission current I_{TE} largely depends on the external voltage (V_c) applied across the electrodes, while the barrier tunneling current I_{BT} changes with (V_e) at the diamond/electrode interfaces, thus introducing a variable contact resistance R_c at the interfaces,

$$R_T(V_e, d) = R_c(I) + R_{\text{bulk}}(d), \quad (3)$$

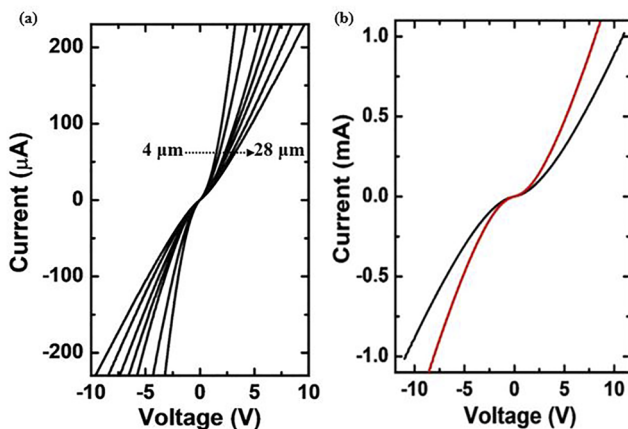


FIG. 2. (a) Typical I–V characteristics of n+ diamond film through nanocarbon electrodes with different electrode spacing d (4–28 μm). (b) Comparison of nanocarbon (red) and Ti (black) electrodes on n+ diamond film for $d = 4 \mu\text{m}$.

where R_{bulk} is the bulk resistance of the n+ diamond film with dependence as $(d/2\pi r_0)R_S$. Contrary to the carrier collection efficiency, an accurate determination of ρ_c demands the total resistance to be solely a function of inter-electrode spacing d . Therefore, in this model, the tunneling probability at the interface is made the same for every electrode spacing by considering a constant current I_0 , i.e., making V_e at the interfaces constant for applied voltage V_c between the electrodes. The voltage at both diamond/electrode interface can be expressed as

$$V_e = R_c(I_0)I_0. \quad (4)$$

We analyzed the interface behavior from $I_0 = 100 \mu\text{A}$ – $350 \mu\text{A}$ with steps of $50 \mu\text{A}$. Figure 4(b) shows the R_T vs d plotting for a constant current $I_0 = 150 \mu\text{A}$.

In the constant current model cTLM approach, the interfacial potential drop (V_e) for a constant current I_0 shows an active declination for nanocarbon electrodes indicating a reduced contact resistance R_c than the conventional Ti electrodes. Contrary

to boron-doped diamonds, ρ_c is mainly dependent on the voltage (V_e) at the diamond/electrode interfaces. From Fig. 4, for a V_e of 1.8 V, nanocarbon and Ti electrodes show a ρ_c of $\sim 4 \times 10^{-1} \Omega\text{cm}^2$ and $\sim 9 \times 10^{-1} \Omega\text{cm}^2$, respectively. For all I_0 , the n+ diamond film showed a constant sheet resistance ($\sim 1.5 \text{ M}\Omega$). This conservation of the sheet resistance might be due to the proportional change in V_e concerning the inter-electrode spacing in the differential resistance-based cTLM approach. Through this approach, the discrepancy observed for Ti electrodes in the previous model holds a detailed explanation as to the specific contact resistance ρ_c changes with the voltage (V_e) at the diamond/electrode interfaces. The Ti contact scheme was found to be further improved from the literature.^{15,17,24} As detailed from the different cTLM approaches to the contact behavior, the nanocarbon electrodes showed enhanced carrier collection efficiency and room temperature specific contact resistance ρ_c than conventional Ti ohmic electrodes.

Device fabrication is an important aspect of ohmic contact research. Nickel/n-type diamond Schottky diodes were fabricated

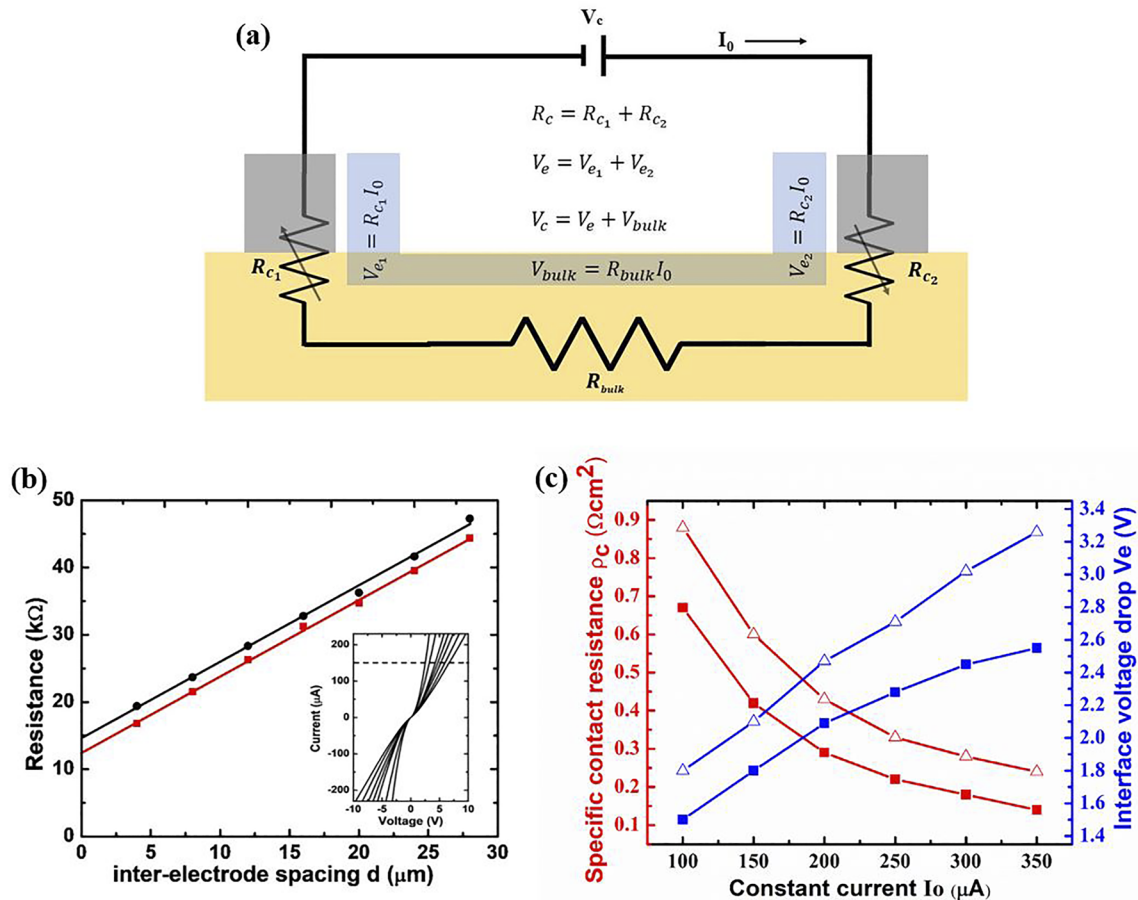


FIG. 4. (a) Schematic of constant current model. (b) R_T vs d plotting $I_0 = 150 \mu\text{A}$. The red line indicates nanocarbon, whereas the black line indicates Ti/Mo/Au. Inset shows the dashed line at $I_0 = 150 \mu\text{A}$ used for total resistance extraction. (c) Double Y axes dependence plot of V_e and ρ_c on current (I_0) flowing between the electrodes. Solid square depicts nanocarbon, whereas hollow triangle depicts Ti.

on a lightly phosphorus-doped ($\sim 10^{17} \text{ cm}^{-3}$) diamond film. The I–V mapping of the Schottky diode was obtained using the concerned ohmic electrodes with the same contact area by means of a micro-prober high resistance electrometer station (Keysight B2985A) at 300 K. The Ti electrodes were annealed at 450°C for 15 min.

In Fig. 5, Ni/n-type Schottky diode exhibits a large rectification ratio as well as fast charge collection achieved at lower biasing voltage for nanocarbon electrodes than conventional Ti electrodes. As the distance between the Schottky and ohmic contact and the contact area of the ohmic electrodes are kept constant, the difference in the turn-ON voltage and rectification ratio for concerned ohmic electrodes solely can be from the improvement of ρ_c for the nanocarbon electrodes. This enhancement in the forward current for nanocarbon electrodes actively suggests the same for lightly phosphorus-doped diamond base electronic applications.

In summary, nanocarbon ohmic electrodes fabricated by CAPD on heavily phosphorus-doped diamond films were studied using the cTLM theory. The carrier collection efficiency has been characterized in terms of room temperature specific contact resistance ρ_c and obtained one order declination for nanocarbon electrodes ($1 \times 10^{-3} \Omega\text{cm}^2$) than conventional Ti electrodes ($1 \times 10^{-2} \Omega\text{cm}^2$) at an intermediate voltage range (5–10 V). Both the electrodes deviate from ideal ohmic behavior. For a detailed study, a constant current model has been applied in the cTLM theory to see the dependence of ρ_c on the combined voltage V_e at both diamond/electrode interfaces. The low V_e value for a constant current I_0 in the case of nanocarbon electrodes indicates a reduced contact resistance R_c . At a constant V_e , the specific contact resistance ρ_c has improved for nanocarbon electrodes. The nanocarbon electrodes exhibit good corrosion resistance and adhesion. They have been electrically durable up to more than ten acid treatment sessions, each of 50 min [$\text{H}_2\text{SO}_4 + \text{HNO}_3$ (3:1) at 250°C]. This shows a tightly bonded interface. The enhanced ohmic behavior is expected to originate from the tightly bonded sp^2

phases in the nanocarbon film. A detailed study about interfacial stability, corrosion resistance, and conduction mechanism will be reported later.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Sreenath Mylo Valappil: Conceptualization (equal); Data curation (lead); Investigation (lead); Methodology (lead); Software (lead); Writing – original draft (lead); Writing – review & editing (lead). **Shinya Ohmagari:** Conceptualization (equal); Funding acquisition (equal); Project administration (equal); Resources (equal); Supervision (equal); Writing – review & editing (supporting). **Abdelrahman Zkria:** Conceptualization (equal); Project administration (supporting); Writing – review & editing (supporting). **Phongsaphak Sittimart:** Investigation (supporting). **Eslam Abubakr:** Investigation (supporting); Writing – review & editing (supporting). **Hiromitsu Kato:** Resources (equal); Investigation (equal). **Tsuyoshi Yoshitake:** Conceptualization (equal); Funding acquisition (equal); Project administration (equal); Supervision (lead).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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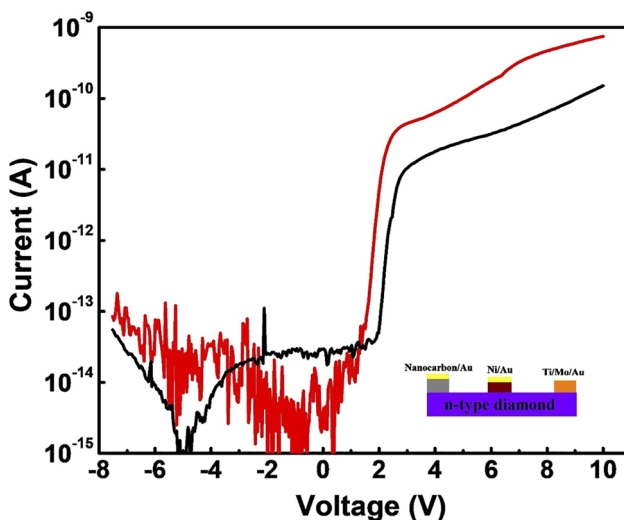


FIG. 5. Typical I–V mapping of Ni/n-type diamond Schottky diode with respective ohmic electrodes of nanocarbon (red) and Ti/Mo/Au (black).

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