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Influence of thickness and porous structure of SiC layers on the electrical properties of Pt/SiC-pSi and Pd/SiC-pSi Schottky diodes for gas sensing purposes

Karima Bourenane [a,*], Aissa Keffous [a], Ghania Nezzal [b], Ahmed Bourenane [c], Yacine Boukennous [a], Assia Boukezzata [b]

[a] *Unité de Développement de la Technologie du Silicium, 02 Bd Frantz Fanon, BP 140 Alger 7 Merveilles, Alger-Algérie, Algeria*
[b] *Université des Sciences et de la Technologie Houari Boumediene, Faculté de Génie Chimique, Algeria*
[c] *Centre de Recherche Nucléaire de Birine, BP 180 Ain Oussera, Algeria*

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Abstract

The aim of this work was to study the influence of the thickness and porous structure of silicon carbide (PSC) layers on the electrical properties of Schottky diodes for gas sensing purposes by using a platinum (Pt) or a palladium (Pd) layer deposited on non-porous silicon carbide (SiC) and porous SiC (PSC) layers. The Schottky diodes were used for the first time for H_2 and hydrocarbon (C_2H_6) gas sensing. The non-porous and porous SiC layers were realized on a p-type silicon (Si(1 0 0)) substrate by pulsed laser deposition using a KrF laser (248 nm) and thermal deposition of a thin Pt and Pd layer. The porous structure of the SiC layer deposited was developed by an electrochemical (anodization) method. The properties of the porous SiC layers formed by this method were investigated by scanning electron microscopy (SEM). The electrical measurements were made at room temperature (295 K) in an air ambience using a cryostat chamber. The effect of the porous surface structure and the thickness of the SiC layer was investigated by evaluating electrical parameters such as the ideality factor (n), barrier height (ϕ_{Bp}) and series resistance (R_s). The thickness of the porous layer significantly affects the electrical properties of the Schottky diodes. Analysis of current–voltage (*I–V*) characteristics showed that the forward current might be described by a classical thermal emission theory. The ideality factor determined by the *I–V* characteristics was found to be dependent on the SiC thickness. For a thin SiC layer (0.16 μm) n was around 1.293 with a barrier height 0.847 eV, while for a thick layer (1.6 μm), n and ϕ_{Bp} were 1.110 and 0.812 eV, respectively, for both Pt/SiC-pSi and Pd/SiC-pSi. The low value of series resistance obtained using Cheung's method clearly indicated the high performance of the Schottky diode for large SiC layer thickness. This effect showed the uniformity of the SiC layer. *C–V* analysis revealed a decrease in the capacitance with increasing voltage. This decrease was less for a thin SiC layer (0.16 μm) than a thick SiC layer (1.6 μm), clearly indicating the role of the SiC thickness in the determining sensor capacitance values. Finally, high sensitivity ($\Delta I/I = 90\%$) and selectivity of the sensors were reached at low voltages below 1 V, by using the PSC layer with catalytic metals of Pt and Pd.
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Keywords: Silicon carbide; Schottky diodes; Platinum; Palladium; Gas sensors

1. Introduction

Crystalline silicon carbide thin films have attracted much attention in recent years because of their potential application in many kinds of optoelectronic devices, such as solar cells, image sensors and photodiodes [1–5]. Silicon carbide is an appropriate material for the fabrication of high temperature, high frequency and high power electronic devices, due to its wide band gap (2.3–3.2 eV), high breakdown field (>2×10^6 V cm^{-1}), high thermal conductivity (5–7 W cm^{-1} K^{-1}), and high electron saturation velocity (2×10^7 cm s^{-1}) [6]. In this paper, the growth of a thin SiC layer by pulsed laser deposition (PLD) using a 6H–SiC hot pressed wafer as a target and p-type Si(1 0 0) as a substrate is characterized by different structural and optical measurements. Pulsed laser ablation has become one of the most powerful methods to obtain a wide class of materials in thin film

* Corresponding author. Tel.: +213 21 43 35 11; fax: +213 21 43 24 88.
E-mail address: bourima2003@yahoo.fr (K. Bourenane).

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form. Typical examples are complex ceramic materials such as high-temperature superconductors and certain magnetic materials [7,8]. The process and consequently the properties of the deposited films are strongly dependent on many parameters: the laser fluency and wavelength, the structural and chemical composition of the target material, the chamber pressure and the chemical composition of the buffer gas, the substrate temperature, and the distance between the target and the substrate. The first gas sensor was developed by Lundström et al. [9]. It was a MOS structure using silicon as a substrate and could only operate at temperatures below 150 °C. In order to develop gas sensors that can operate in the high temperature environment of aerospace and automobile applications, new technologies were created. One new technology is based on the development of high temperature semiconductors, especially silicon carbide (SiC) which allows to the sensor to operate potentially up to 1000 °C [10–13].

In this work, a novel metal/PSC Schottky diode for gas sensing is fabricated to increase sensitivity and to select different gases such as hydrogen (H_2) and hydrocarbon (C_2H_6). This paper will first explain the experimental method of growing crystalline SiC layers, formation of porous SiC layers and electrical characterization of the Schottky diodes with structures SiC-pSi(1 0 0) and PSC-pSi(1 0 0), using two different metals (Pt and Pd) and SiC layers of different thickness (0.16 and 1.6 μm). The excellent hydrogen (H_2) sensing characteristics are due to the formation of porous SiC layers in comparison with the SiC layer with a smooth surface.

2. Experimental procedure

p-Type porous structure silicon carbide (PSC) layers were fabricated by anodization in HF on a 0.16 μm thin and 1.6 μm thick SiC layer, deposited onto a p-type Si(1 0 0) substrate by pulsed laser deposition (PLD), using a hot pressed p-type 6H–SiC as a target. In order to facilitate the electrochemical etching of the substrate, an ethylene glycol electrolyte was been added to the solution and a thin metallic film of aluminium (Al) was deposited onto a SiC layer prior to anodization.

The p-type Si(1 0 0) substrate with a resistivity of 6 Ω cm was mounted on a plate. The plate was attached on top of a rotating cylinder and heated in situ to 500 °C using halogen lamps mounted on a fixed water-cooled reflector. The target is a hot pressed (Goodfellow) granular SiC target. The interaction of the laser beam with the target produced a highly oriented material stream, usually ejected normal to the target surface, which was deposited onto the substrate positioned in front of the target. The reaction chamber vacuum pressure was fixed at 10^{-6} Torr. The laser pulses were produced by a Lambda Physik Compex 205 KrF laser excimer. The most important parameters were: wavelength $\lambda = 248$ nm, maximum pulse energy $E_{max} = 600$ mJ and pulse duration $\tau = 25$ ns. The thickness of the SiC layers deposited onto p-Si substrates value was obtained to be 0.16 and 1.6 μm by a Tencor 250 profilometer, and the electrical resistivity was measured to be 100 and 564 Ω cm, respectively, by a four probe method. After the growth of the SiC layer, an ohmic contact layer of Al with a thickness of 490 nm was deposited by thermal evaporation on the backside. Development of porous structure in the SiC layers was performed by an electrochemical (anodization) method. Before this step, the samples were provided with an Al contact on the front side and were annealed at 650 °C for 1 h in air ambience. The annealing step reduced the electrical resistivity of the SiC substrate. The electrochemical setup was a standard three electrode configuration with SiC/pSi as a working electrode, a Pt sheet as a counter electrode and a saturated calomel electrode as a reference. The electrochemical process was performed by using a potentiostat–galvanostat model EG&G PAR 362 [14,15]. The morphological analysis of the porous structure was made by using a SEM Philips 505.

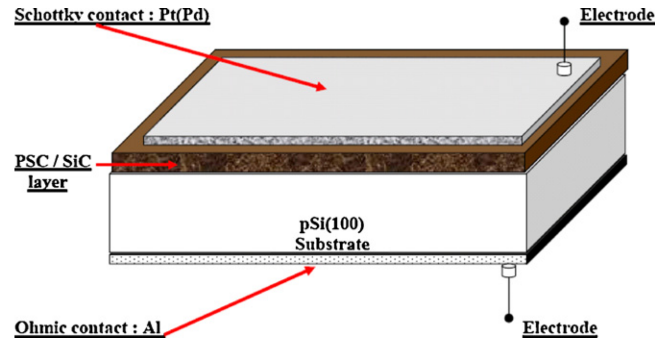

Fig. 1. Cross-sectional view of Pt (Pd)/SiC-pSi/Al Schottky diode.

After formation of the ohmic contact, the samples were placed into a deposition chamber in order to evaporate a thin layer of platinum (99.9% purity) and palladium (99.9% purity) on the SiC-pSi to form Schottky contact. The thickness of the platinum and palladium layer as measured by a Tencor 250 profilometer was 4 and 250 nm, respectively. The layers served as a catalytic metal. The current–voltage measurements were performed at room temperature using a Keithley voltage/current source unit model 237 while the capacitance–voltage characteristics were measured by an Agilent 4278 capacitance meter at 1 MHz.

Figs. 1 and 2 illustrate a schematic cross-sectional view of the Pt(Pd)/SiC-pSi/Al Schottky diode structure and the experimental setup for *I*–*V* and *C*–*V* measurements, respectively.

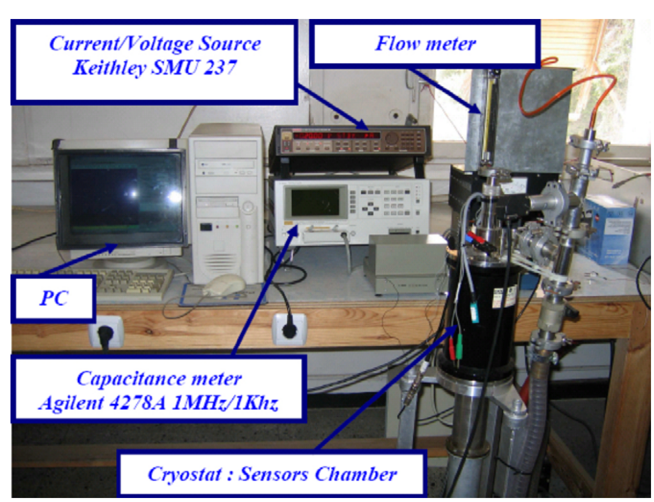

Fig. 2. Automatic of acquirement *I*–*V* and *C*–*V* measurements using a cryostat sensor gas chamber.

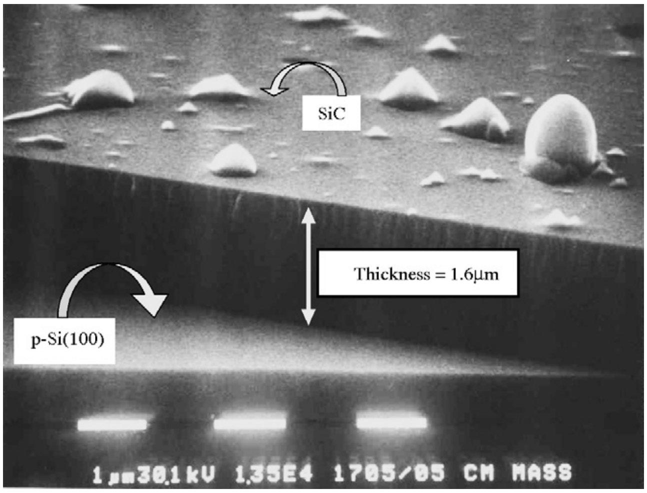

Fig. 3. SEM image of a 1.6 μm thick SiC layer deposited onto pSi(1 0 0).

3. Results and discussion

3.1. Morphological observation

Fig. 3 shows a SEM view of a thin (0.16 μm) SiC layer, revealing a smooth and a planar surface with a low defect density due to the high flux of matter. Fig. 4a and b shows a plan view and a cross-sectional SEM image of the anodized 1.6 μm thick SiC layer. This sample was anodized at a constant current density of 10 mA/cm^2 for an anodization time of 60 s. Note that the surface appears more porosified with formation of nanocrystallites as correlated with the cross-sectional image (Fig. 4b) and that the average pore size is around 2 μm. Note that it is difficult to precisely measure the pore diameter.

Fig. 5a and b shows a plan view and a cross-sectional SEM image of the 0.16 μm SiC layer anodized at a constant current density of 10 mA/cm^2 for an anodization time of 10 s. This current density and anodization time are the optimum etching conditions. The surface appears porosified with formation of spherical structures under the layer, as shown in Fig. 5b. The formation of a porous layer increases the sensitivity of gas detection compared to a smooth surface and is the main objective of this work [16].

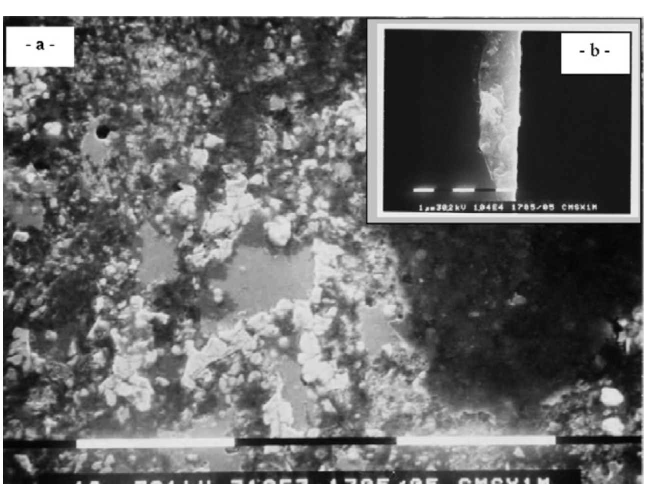

Fig. 4. SEM images of a 1.6 μm thick SiC deposited onto pSi(1 0 0) after anodization in 50% HF/50% ethylene glycol at J_{anod} = 10 mA/cm^2 for 60 s. (a) Plan view and (b) cross-sectional image.

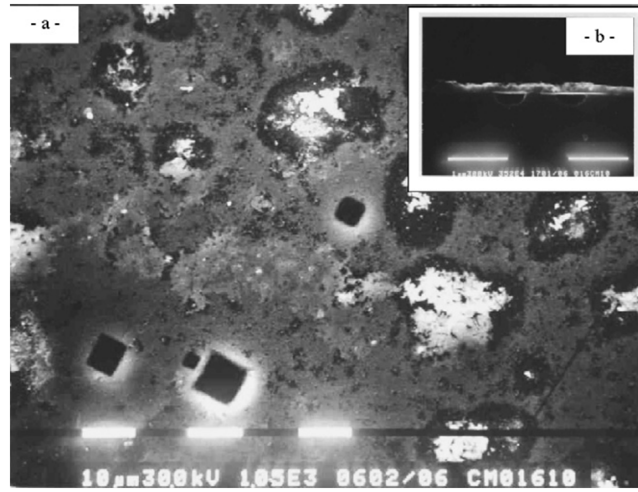

Fig. 5. SEM images of a 0.16 μm thin SiC layer deposited onto pSi(1 0 0) after anodization in 50% HF/50% ethylene glycol at J_{anod} = 10 mA/cm^2 for 10 s. (a) Plan view and (b) cross-sectional image.

3.2. Electrical measurements

3.2.1. Current–voltage

The thermionic emission theory for a bias voltage, V, greater than $3kT/q$ is valid for the analysis of electrical characteristics of the Schottky barrier structures of Pt/SiC-pSi and Pd/SiC-pSi. The junction current can be expressed by [17,18]:

$$I = I_s\left[\exp\left(\frac{qV}{nkT}\right) - 1\right] \quad (1)$$

where I_s is the saturation current given by:

$$I_s = aA^{**}T^2\exp\left(\frac{-q\phi_{Bp}}{kT}\right) \quad (2)$$

I_s is derived from the straight line of ln(I) = f(V) plot at V = 0, q the electron charge (1.6×10^{-19} C), V the forward applied voltage, a the active area (0.20 cm^2), T the measurement temperature (295 K), k the Boltzmann constant (1.38×10^{-23} J/K), A^{**} the effective Richardson constant for p-type 6H–SiC (76 A cm^{-2} K^{-2}), ϕ_{Bp} is the barrier height for p-type SiC.

The ideality factor (n) of the Schottky diode can be written as:

$$n = \frac{q}{kT}\cdot\frac{dV}{d\ln(I)} \quad (3)$$

The barrier height ϕ_{Bp} is determined from the extrapolated I_s and is given by the relation:

$$q\phi_{Bp} = kT\ \ln\left[\frac{aA^{**}T^2}{I_s}\right] \quad (4)$$

Measurements of current–voltage (I–V) characteristics were made at room temperature (295 K) using a Keithley model

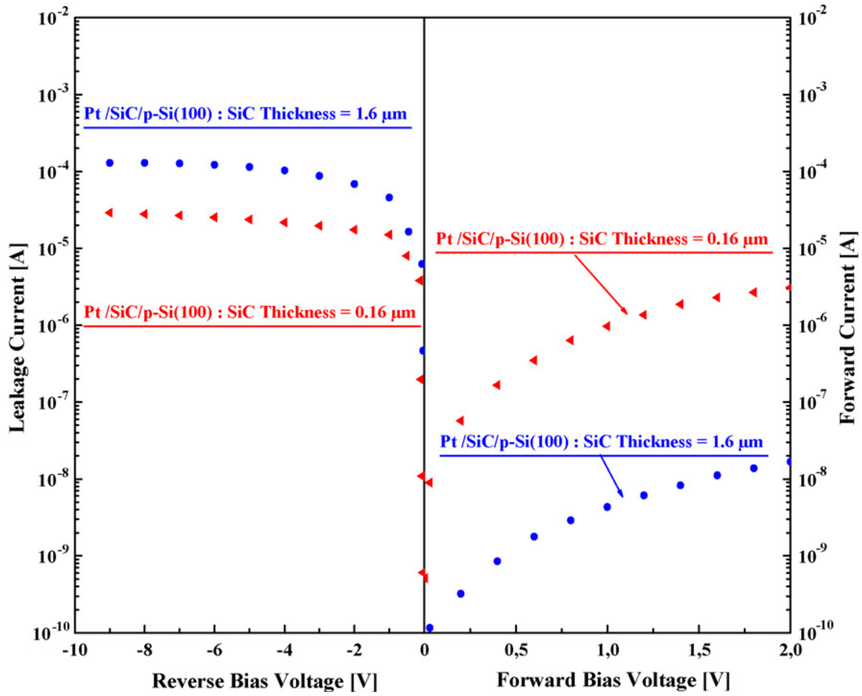

Fig. 6. Forward and reverse *I–V* characteristics of the Pt/SiC-pSi Schottky diode as a function of SiC thickness.

237 voltage/current source. The barrier height ϕ_{Bp} and the ideality factor n were determined from the intercept and the slope of the linear part of the forward bias voltage at exponential region ln(I) versus V plots, respectively. Typical current–voltage (I–V) characteristics of the Pt/SiC-pSi and Pd/SiC-pSi Schottky diodes under forward bias voltage are shown in Figs. 6 and 7, respectively.

The results show excellent junction parameters with a value of ideality factor close to unity, a high height barrier and a small value of series resistance for both the Pt and Pd Schottky contacts with a thick SiC layer (Table 1).

The parameters n, ϕ_{Bp} and R_s experimentally obtained are reported in Table 1. The values of n (>1) indicate that the diodes obey the metal–interface–semiconductor (MIS) configuration rather than an ideal Schottky diode. The interface oxide layer may be formed during either surface preparation and metal evaporation or thermal annealing during ohmic contact forming. The surface of the SiC layer is inevitably covered by a very thin oxide layer around 3 nm thick corresponding to native oxide, if evaporation of metal is carried out at a pressure of around 2×10^{-5} Torr. A value of $n > 1.320$ usually leads to a larger interfacial thickness than 3 nm [19,20].

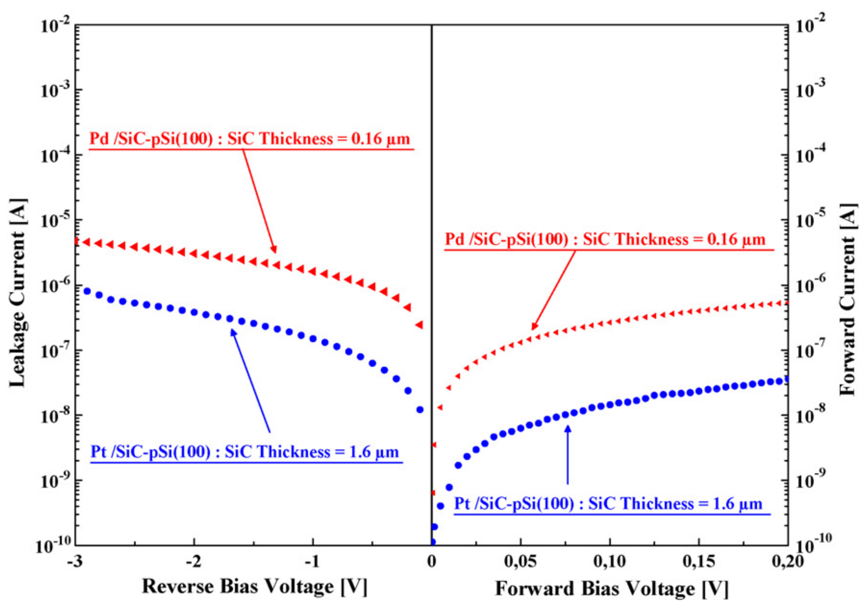

Fig. 7. Forward and reverse *I–V* characteristics of the Pd/SiC-pSi Schottky diode as a function of SiC thickness.

Table 1
Electrical parameters deduced by *I–V* measurements

Schottky diode	n	Built in-voltage V_{bi} (V)	Barrier height ϕ_{Bp} (eV)	R_s (kΩ)
Pt/0.16 μm SiC-pSi (unetched)	1.262	0.465	0.858	847.29
Pt/1.6 μm SiC-pSi (unetched)	1.195	0.582	0.735	1.67
Pt/PSC (0.16 μm SiC)-pSi (50% HF/50% ETG − t = 15 s)	1.065	0.420	0.857	0.17
Pt/PSC (1.6 μm SiC)-pSi (50% HF/50% ETG − t = 2 min)	1.505	2.807	0.935	1.55
Pd/0.16 μm SiC-pSi (unetched)	1.325	0.972	0.798	24.58
Pd/1.6 μm SiC-pSi (unetched)	1.026	0.795	0.890	1.44
Pd/PSC (0.16 μm SiC)-pSi (50% HF/50% ETG − t = 10 s)	1.194	0.137	0.669	0.05
Pd/PSC (1.6 μm SiC)-pSi (50% HF/50% ETG − t = 2 min)	1.344	0.754	0.792	0.71

The forward *I–V* characteristics are linear in the semi-logarithmic scale but deviate from linearity due to the effect of interface state and series resistance, associated with the layer (SiC), the substrate (Si) and the ohmic back contact, when the applied voltage is sufficiently large [20]. The voltage across the diode can be expressed in terms of the total voltage drop across the diode and the resistance R_s. This is accounted for by replacing the voltage V by $V–IR_s$ in Eq. (1).

$$\frac{\mathrm{d}V}{\mathrm{d}\ln(I)} = IR_s + \frac{nkT}{q} \quad (5)$$

Figs. 8 and 9 show dV/dln(I) versus I for the Pt and Pd Schottky diodes with the two values of SiC thickness. The low value of series resistance indicating clearly the high performance of the Schottky diodes.

The values of electrical parameters determined by *I–V* measurements indicate that the ideality factor n was very high for thin SiC layers (0.16 μm) in comparison with thicker ones (1.6 μm), independently of the type of Schottky contact (Pt or Pd). Conversely, a reverse effect was observed on the barrier height ϕ_{Bp} and the series resistance R_s, i.e. smaller values were obtained for thicker SiC layers. The low value of series resistance obtained using Cheung's method clearly indicates the high performance of the Schottky diode with thicker SiC layer. These results confirm theoretically the variation of barrier height and series resistance with the ideality factor. In addition, the high performance of the Schottky diode Pt(Pd)/SiC-pSi using a thin SiC layer (0.16 μm) is due to the low density of defects that can be present in this layer compared to the thick one.

For the Schottky diode with a PSC layer Pt (Pd)/PSC-pSi, different phenomenon was observed in the dependence on the SiC layer thickness. After anodization of the SiC layer to form the porous layer (PSC), the electrical parameters totally changed. A high electrical performance was reached when using the PSC layer instead of the SiC layer, indicating the low value of series resistance (R_s = 0.05 kΩ) and ideality factor of close unity (n = 1.065) especially for a thin SiC layer, as shown in Table 1 was evaluated.

Figs. 10 and 11 illustrate *I–V* characteristics of Pt/PSC-pSi and Pd/PSC-pSi, respectively, in hydrogen (H_2) and hydrocarbon (C_2H_6) gas. It reveals a difference in signal between the two gases, indicating clearly the gas selectivity of this type of Schottky diode. In contrast, for a Schottky diode using an unetched (non-porous) SiC layer no separation of the signal was observed. These results allow us to say that the porous layer PSC gives a novel sensor gas with a high sensitivity as shown in Figs. 12 and 13.

Table 2 illustrates the variation of barrier height of the Schottky diodes elaborated with SiC and PSC layers. The large barrier height clearly shows the important role of the PSC layer on the performance of gas sensing. A high sensitivity $\Delta I/I$ = 90% and a good selectivity of the sensors were reached at low voltages below 1 V, especially by using a PSC layer for the two catalytic metal Pt and Pd.

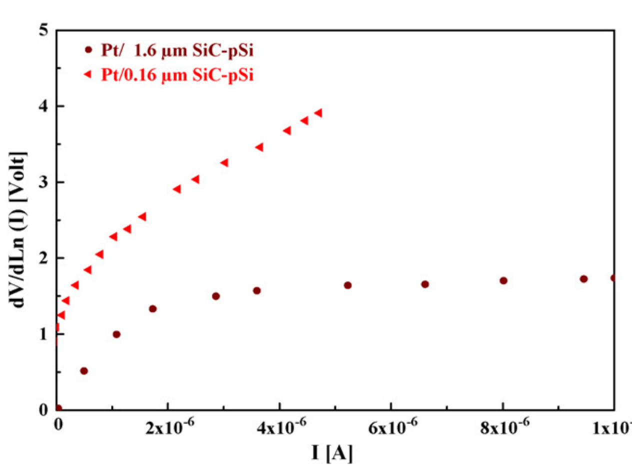

Fig. 8. dV/dln(I) vs. I plots of the Pt/SiC-pSi Schottky diodes.

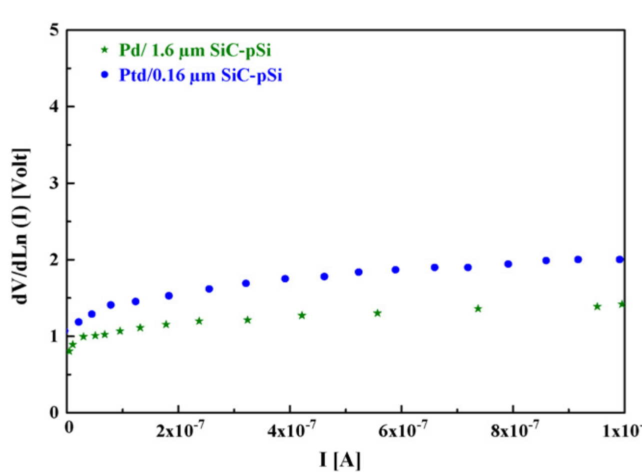

Fig. 9. dV/dln(I) vs. I plots of the Pd/SiC-pSi Schottky diodes.

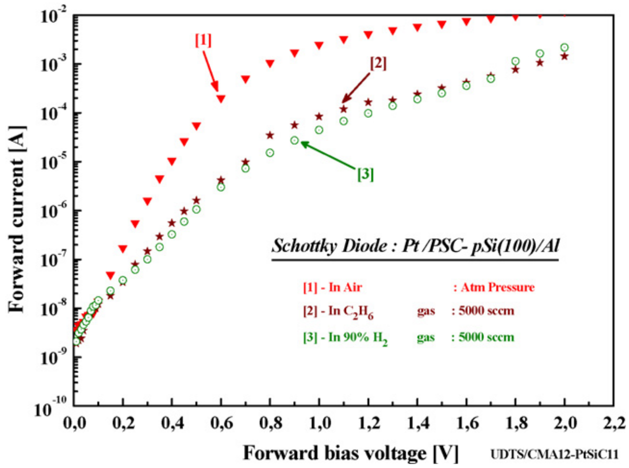

Fig. 10. *I–V* characteristics vs. forward bias voltage of Pt/PSC-pSi in air, 90% H_2 and C_2H_6.

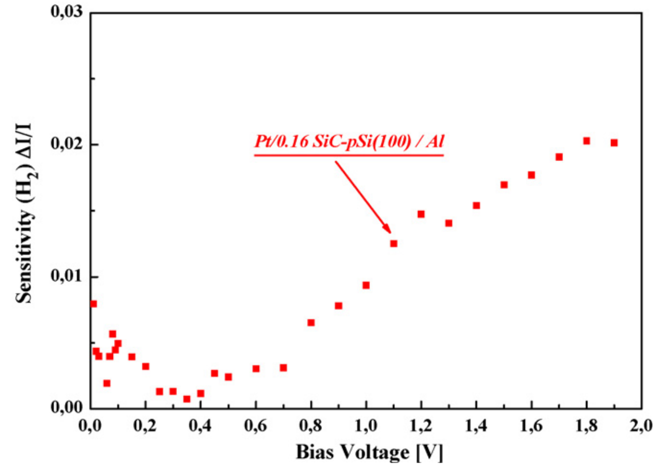

Fig. 12. Sensitivity to H_2 gas vs. bias voltage of the Pt/SiC-pSi Schottky diodes.

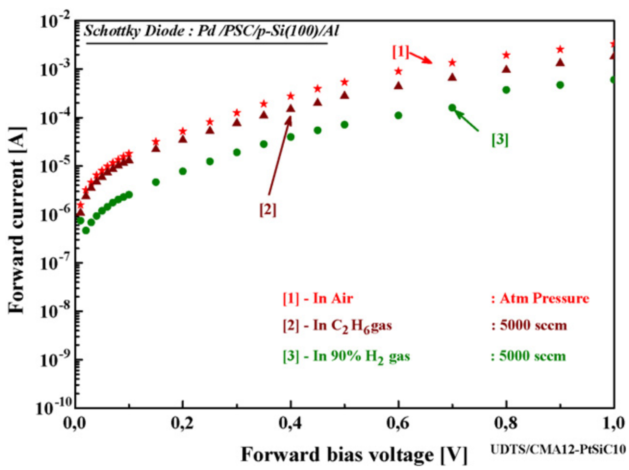

Fig. 11. *I–V* characteristics vs. forward bias voltage of Pd/PSC-pSi in air, 90% H_2 and C_2H_6.

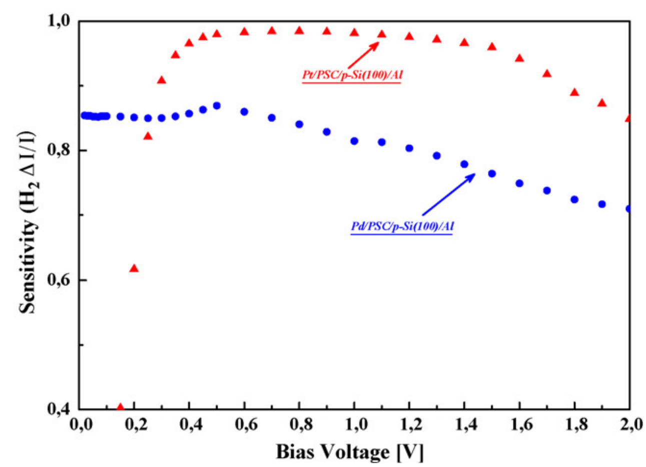

Fig. 13. Sensitivity to H_2 gas vs. bias voltage of the Pt and Pd/PSC-pSi Schottky diodes.

3.2.2. Capacitance–voltage

The capacitance of the Schottky photodiode depends on the depletion width (W). Increasing the bias voltage leads to an increase of the depletion width and a decrease in capacitances until the full depletion condition is achieved. When the depletion region reaches the back contact and the capacitance becomes constant the device is said to be fully depleted. The depletion width plays an important role in the diode performances [20].

The junction capacitance is given by:

$$C = \frac{a\varepsilon_{\mathrm{SiC}}}{W} \tag{6}$$

The depletion width (W (μm)) is defined as the region where the carriers are generated is related to the applied reverse bias voltage V (volt) by the relation:

$$W = \sqrt{2\varepsilon_{\mathrm{SiC}}\mu_{\mathrm{p}}\rho_{\mathrm{p}}(V_{\mathrm{bi}} + V)} \tag{7}$$

Table 2
Electrical parameters deduced by *C–V* measurements

Schottky diode	Built in-voltage V_{bi} (V)	Barrier height ϕ_{Bp} (eV)	Concentration $N_{A(exp)}$ (at/cm^3)	$N_{A(ref)}$ (at/cm^3)
Pt/0.16 μm SiC-pSi (unetched)	0.483	0.753	7.09×10^{14}	4.88×10^{14}
Pt/1.6 μm SiC-pSi (unetched)	0.689	0.977	2.96×10^{14}	1.24×10^{14}
Pt/PSC (1.6 μm SiC)-pSi (50% HF/50% ETG – t = 2 min)	0.400	0.622	4.02×10^{15}	1.24×10^{14}
Pt/PSC (0.16 μm SiC)-pSi (50% HF/50% ETG – t = 15 s)	0.680	0.961	3.90×10^{14}	4.88×10^{14}
Pd/0.16 μm SiC-pSi (unetched)	0.322	0.598	5.59×10^{14}	4.88×10^{14}
Pd/1.6 μm SiC-pSi (unetched)	0.588	0.820	2.74×10^{15}	1.24×10^{14}
Pd/PSC (1.6 μm SiC)-pSi (50% HF/50% ETG – t = 2 min)	0.517	0.777	9.00×10^{14}	1.24×10^{14}

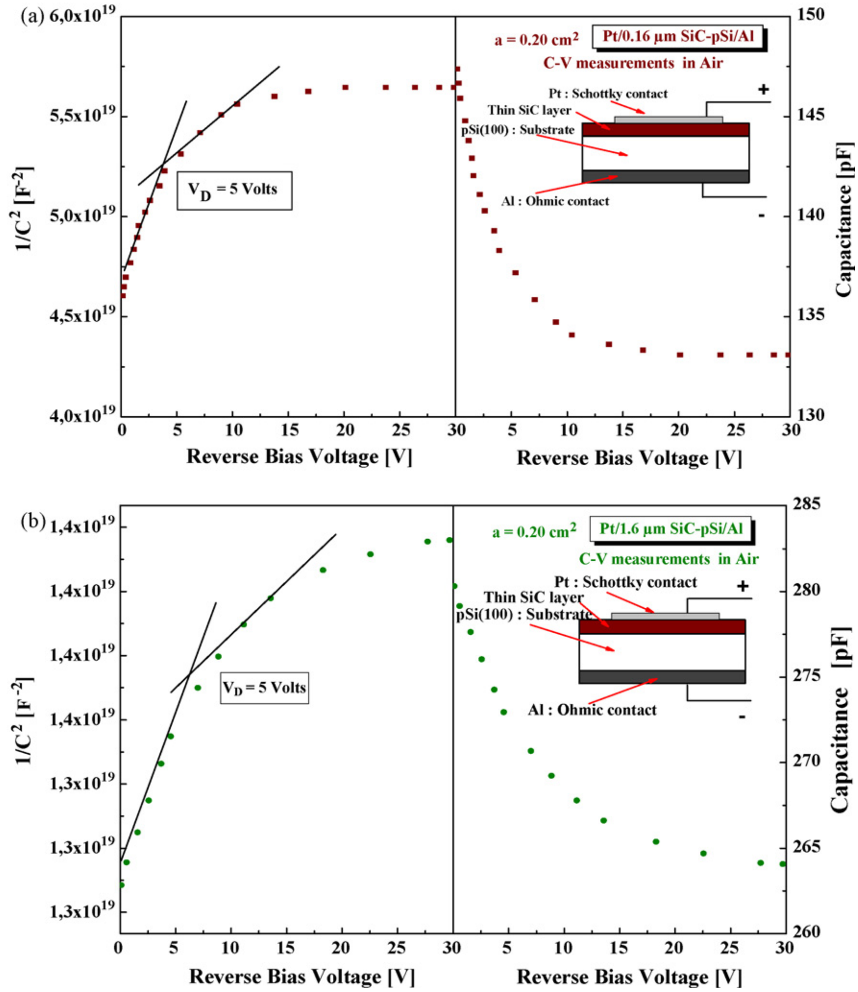

Fig. 14. C and $1/C^2$ vs. V of the Schottky diodes. (a) Pt/0.16 μm SiC-pSi and (b) Pt/1.6 μm SiC-pSi.

where μ_p is the hole mobility of p-type 6H–SiC (90 cm^2/V s) and ρ (Ω cm) is the electrical resistivity of the layer (SiC). The values of built-in voltage V_{bi} and the concentration of acceptors N_A (at/cm^3) are evaluated by calculating and the slope of the plot $1/C^2$ versus V:

$$\frac{1}{C^2} = \frac{2}{qa^2\varepsilon_{SiC}N_A}[V_{bi} + V] \tag{8}$$

where a equal to 0.20 cm^2 is the junction area (active area), W the depletion width, ε_{SiC} the permittivity of SiC (= 9.7), V_{bi} the built-in voltage, N_A the concentration of acceptors in the starting material, and V is the applied reverse bias voltage. Using the relation above, we can also determine the barrier height by capacitance measurements by using the following equation for n-type silicon:

$$q\phi_{Bp} = qV_{bi} + (E_F - E_V) = qV_{bi} + qV_p \tag{9}$$

where

$$qV_p = kT\ \ln\left[\frac{N_V}{N_A}\right] \tag{10}$$

then

$$q\phi_{Bp} = qV_{bi} + \frac{kT}{q}\ln\left[\frac{N_V}{N_A}\right] = qV_{bi} + 0.0258\ \ln\left[\frac{2.531 \times 10^{19}}{N_A}\right] \tag{11}$$

N_V is the effective density of states in the valence band of silicon carbide at room temperature ($N_V = 2.513 \times 10^{19}$ at/cm^3). High frequency capacitance measurements were performed using an Agilent 4278 capacitance meter at 1 MHz. Figs. 14 and 15 show C and $1/C^2$ versus V plots of Pt/SiC-pSi and Pd/SiC-pSi structure, respectively. Table 2 shows electrical parameters deduced by the C–V measurements, such as V_{bi}, ϕ_{Bp} and N_A. The built-in potential and the barrier height for the Schottky contact (platinum and palladium) obtained by capacitance measurements were in agreement with those given by I–V the two preceding techniques taking account the errors of measurements. However, this measurement technique is not very precise.

C–V analysis reveals a decrease in the capacitance with increasing voltage. This decrease is less for a thin SiC layer (0.16 μm) in comparison to that of a thick SiC layer (1.6 μm), clearly indicating the role of the SiC thickness in determining

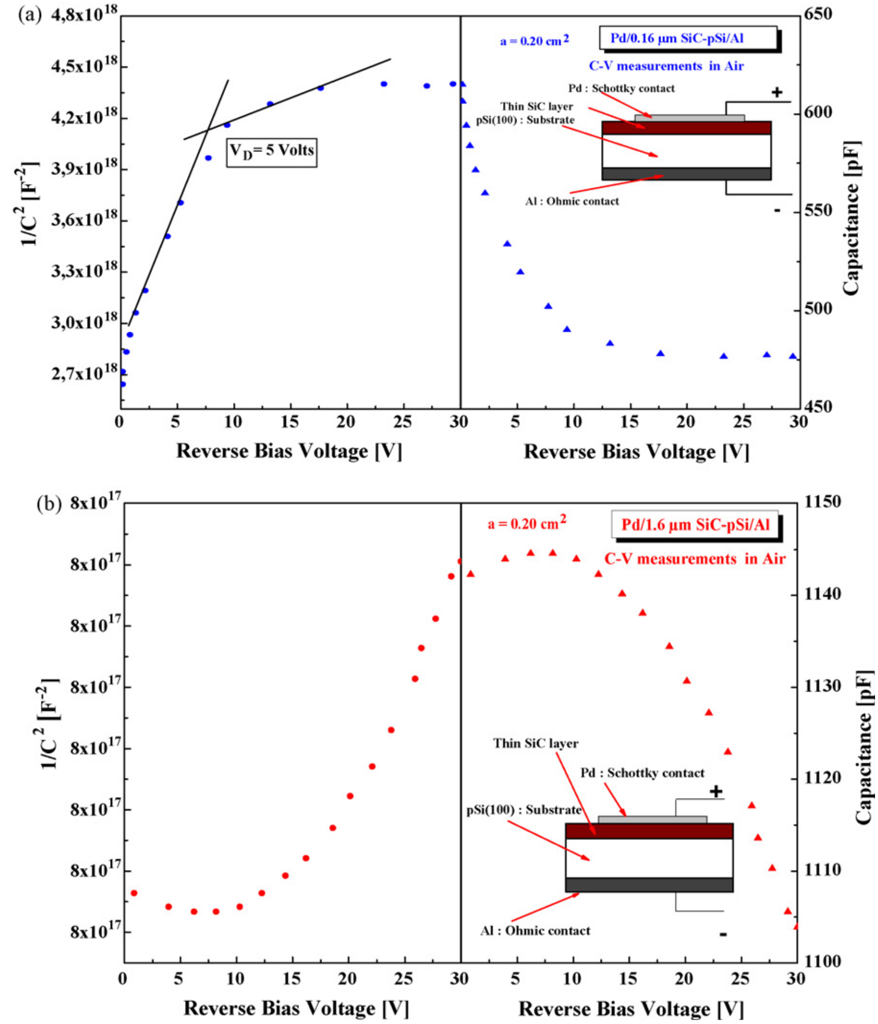

Fig. 15. *C* and $1/C^2$ vs. *V* of the Schottky diodes. (a) Pd/0.16 μm SiC-pSi and (b) Pd/1.6 μm SiC-pSi.

of sensor capacitance values that play a role to reduce the noise of the sensors.

We have used *C–V* measurements to determine an important parameter characterizing the performance of the Schottky diode barrier height. Two effects influence the barrier height, the SiC thickness and the nature of the smooth or porous surface. In Table 3 we can see that's the barrier height increasing with increasing layer thickness. This fact confirms the values obtained by *I–V* measurement. In contrast, the barrier height decreases for the Schottky diode with a porous SiC layer independently of the type of catalytic metals (Pt or Pd).

We note also that *C–V* measurements give an agreement value of acceptor concentration (N_A) of the thin layer which is consistent as the one calculated by using the relation ($N_A = 1/(q\mu_p\rho)$).

Table 3
Variation of barrier height of Pt (Pd)/SiC-pSi Schottky diode as a function of gas environment

		ϕ_{Bp} (eV)	$\Delta\phi_{Bp} = \phi_{Bp}$ (gas) − ϕ_{Bp} (air) (meV)
Schottky diode: Pt/SiC-pSi			
Air	Ambient	0.7278	–
C_2H_6	Gas	0.7283	0.5
H_2	Gas	0.7285	0.7
Schottky diode: Pd/SiC-pSi			
Air	Ambient	0.7021	–
C_2H_6	Gas	0.7098	7.7
H_2	Gas	0.7465	44

4. Conclusion

The results presented in this work show the roles of the catalytic metal and the thickness of the SiC and porous SiC (PSC) layers on the performances of the Schottky diodes. A good *I–V* and *C–V* characteristics were obtained on platinum and palladium Schottky diodes, which were fabricated as a function of the thickness of SiC and porous structure SiC layers. A best result was obtained on the Schottky diodes with a non-porous thick SiC (1.6 μm). In contrast, a high performance of electrical characteristics was obtained with porous structure SiC layer which performed on a thin layer (0.16 μm), independently of the catalytic metal type (Pt or Pd). Finally, a high sensitivity (90%) with a good selectivity of the gases were reached at low voltages below 1 V, especially by using a PSC layer for the two catalytic metal Pt and Pd.

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Biographies

Karima Bourenane received her master degree in chemical from the National Polytechnic School of Algiers in January 2004. Since 2004, she works in Silicon Technology Unit in Algiers and she started research as a doctoral candidate in 2005 at Mechanical and Chemical Department from Sciences and Technology Houari Boumediene University of Algiers. Subject of her doctoral work was chemical and electrochemical treatment of thin SiC layer based on SiC and porous SiC gas sensors. She is currently working in the field of sensors for automotive safety and exhaust gas monitoring for advanced environmental safety.

Aissa Keffous received his master degree in physics from Silicon Technology Unit in Algiers in March 1994. Since 2004, he has been working towards his Doctoral Research Silicon Technology Unit and Department of Physical in Houari Boumedienne University, Algiers. His research is concentrated on the field of elaboration and characterization of thin SiC layer by pulsed laser deposition and by sputtering.

Ghania Nezzal is the director of National Polytechnic School of Algiers and a teacher in Environment and chemical field at Houari Boumediene University of Algiers, Mechanical and Chemical Department. His research is concentrated on the field of environment in its all aspects.

Ahmed Bourenane received his degree in electronics from the Sciences and Technologies University of Blida, Algeria, in 1993. In the same year, he joined the Nuclear Research Centre of Birine, NRCB. Since then, after a completion of one year of additional courses in nuclear sciences, he has been working in the Exploitation Department of the Reactor. In addition to his usual activities in NRCB, the nuclear detectors, in particular those silicon-based, are among his main interest of research.

Yacine Boukennous received his engineer degree in electronic from National Polytechnic School of Algiers in February 1982. Since 1984, he is being working in Silicon Technology Unit in Algiers where he is responsible of the technical division. In 1996, he received his master degree in photovoltaic field from Silicon Technology Unit.

Assia Boukezzata received her engineer degree in chemistry from the Sciences and Technology Houari Boumediene University, Department of Mechanical and Chemical in September 2005. In the same year, she started research as a master candidate at Houari Boumedienne University (USTHB), Mechanical and Chemical, Department, Algiers. Subject of her doctoral work was chemical and electrochemical treatment of thin layers based on SiC for photodiode application.