

A Low-Cost, Nanowatt, Millimeter-Scale Memristive-Vacuum Sensor

Khaled Humood, Sueda Saylan, Baker Mohammad, and Maguy Abi Jaoude

Abstract— This work demonstrates a low-power, light-weight, and cost-efficient vacuum pressure sensor and air leak detector based on 2 mm x 2 mm Cu/HfO₂/p⁺-Si memristor device. The operating principle of the proposed sensor relies on monitoring the off-state resistance (R_{off}) of the device, wherein the experimental results show that the R_{off} value is inversely proportional to the vacuum pressure. The fabricated sensors demonstrated a sensitivity of 493 Torr⁻¹ and power consumption as low as ~ 8 nW, when tested in a wide range of sub-atmospheric pressure (4.9x10⁻⁵ – 760) Torr, making them highly suitable for low-power applications. Furthermore, it is shown that the sensor can be integrated with a microcontroller-based circuitry to serve as a standalone sensing system.

Index Terms—vacuum sensor, air leak detector, memristor, ReRAM.

I. Introduction

Air leaks pose a significant concern for pressurized space structure integrity and astronaut safety [1]. Micrometeoroids and orbital debris (MMOD) with diameters ranging from 25 mm to 125 mm, and traveling at approximately 9-10 km/s in the Low Earth Orbit (LEO) frequently impact and damage the International Space Station (ISS) and other space-based structures [1, 2]. Moreover, this is nowadays becoming a severe threat as the number of MMODs in the LEO increases due to accelerated space mission programs [3]. Therefore, continuous monitoring of air leaks is needed and this requires low-power, low-weight vacuum pressure sensing systems.

Pressure sensors are widely used in space pressurized structures and environments. Various solutions in the literature enable to sense changes in the pressure from the atmospheric pressure (~760 Torr) down to high vacuum (~6.5 × 10⁻⁶ Torr) [4-11]. These solutions are thermal sensors that depend on the heat transfer principle [7-11], or they rely on frequency shifts caused by damping variation under different pressures [4-6]. However, most of the proposed solutions operate at high power and require heavy and oversized circuitry to be used (see Table II), not being able to comply with low power, light weight, and small footprint requirements for mobile applications [1, 2].

In this work, a novel low-cost, small-form-factor, and low-power vacuum pressure sensor, based on 2 mm x 2 mm Cu/HfO₂(~ 80-nm-thick)/p⁺-Si memristor devices, is proposed for the potential development of air-leak sensors. Furthermore, a complete system implementation using an Arduino microcontroller that is compatible with the low-power and small-area targets of the application is presented.

Emerging memristor (MR) technologies play an important role in electronic applications due to their low-power operation,

small footprint, multi-level and high-speed switching behavior [12-14]. The two-terminal structure of memristive devices enables the integration of high dense crossbar arrays with a minimal bit area of 4F², where F is the minimum feature size [15]. The resistive switching (RS) property in MRs has also been exploited in applications beyond memory, such as matrix multiplication, in-memory computing (IMC), and hardware-accelerated processing [16-19]. Moreover, the capability of the MR electrical parameters to respond to external stimuli is promising for expanding the use of memristors as sensing elements [20-22]. Further, the intrinsic variability in MR electrical characteristics, from cycle to cycle and from device to device, could be exploited for security applications [23-25].

The proposed sensor utilizes the change in the off-state resistance (R_{off}) of the memristor in response to a change in the pressure, to sense a transition from the atmosphere to vacuum environment. Such a sensing method does not depend on temperature or frequency changes, and hence it is less susceptible to noise and interference [4]. Furthermore, the utilization of R_{off} as the basis of sensing leads to extremely low power consumption, as low as 8 nW. Compared with the state-of-the-art (see Table II), the proposed sensor provides more than three orders of magnitude improvement in the power consumption, making the sensor critically important for low power applications. Moreover, the sensor makes use of a light-weight and low-power circuitry based on Arduino microcontroller, eliminating the need for heavy and expensive equipment such as amplifiers, function generators, spectrum analyzers.

Device fabrication, performance characterization, and circuit implementation of the memristor-based vacuum sensor are described in detail in the following sections.

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II. MATERIALS AND METHODS

A. Device Fabrication

Fig. 1(a) shows the schematic representation and top view photograph of the fabricated devices, where the Cu top electrode (TE) defines the effective area for one device. The Cu TE was deposited by DC sputtering using a Q300T T coating system (Quorum Technologies, UK). The details of the HfO_2 deposition, as well as a comprehensive electrical characterization and statistical data analysis for the devices, can be found in [20, 26].

B. Experimental Setup

Fig. 1(b) and **Fig. 1(c)** show the experimental setup used for measuring the device resistance in alternating atmospheric and vacuum conditions. The setup consists of a cryogenic vacuum probe station (TTPX, Lake Shore Cryotronics Inc., USA) and a parameter analyzer (Keithley 2450, Tektronix, USA). The sensor is placed inside the vacuum chamber of the probe station (**Fig. 1(b)**) and its off-state resistance R_{off} is measured using the parameter analyzer that is connected to the probe arm base of the probe station (**Fig. 1(c)**). The details of the R_{off} read operation are given in Section II.D. A turbo pumping system (X3580A, Agilent Technologies, Switzerland) is used to control and read the pressure in the system.

C. I-V Characterization

The Current-Voltage (I - V) characteristics were obtained using a Keithley 4200-SCS parameter analyzer, by applying DC linear sweep functions. Dual positive sweeps ranging from 0 to 3.5 V and 0 to -3.5 V were applied for the SET and RESET operations, respectively, with a voltage step size of 0.05 V in both cases. The p^+ -Si bottom electrode was grounded during both operations. These measurements were performed at room temperature and in ambient air. This procedure was used to pre-screen the memristor characteristics and is not required when the device is deployed in the intended application. An account of the material and electrical characterization studies of the devices under atmospheric and vacuum environment is reported elsewhere [26].

D. R_{off} Read Operation

The R_{off} read operation illustrated in **Fig. 2** was performed by using the experimental setup described in Section II.B. A read pulse was applied every 30 seconds to take a resistance measurement. The pulse amplitude was set to -0.4 V with a duration of 60 μs , which is the minimum pulse duration applicable by the tool. The read pulse magnitude and polarity were selected to minimize any changes in the resistance state of the device due to the read operation.

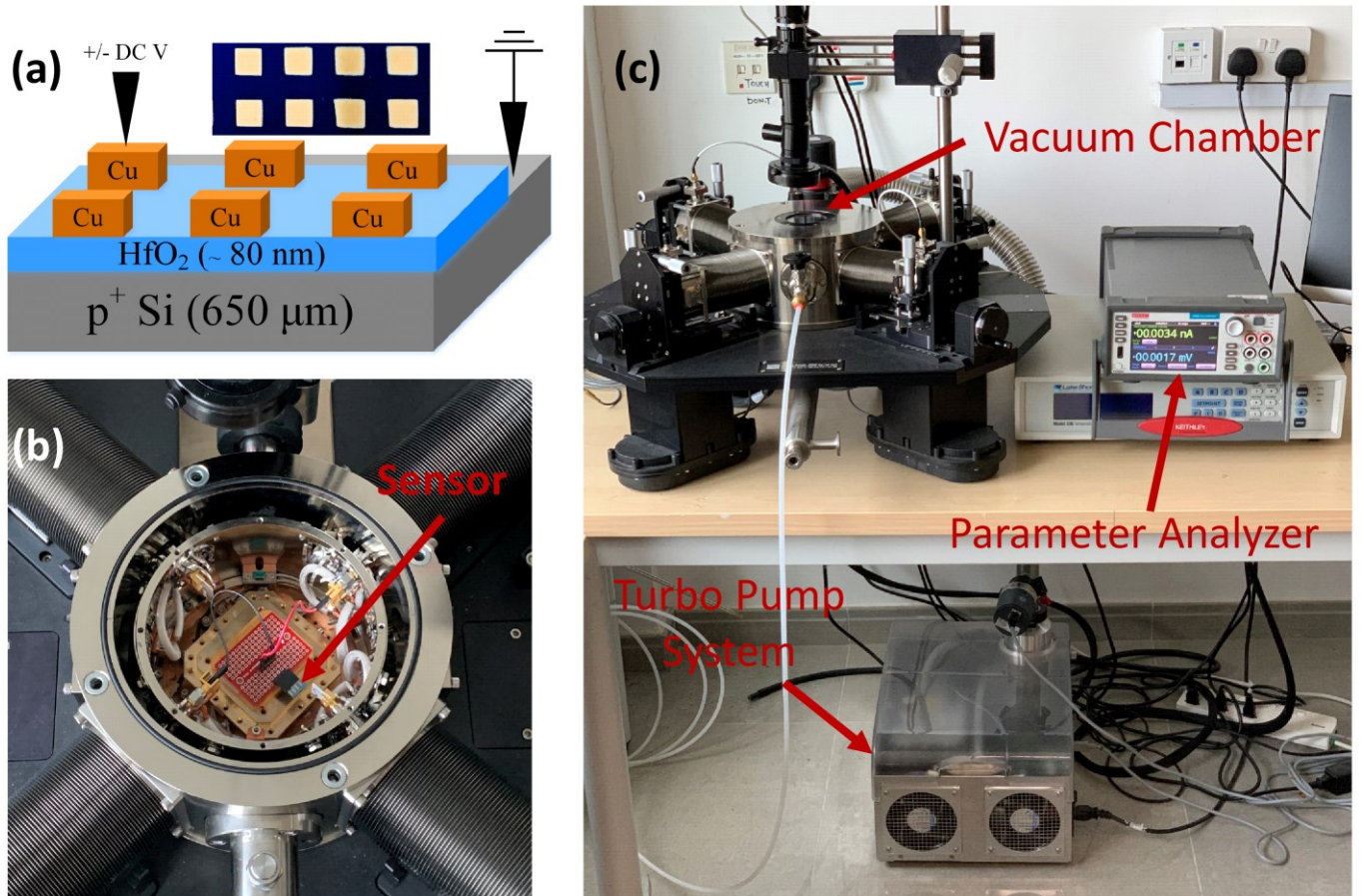


Fig. 1. (a) Schematic representation and top view photograph of the fabricated $\text{Cu}/\text{HfO}_2/\text{p}^+\text{-Si}$ devices, where each Cu electrode (2 mm x 2 mm) defines a single device. The Cu electrode is supplied with a DC voltage, while the silicon electrode is grounded. Photograph of the experimental setup used for measuring the device resistance in alternating atmospheric and vacuum conditions, showing (b) the sensor prototype on a breadboard and (c) the vacuum chamber, parameter analyzer, and turbo pumping system.

III. RESULTS AND DISCUSSION

A. Resistive Switching and Capacitance Characteristics of Cu/HfO₂/p⁺-Si Devices in Air Atmosphere

Fig. 3 shows a pristine device tested through consecutive 10 SET-RESET voltage sweeps in the air atmosphere, to verify the cycle-to-cycle stability before investigating the vacuum sensing performance. In agreement with our previous findings [20], the device exhibited bipolar resistive switching within a bias voltage sweep window of +3.5 to -3.5 V and did not require an electroforming step, which is typical for the devices operating under the ECM mechanism [27, 28]. During the SET sweeps, a compliance current $I_{CC} = 500 \mu\text{A}$ was maintained to avoid the permanent breakdown of the dielectric material of the device [29, 30]. A detailed investigation of the device-to-device and cycle-to-cycle variation of the Cu/HfO₂/p⁺-Si device characteristics can be found in our previous work [20, 26]. In addition, the capacitance characteristics of the device for the HRS and LRS are shown in the Supplementary Material, Fig. S1.

B. Vacuum Behavior of Cu/HfO₂/p⁺-Si Devices

After screening the devices in the air atmosphere (Fig. 3), the electrical characterization was performed in alternating air and vacuum conditions to investigate the performance of the developed technology as a vacuum pressure sensor. In particular, we measured the off-state resistance R_{off} by executing the read operation described in Section II.D. Fig. 4 shows the device R_{off} as a function of vacuum pressure ranging from $\sim 3.2 \times 10^{-3}$ to 4.9×10^{-5} Torr while the turbo pumping system evacuates the vacuum chamber. Before evacuating the testing chamber, the resistance was measured in the air atmosphere for 10 min and recorded to be $\sim 27 \text{ M}\Omega$ as shown in the Supplementary Material Fig. S2. After starting the turbopump, the resistance increased gradually as the pressure reduced until a pressure threshold value $P_{\text{th}} = 5.5 \times 10^{-5}$ Torr (see Fig. 5(a) for the range of experimentally recorded P_{th}), at which an abrupt increase in the resistance from $39 \text{ M}\Omega$ to $126 \text{ M}\Omega$ was observed.

Thus, in response to a change in vacuum pressure, the off-state resistance exhibited two distinct regimes: first, the resistance gradually increased as shown in the inset of Fig. 4, following the power equation shown in the inset; and second, it abruptly increased beyond the threshold pressure P_{th} . This allows the use of the developed technology as a vacuum pressure sensor in the first region and as a vacuum detector in the latter. The resistance versus time plot of the data presented in Fig. 4 is shown in the Supplementary Material, Fig. S2. In addition, the R_{off} response of another pristine device as a function of pressure (Fig. S3), as well as the response of the device to pressure changes between atmosphere and vacuum over multiple cycles (Fig. S4) are provided in the Supplementary Material. The data presented in Fig. S4 show that the time response of the proposed sensor during the pumping stage (i.e., when the pressure changes from atmospheric to vacuum pressure levels) is not equivalent to the time response for the venting stage (i.e., when the pressure changes from vacuum to atmospheric). Hence, the proposed sensor can be utilized in vacuum sensing and detection applications wherein the pressure only changes in one direction

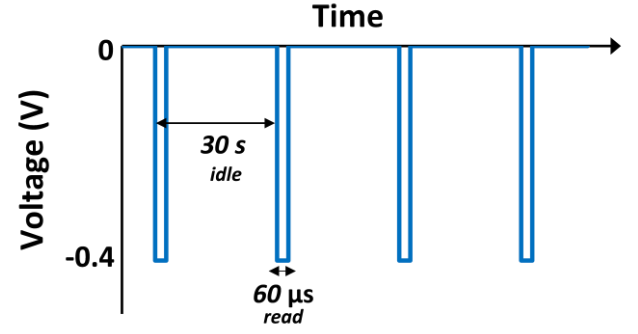


Fig. 2. R_{off} read pulses. The read voltage amplitude and the read period were set to -0.4 V and 30 s, respectively. The pulse width was set to 60 μs , which is the minimum width achievable by our setup.

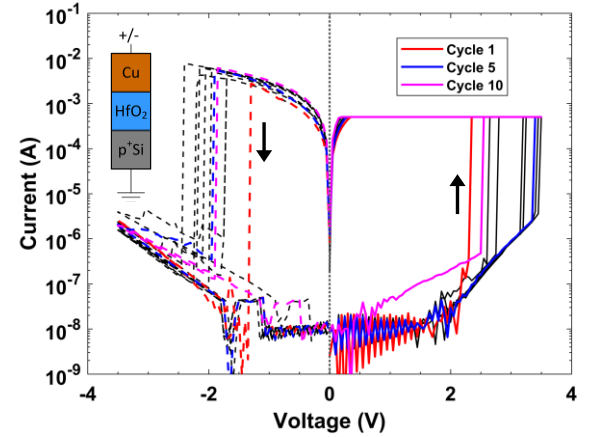


Fig. 3. I - V characteristics for SET (solid lines) and RESET (dashed lines) of a Cu/HfO₂/p⁺-Si device programmed using a compliance current of 500 μA . Cycles 1, 5, and 10 are highlighted in red, blue, and magenta, respectively. Other cycles are highlighted in black. The arrows indicate the direction of current. The biasing scheme is shown in the inset.

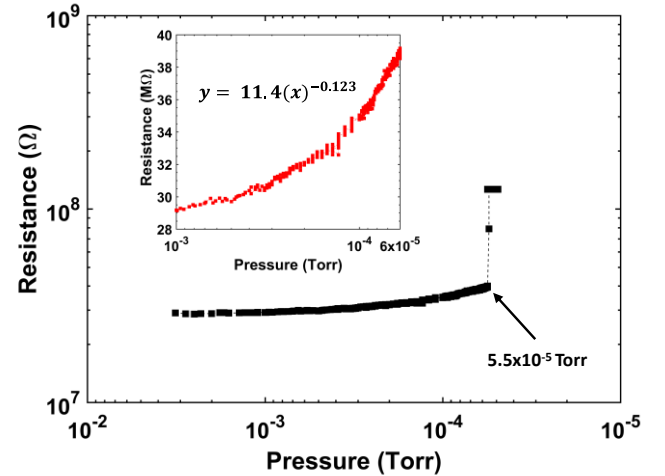


Fig. 4. R_{off} as a function of pressure from 3.2×10^{-3} to 4.9×10^{-5} Torr. The resistance was measured as 29 $\text{M}\Omega$ and 126 $\text{M}\Omega$ at 3.2×10^{-3} and 4.9×10^{-5} Torr, respectively. Prior to the measurements in vacuum, the atmospheric off-state resistance was measured to be $R_{\text{off}}^{\text{atm}} = 27 \text{ M}\Omega$. The inset shows the change in resistance as a function of pressure from 1×10^{-3} to 6×10^{-5} Torr. The sensitivity within this pressure range is 493 Torr^{-1} . Note: the x-axis values are reversed.

from atmospheric to vacuum. Moreover, Fig. S4 shows that the sensor could be reused for vacuum detection after the relaxation time in atmosphere has passed.

Furthermore, to confirm that the change in R_{off} is due to a change in the vacuum pressure but not due to electrical biasing over a long period, the resistance of a pristine device was measured over 70 hours at atmospheric pressure (760 Torr) as shown in the Supplementary Material, Fig. S5. As demonstrated by the results, the off-state resistance did not change during this experiment.

The sensitivity of the vacuum pressure sensor in the gradual region was calculated using the formula:

$$S = \frac{\Delta R_{\text{off}} / R_{\text{off}i}}{\Delta p}, \quad (1)$$

where ΔR_{off} is the change in resistance, $R_{\text{off}i}$ is the initial resistance in air and Δp is the change in pressure [31]. Based on the data presented in Fig. 4, the sensitivity is $\sim 493 \text{ Torr}^{-1}$ in a pressure range from 1×10^{-3} to 6×10^{-5} Torr. Utilizing the change in R_{off} as the quantitative parameter for sensing significantly reduces the power consumption. The R_{off} values of the fabricated devices lie in the range of 20-200 M Ω . At a read voltage of $V_{\text{read}} = 0.4 \text{ V}$, the maximum power is estimated to be $\sim 8 \text{ nW}$ by using (2).

$$\text{Power} = \frac{V_{\text{read}}^2}{R_{\text{off}}} \quad (2)$$

Fig. 5(a) shows the device-to-device distribution of the pressure threshold P_{th} versus the ratio of R_{off} values measured in vacuum ($R_{\text{off}}^{\text{vac}}$) at P_{th} and in air atmosphere ($R_{\text{off}}^{\text{atm}}$). Each data point represents an individual device, presenting the data for 24 devices in total. Figure 5(b) shows the cumulative distribution function (CDF) of the ratio $R_{\text{off}}^{\text{vac}} / R_{\text{off}}^{\text{atm}}$, which is defined as the probability that the random variable X , corresponding to the resistance ratio in this case, assumes a value less than or equal to a given x . That is, $F(x)$ is the probability that the resistance ratio will be less than or equal to x , whereas, $1 - F(x)$ is the probability that the ratio will be greater than x . As seen in Fig. 5(a), most devices exhibited a resistance ratio above 2 at the measured pressure threshold P_{th} values. Based on the CDF plotted in Fig. 5(b), the probability that the resistance ratio will be greater than 2 is estimated at $\sim 83\%$. This result indicates that the measured resistance ratio of the $\text{Cu}/\text{HfO}_2/\text{p}^+\text{-Si}$ memristive devices can be used as a reliable metric to distinguish a measurable change in the ambient pressure between atmosphere and vacuum.

C. Sensing Principle

The vacuum pressure sensing mechanism by $\text{Cu}/\text{HfO}_2/\text{p}^+\text{-Si}$ memristor device relies on a change in the electronic current conduction in the HRS. As shown in Fig. 6(a) and Fig. 6(b), respectively, the device involves two Schottky barriers connected back-to-back with a series resistance [32, 33] and the current conduction at HRS follows the Schottky emission, as evidenced by a linear dependence of $\ln(I)$ on $V^{1/2}$ [34]. The current-voltage data presented in Fig. 6(b) have been extracted from the I - V sweeps performed in air and vacuum by using the same device in both cases (Supplementary Material Fig. S6). The bias voltage range (0.2 - 1 V) is selected to be less than the SET voltage to ensure that the device is in the HRS. The regression equations obtained for the linear fits to the data (*solid lines*) are also provided in Fig. 6(b). The slope (a) and the

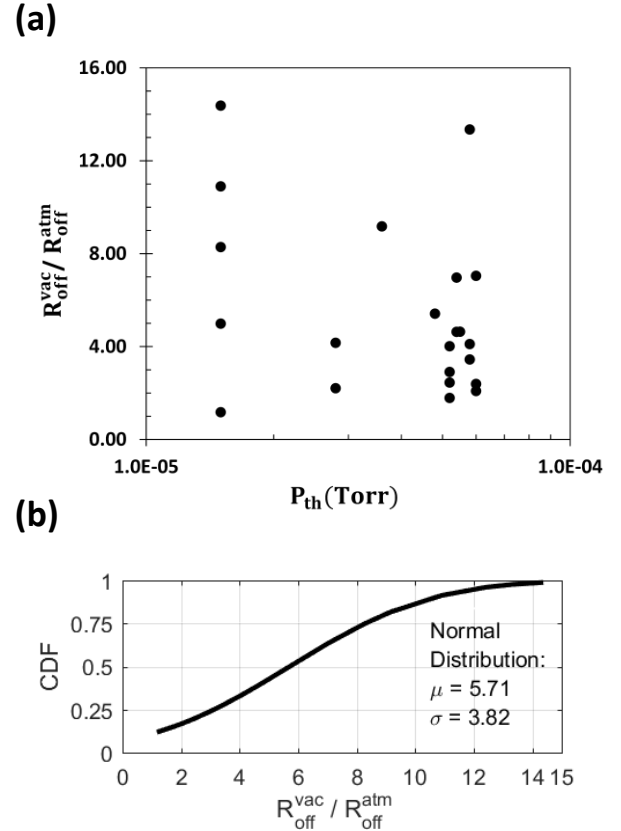


Fig. 5. (a) Device-to-device distribution of the threshold pressure (P_{th}), along with the ratio of R_{off} values measured in atmosphere ($R_{\text{off}}^{\text{atm}}$) and in vacuum ($R_{\text{off}}^{\text{vac}}$) after the threshold pressure is reached. Each data point represents an individual device. (b) Cumulative distribution function (CDF) fit of the resistance ratio, $R_{\text{off}}^{\text{vac}} / R_{\text{off}}^{\text{atm}}$.

y-intercept (b) of the linear regression equations ($y = ax + b$) are proportional to, respectively, the Schottky field-lowering coefficient (β_s) and the potential barrier at the interface under zero applied bias (ϕ_0) according to the Schottky emission equation [34]:

$$I = AT^2 \exp\left(-\frac{\phi_0}{k_B T}\right) \exp\left(\frac{\beta_s V^{1/2}}{k_B T d^{1/2}}\right), \quad (3)$$

where the constant A is the product of area and Richardson constant, T the absolute temperature, k_B the Boltzmann constant, d the film thickness. The Schottky field-lowering coefficient β_s is given by

$$\beta_s = \left(\frac{e^3}{4\pi\epsilon_r\epsilon_0}\right)^{1/2}, \quad (4)$$

where ϵ_r is the relative permittivity of the material, ϵ_0 the permittivity of free space. The linear regression equations similarly obtained for two other devices (D2 and D3) are also listed in Table I.

A comparison of the slope a allows the comparison of β_s for different devices and different ambient conditions, wherein different β_s values imply different relative permittivity of the HfO_2 film. Similarly, a comparison of the y-intercept b allows the comparison of the potential barrier ϕ_0 . As seen in Table I, a and b can be different for different devices under the same ambient conditions, indicating the formation of different interface products and/or different formation amounts, as well as the formation of different bonding structures and molecules

TABLE I

LINEAR REGRESSION PARAMETERS FIT TO THE $\ln(I)$ VERSUS $V^{1/2}$ DATA OBTAINED FOR DIFFERENT DEVICES IN ATMOSPHERE AND VACUUM

$y = ax + b$	Atmosphere			Vacuum		
	a	b	R^2	a	b	R^2
D1	5.4	-20.48	0.9998	5.5	-21.85	0.9992
D2	6.5	-17.52	0.9932	4.3	-17.73	0.9971
D3	4.5	-15.66	0.9923	3.2	-15.78	0.9949

in the HfO_2 film in individual devices, during the electrical biasing. In addition, these structural variations may be existent in the as-fabricated devices to an extent. For D2 and D3, a is diminished in vacuum, suggesting a decrease in the field-lowering coefficient β_s and thus an increase in the relative permittivity ϵ_r of the HfO_2 film, while it remains almost the same for D1. On the other hand, b , which is proportional to ϕ_0 , increased in vacuum for all three devices, suggesting an increase in the barrier height to be the main source of reduced current conduction in vacuum. It can additionally be argued that compositional changes may have also occurred in the HfO_2 film, linked to the changes at the electrode interface that have led to an increase in the barrier height. Thus, a change in the series resistance of the HfO_2 film may also be playing a role in the changing electronic transport in vacuum.

Functional groups can play an essential role in the operation of memristive devices [35–37]. For instance, recently, a type of memristor that is based on the proton movement and proton-coupled electron transfer (PCET) processes has been reported [35], wherein the proton conductivity has increased with increasing humidity and this has led to a strong dependence of I - V plots on the humidity. In another study, it is reported that the OH^- groups attached on the lateral surfaces of ZnO nanowires is responsible for altering the Schottky barrier at the electrode/ZnO interface and decreasing the overall electronic conductivity in a memristive nanowire [36]. It was also shown by means of ab initio density functional theory (DFT) calculations that the OH^- groups, as well as $\text{CH}_3\text{-O}^-$ and $\text{CH}_3\text{-COO}^-$, cause deep surface states to form at the top of the ZnO valence band that act as electron traps and thus reduce the nanowire conductivity [37]. As we have shown earlier [26], the FT-IR spectrum for a vacuum-tested Cu/ HfO_2 /p $^+$ -Si device (subjected to electrical biasing in vacuum) can be quite different from that of a pristine sample (not subjected to electrical biasing and vacuum conditions). While the distinct symmetric stretching vibrations for carboxylate groups (COO^-) and carbonate species were present only in the vacuum-tested device, the representative FT-IR absorption bands for stretching vibrations of water molecules were substantially present only in the pristine device. As suggested in the literature, water molecules can be adsorbed in metal oxide films with various bonding states, including water molecules strongly bound to hydroxyl groups and hydrogen-bonded water molecules [38]. It was also shown that Hf–OH stretching vibrations can appear as a band at around 1575 cm^{-1} in the FT-IR spectrum of HfO_2 - SiO_2 binary oxides, associated with water absorption and increase in OH [39]. Multiple weak peaks dispersed around this wavenumber are seen in the zoom-in FT-IR absorbance spectrum for the pristine device shown in

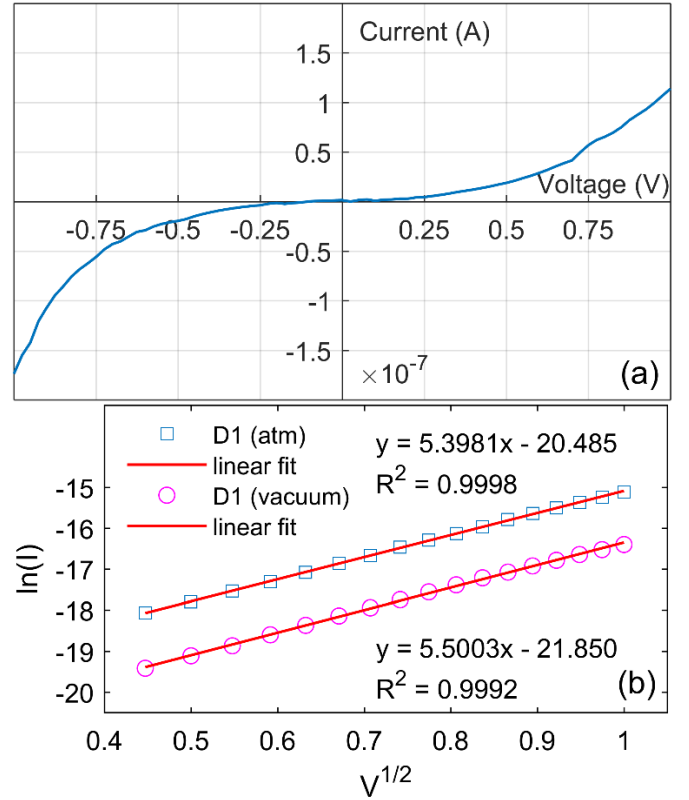


Fig. 6. (a) High resistance state (HRS) of Cu/ HfO_2 /p $^+$ -Si devices exhibiting back-to-back Schottky diode characteristics. (b) Current-voltage relation in the HRS follows the Schottky emission.

Supplementary Material Fig. S7, whereas these are not visible in the spectrum of the vacuum-tested device. As suggested by this data, the desorption of water in the vacuum-tested device maybe forming a partially damaged hydroxyl-bond network, wherein the associated defects in the network act as electron traps at the grain boundaries. These findings suggest that the defects generated due to the desorption of water, in addition to the presence of carboxylate groups (COO^-) and carbonate species, play a role in the electronic transport when the device is biased in vacuum, under the described biasing conditions.

D. Air Leak Detector Circuit Implementation

Fig. 7 shows the circuit implementation of the sensor prototype. The proposed portable, cost-effective, power-efficient, and easy-to-implement prototype consists of an Arduino Uno microcontroller (5 V and 16 MHz), a low pass filter (LPF), a resistor connected in series with the memristor, and a couple of LEDs to serve as pressure/leak indicators. The Arduino microcontroller is used to power and control the circuit. The LPF transforms the Arduino pulse width modulation (PWM) output to a constant DC supply (V_s) that is supplied to the circuit. The filter design parameters are selected to achieve adequate time response without affecting the output voltage (V_s). The potential difference across the MR (V_m) is read through the Arduino analog input (A1). The resistor R_1 connected in series with the memristor provides a voltage reference and it is used to determine the value of the current I using (5):

$$I = \frac{V_s - V_m}{R_1} \quad (5)$$

TABLE II

Fig. 7. Prototype implementation of the vacuum sensing system.

with the United Arab Emirates Space Agency's Space Science, Technology and Innovation (ST&I) Roadmap which aims at developing enabling technologies for Space exploration that are intended to accomplish the objectives of the UAE Space Strategy. The authors also acknowledge the access granted to the System-on-Chip Centre (SoCC) that is supported by the Khalifa University (KU) award No. [RC2-2018-020], as well as the access granted to the electron microscopy suite through the KU-Research Laboratories, to achieve, respectively, the electrical and material characterization studies presented in this work. The authors acknowledge Dr. Anas Al Azzam for granting access to the MEMs Lab at KU to achieve the device fabrication. The authors declare that they have no conflicts of interest.

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