Investigation properties of Au-Porous a-Si0.60C0.40 as humidity sensor

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Investigation properties of Au–porous a-$Si_{0.70}C_{0.30}$ as humidity sensor

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ABSTRACT

In this letter, we report the study of a humidity sensor based on porous amorphous silicon carbide (PASiC) as a function of exposure time and relative humidity (RH). Resistive humidity sensors Au–PASiC/Si(p) were fabricated through evaporating coplanar interdigital gold electrodes. The PASiC was formed by anodization of a-$Si_{0.70}C_{0.30}$ in HF/ethylene glycol (ETG) solution at current density of 50 mA/cm^2 for 30 s. The structural properties of a-$Si_{0.70}C_{0.30}$ thin films deposited by DC magnetron co-sputtering using single silicon crystal, 6H-SiC targets and the PASiC films were investigated by FT-IR and Raman spectroscopy. Hygroscopicity was studied by measuring the resistance of the PASiC under various RH. The results show that the measured resistance highly depends on the applied bias voltage. Moreover, the response signal against RH is found linear for an applied voltage of 2 V. Finally, the response and recovery times were determined around 13 s and 20 min, respectively.

1. Introduction

Humidity sensing or control is of great importance for many situations, including food processing, air-conditioning for office buildings, greenhouse industries, chemical plants, storage depots, etc. Many current humidity sensors suffer badly from the effects of harsh chemical environments. Humidity sensors have been successfully fabricated using many different porous materials as the sensing material, including among others, metal-oxides, polymers, and single-crystalline Si [1–3]. Due to the very well developed silicon industry, Si has a big advantage in terms of integration with standard Si processing techniques, enabling much easier routes to smart sensors with, for example on-chip signal electronics, data storage and processing, etc. To make (single-crystal) Si suitable for humidity sensing, it first must be made porous. This is usually achieved by anodization, which involves immersing the Si in HF-based solutions, and passing an anodic current through the wafer. The Si is effectively slowly 'dissolved' and the surface becomes porous. For good humidity sensing the size and size distribution of the pores is important—the best pore sizes humidity sensing are in the range of ∼10–100 nm. Porous silicon has been demonstrated to be very effective as a humidity sensor [3,4]. However, a common problem with porous Si though, is that it is very 'reactive' and can easily become contaminated by its environment.

It is well-known that the SiC material, which is capable of withstanding harsh environments, should in principle be a better material for relative humidity sensing in such environments such as a high temperature above 600 °C, the SiC resist to much acids and basics products, due to its high optical gap (2.4–3.2 eV) compared to Si material (1.16 eV). We have chosen SiC material as film to humidity sensor essentially for much best properties of this material. SiC with its well-known ability to withstand harsh chemical environments should in principle be a better material for relative humidity sensing in such environments.

In this work, thin (∼1.4 μm) amorphous a-$Si_{0.70}C_{0.30}$ layers were elaborated by DC magnetron co-sputtering technique, on a p-type Si wafer [5]. Because the SiC is amorphous, the electrical current flowing through during porous formation is difficult to predict (at least compared to single-crystalline Si). Nevertheless, we have prepared porous a-$Si_{0.70}C_{0.30}$ by electrochemical anodization in HF, similarly to single-crystal silicon [6,7], and successfully used it for humidity sensing. On the other hand, the advantage of using SiC is that it offers the possibility of a humidity sensor that could withstand very harsh chemical environments. We have first attempted to make SiC porous with uniform pores and then to fabricate humidity sensors based on Au–PASiC/Si(p) structure. The

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electrical performance (resistance, response times, etc.) with variation of relative humidity (RH) has been studied.

2. Experimental procedure

Amorphous a-$Si_{0.70}C_{0.30}$ films were prepared in DC magnetron co-sputtering system from 86 sprigs of a silicon carbide (6H-SiC) target of 5 mm × 10 mm, placed onto a high pulverization region of single crystal silicon (3 in. diameter). The 6H-SiC sprigs were radially fixed on a silicon base. The a-$Si_{0.70}C_{0.30}$ films of thickness ranging from 0.18 to 2.5 μm were deposited on single crystalline silicon (1 0 0) and corning glass 9075 substrates, which were cleaned with ethanol before deposition. Deposition rates of 2–10 Å s^{-1} were achieved in argon and hydrogen plasma mixture, using an operating pressure of 1×10^{-5} mbar with constant gas flow rates of 2 and 10 sccm for H_2 and argon (Ar), respectively. Atomic hydrogen is used for its important role in controlling the film network [5]. All samples were deposited with a 100 W power at 300 °C.

The PASiC humidity sensor structure was fabricated on an amorphous silicon carbide thin films (a-$Si_{0.70}C_{0.30}$) deposited on p-type silicon substrate (Si(p)) by DC magnetron co-sputtering of Si and 6H-SiC as targets. The thickness of the elaborated a-$Si_{0.70}C_{0.30}$ films was 1.4 μm. Porous SiC were made by electrochemical etching in HF/ETG solution (1:1 by vol.) at an anodization current density (J) of 50 mA/cm^2 for 30 s.

The surface morphologies of PASiC were characterized by scanning electron microscopy (SEM), infrared spectroscopy (FT-IR) and Raman spectroscopy. Raman scattering measurements were performed with a *Renishaw Invia Reflex spectrometer* (grating 2400 grooves/mm), with a 457 nm excitation wavelength and 100× objectives. The surface morphology of the films was analyzed by scanning electron microscopy (SEM) in a *JEOLJCM-6300* electron microscope. Fourier transform infrared spectroscopy (FT-IR) was performed using a *Thermo Nicholet Nexus* with a resolution of 4 cm^{-1}.

Relative humidity (RH) control was achieved by using a standard DRIERITE saturated salt (97% $CaSO_4$ + 3% $CoCl_2$), which yields a sealed atmosphere for reducing relative humidity (RH) and we add water vapour to increase the value of RH from 5 to 95% RH in flasks. The resistance variation with relative humidity (RH) was carried out using an *ITECH 6121 voltage source meter* and *Keithley 6485 picommeter* by using a program under *LabView environment*. All electrical measurements were carried out at atmospheric pressure and room temperature. The sensing principle of a resistive humidity sensor is based on the variation of resistance of a sensor's surface caused by the vapour adsorption and desorption.

2.1. Macrostructure of PASiC

The typical surface morphology of unetched a-$Si_{1-x}C_x$ sample and PASiC made at 50 mA/cm^2 for 30 s etching time obtained by SEM is shown in Fig. 1(a) and 1(b), respectively. Fig. 1(a) shows the surface of a thin a-$Si_{0.70}C_{0.30}$ film where a uniform deposit is observed. Whereas the formation of a macroporous layer with a porosity value of macro amorphous films around 60–65% and an average pore diameter of 90 nm (Fig. 1(b)). Note that, the value of x was 0.3, it corresponds to the fraction of carbon in the film calculated from SIMS profiles using the ration $1 - x/x$ = Signal (^{28}Si)/Signal (^{12}C). The a-$Si_{0.70}C_{0.30}$ can be edited as a-$Si_{0.70}C_{0.30}$ and compared to energy dispersive spectroscopy analysis. The average composition of carbon (^{12}C) and silicon (^{28}Si) is around 40 and 60%, respectively. Moreover, the variation of carbon content as a function of a-$Si_{0.70}C_{0.30}$ film thickness shows clearly that the carbon content in the layer increases with the thickness of the film [8].

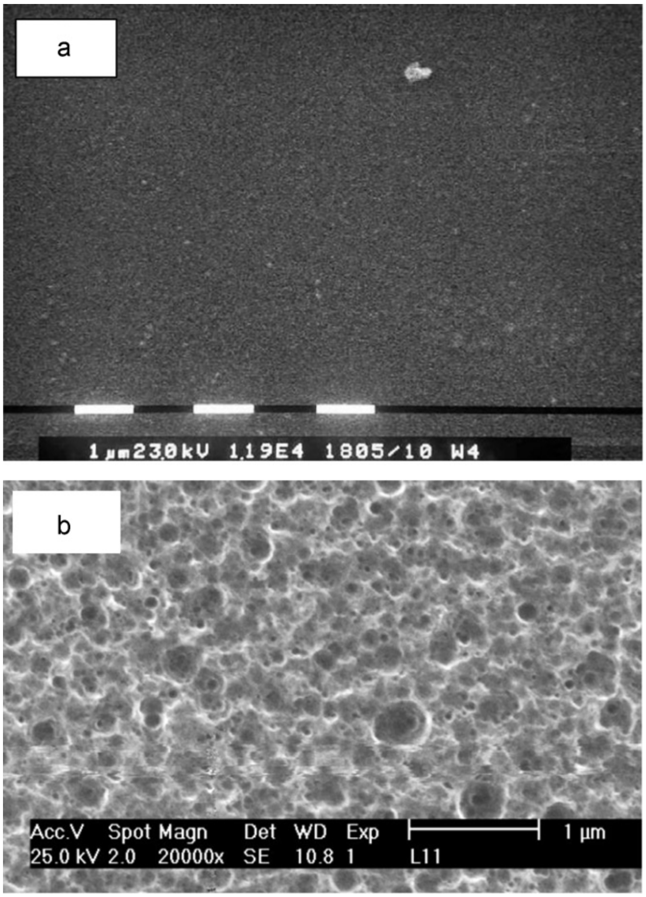

Fig. 1. Plan view SEM image of (a) unetched and (b) anodized a-$Si_{0.70}C_{0.30}$ sample.

2.2. Au–PASiC/Si(p) humidity sensors

The produced sensors of moisture are of resistive type, where interdigital gold contact was deposited onto PASiC film by thermal evaporation at a deposition pressure of 10^{-5} mbar with a thickness of about 600 Å and 1 mm × 0.5 mm surface in width separated from 2 mm (Fig. 2(a)) and the an automatic of acquirement signal measurements as resistance–RH and resistance–time have been carried out by using a humidity sensor chamber (Fig. 2(b) and (c)). Finally, the copper wires were chosen as the leads to connect the electrodes solidified through conductive silver paste. The choose of gold (Au) as a coplanar interdigital electrodes our sensor, because the gold is a metal that is chemically very low reactive with environment, it has a very great heat-transferring surface and thus it has a good sensitivity as well as a certain immunity to pollution. Fig. 3 shows a prototype of Au–PASiC/Si(p) humidity sensor.

3. Results and analysis

3.1. PASiC structural characterization

The surface of the thin a-$Si_{0.70}C_{0.30}$ films before and after anodization in a solution of 50% HF/50% ETG during 30 s has been characterized by infrared absorption spectroscopy. Fourier Transform Infrared Spectroscopy (FT-IR) revealed several vibrations bands. The peaks centered at 616 and 765 cm^{-1} correspond to Si–C band for amorphous film by report to 810 cm^{-1} of the crystalline SiC film. The stretching vibration located at 680 cm^{-1} corresponds to Si-H; their intensity decreases after anodization (Fig. 3(a)). We also noticed the existence of a band centered at 2286 cm^{-1} corresponding to CO_2 (Fig. 4(b)) [9,10].

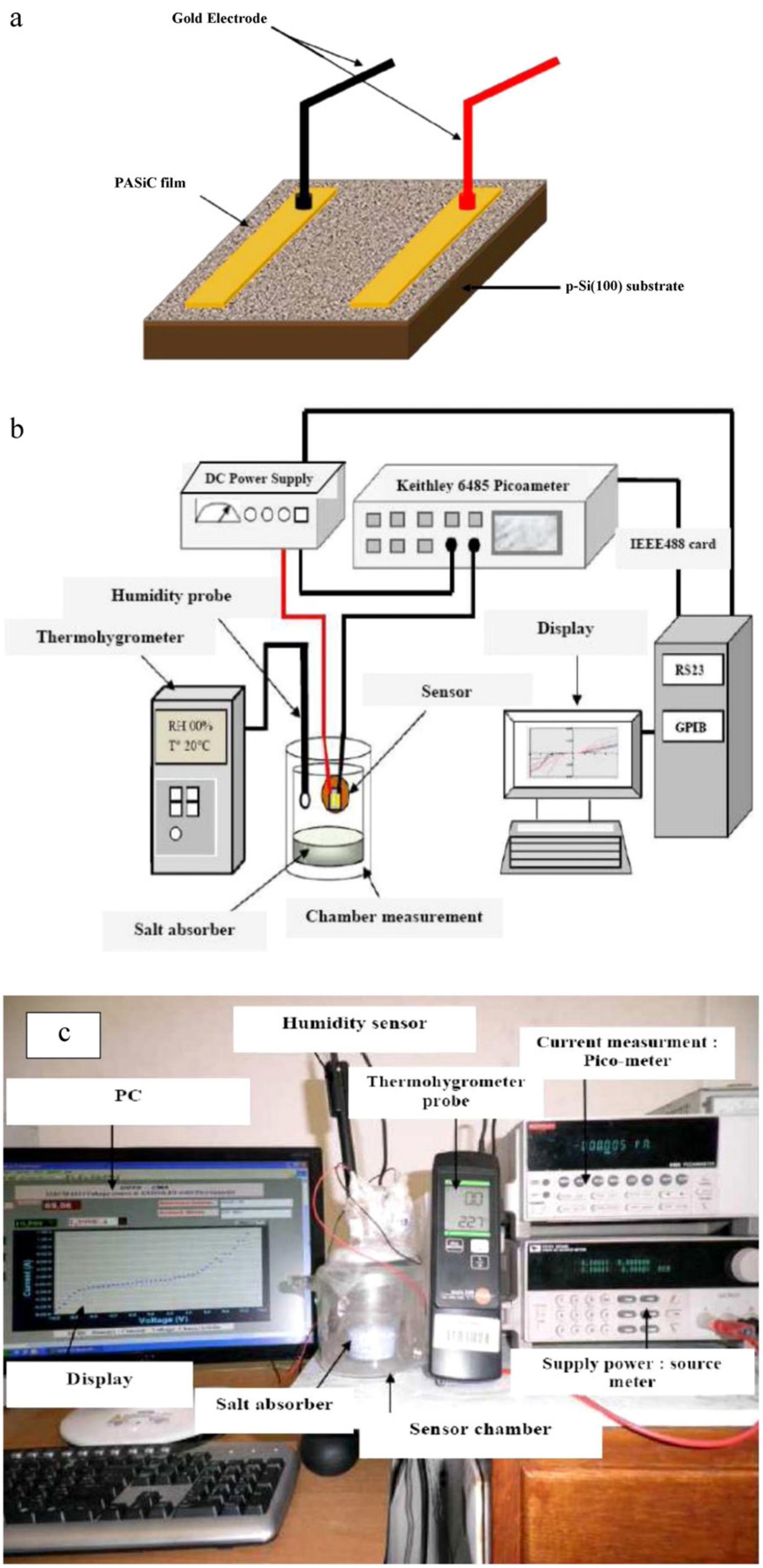

Fig. 2. Illustrate (a) a cross-sectional view of Au–PASiC humidity sensor, synoptic schema of electrical measurements and (b) an automatic of acquirement resistance–RH and resistance–time measurements using a humidity sensor chamber.

Fig. 3. Prototype of Au–Porous a-$Si_{0.70}C_{0.30}$/Si(p) humidity sensor.

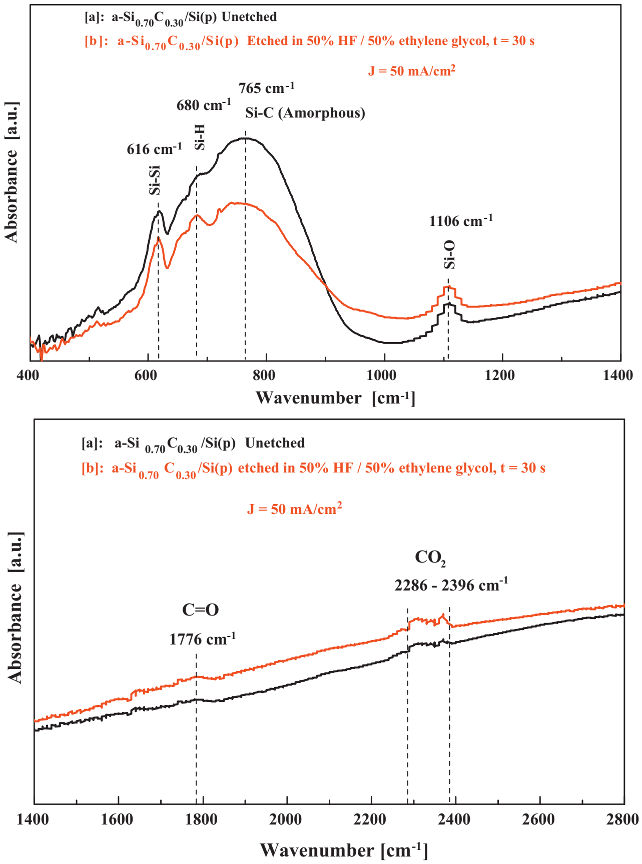

Fig. 4. FT-IR spectra of unetched and anodized amorphous thin a-$Si_{0.70}C_{0.30}$ layer in HF/ETG (1:1 by vol.) solution at 50 mA/cm^2 for 30 s: (a) spectral range 400–1500 cm^{-1},and (b) spectral range 1400–2800 cm^{-1}.

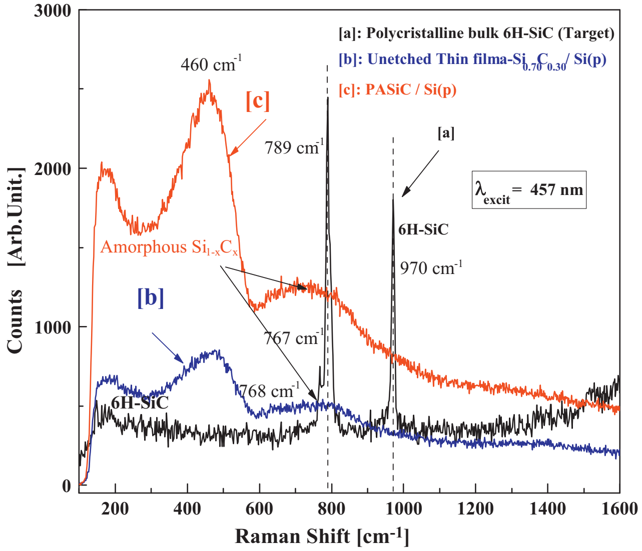

Fig. 5. Raman spectra of unetched and anodized amorphous thin a-$Si_{0.70}C_{0.30}$ layer in HF/ETG (1:1 by vol.) solution at 50 mA/cm^2 for 30 s.

Fig. 5 depicts RAMAN spectra of thin a-$Si_{0.70}C_{0.30}$ films deposited on Si substrate by DC magnetron sputtering using 86 sprigs of silicon carbide (6H-SiC) bulk as target. Other authors as Y. Inous et al. [11] have used plasma CVD and reactive sputtering with a mixture of SiH_4 and CH_4 gases and the elaborated films have a structure a-Si_xC_{1-x}, where x was determined by a ratio SiH_4/CH_4.

In this work, we varied the number of 6H-SiC sprigs from 32 to 86. There is an obvious broad band in the region of 300–600 cm^{-1}. The Raman band of amorphous silicon is attributed to Si—Si bonds. A band associated with Si—C bond is expected to be observed in the region of 600–1000 cm^{-1} because the infrared absorption band due to Si—C bond is located at 765 cm^{-1} [12,13] and a-SiC which was made by the carbon ion implantation into a silicon substrate has an absorption band in the region of 700–725 cm^{-1} [14]. The Raman bands of Si—C bonds for a-SiC which was produced by ion bombardment into crystalline SiC are much broader than those for crystalline SiC and are located at the same frequencies as those for SiC crystal [15]. The Raman spectra for the samples with $x > 0.5$ show weak bands in the region of 700–980 cm^{-1}. A sharp peak observed at 460 cm^{-1} in Fig. 5 arises from silicon crystal substrate.

Fig. 5(a)–(c) shows Raman spectra of polycrystalline bulk 6H-SiC, unetched thin a-$Si_{0.70}C_{0.30}$/Si(p) film and anodized thin a-$Si_{0.70}C_{0.30}$/Si(p) film in HF/ETG (1:1 by vol.) solution at 50 mA/cm^2 for 30 s, respectively. The primary peaks in polycrystalline bulk 6H-SiC are two planar transverse optical modes of E2 symmetry at 768 and 788 cm^{-1}, and an A1 longitudinal optical phonon at 966 cm^{-1} [16,17]. The mode at 796 cm^{-1} is a planar optical mode of E1 symmetry. For the investigation procedure we first identified an area on the sample surface which can be considered representative of the overall surface topology of the sample. Then Raman spectra were collected over such an area. As can be seen from Fig. 5, the Raman spectra look very different as a function of the sampling point, reflecting different local structural arrangements. The primary peaks in 6H-SiC (Fig. 5(a)) are two planar or transverse optical modes of E2 symmetry at 768 cm^{-1} and 789 cm^{-1}, and a longitudinal optical phonon at 970 cm^{-1}. The spectral features presented in spectra b and c are evident: a band at 480 cm^{-1} which arises from amorphous silicon; a relatively strong and broad band located at around 800 cm^{-1} is typical for amorphous SiC configuration, a weak peak at about 1410 cm^{-1}, seen only on etched a-$Si_{0.70}C_{0.30}$ films is due to sp^2 amorphous carbon [18]. One can note that the etching of amorphous SiC films increases the amorphous character of the films. The corresponding shapes are typical of disordered or amorphous compounds, with in place of the sharp lines of 6H-SiC, some broad band's characteristic of phonon density of states due to the release of the zone-center Raman selection rules.

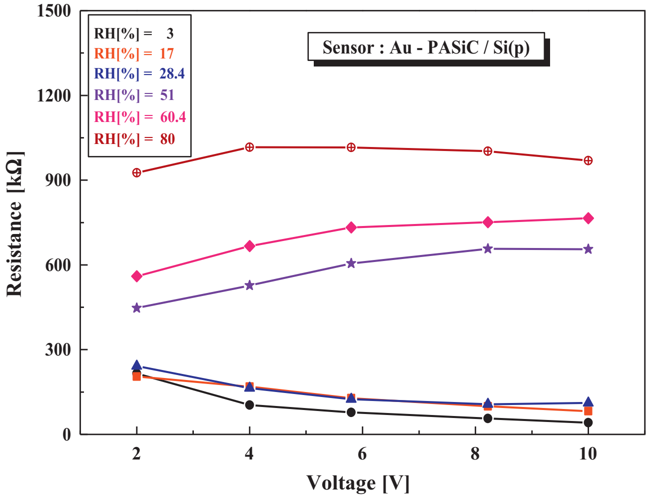

Fig. 6. Resistance versus voltage at different RH[%].

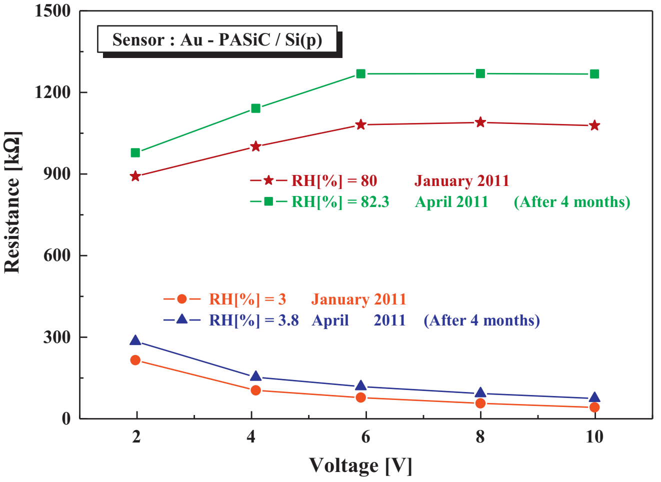

Fig. 8. Resistance versus voltage at different RH[%] just elaborated and after 4 months.

Nevertheless, the shapes displayed in Fig. 5 do not look similar to amorphous SiC, obtained for instance by ion implantation [19–22]. The amorphous bands seen here look more analogous to amorphous Si [23] consistent with a carbon-deficient a-$Si_{1-x}C_x$ ($x < 0.5$) composition.

3.2. *Sensor testing: resistance–voltage and resistance–RH*

Fig. 6 shows the variation of resistance of Au–PASiC/Si(p) sensor structure versus bias voltage for different percentage of relative humidity (RH), It shows that for low RH below 50% [from 3% to 50%], the resistance decreases with an increase of the bias voltage. On the contrary, for high RH level above 50%, the resistance value remains virtually constant; this can be explained by the fact that the pores are saturated by excess of the water molecules (moisture) which are stored in the pores, consequence of the dimension of the pore size and then the purging time or recovery can vary from few minutes to few hours. In the same case, Fig. 7 depicts the resistance variation function of relative humidity (RH) at different bias voltage, where we can observe the resistance varies exponentially with RH; and now, there is no explanation in the literature of this variation type. Also, when RH increases therefore there has a lot of molecules of water, increasing number of water molecules induces obstruction of the pore and then the resistance increases implies a decreases of electrical conductivity, the value of 50% is governed by the value of the pore diameter that saturates itself therefore they become insulating and the current does not pass.

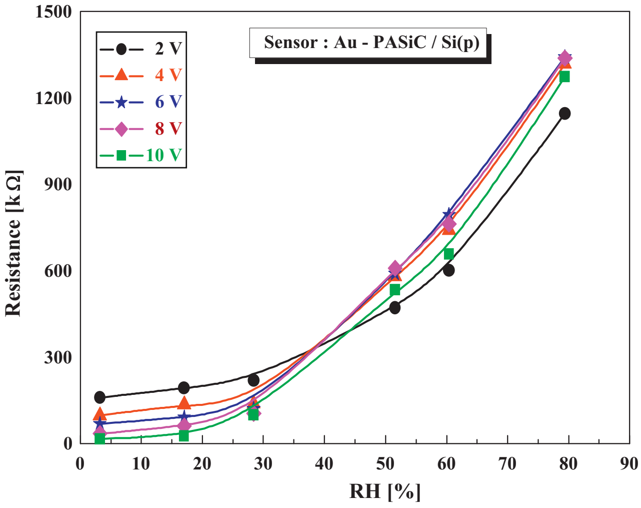

Fig. 7. Resistance versus RH of Au–PASiC/Si(p) sensor.

The results presented here suggest that the undulate surface brought by the macroporous structure of the sensor would greatly increase the sensing area and would provide spacious rooms for vapour adsorption and condensation through capillary effect. Therefore, the Au–PASiC/Si(p) structure might be a favorable humidity sensing material. The variation of the resistance with the relative humidity RH can be related to the modification of the conductivity of PASiC in the presence of water molecules. When dry sample is kept in contact with humid air, water molecules chemisorbs on the available sites of the surface, by a dissociative mechanism to form two hydroxyl ions for each water molecule. The hydroxyl group adsorbed on the surface possesses high local charge density and strong electrostatic field, and the proton reacts with an adjacent surface O^{2-} group to form a second OH^- group as charge carriers, as suggested by Su et al. [24]. When the first layer of water molecules is formed, subsequent layers of water molecules are physically adsorbed on the first hydroxyl layer. The physisorbed water easily dissociates to form H_3O^+ due to the high electrostatic fields in the chemisorbed layer. However, the water molecules in the first physisorption layer, which are doubly bonded to two surface hydroxyls [25], cannot move freely. As suggested by several authors, the water molecules in other physisorption layers are only singly bonded together by hydrogen bonds and therefore much easier to be polarized under external electric field, which would result in a significant change of the dielectric constant and resistance [24,26,27]. The water condensation leads to a liquid-like layer and forms electrolytic conduction. These processes result in progressively decreasing the resistance of Au–PASiC/Si(p) structure. In addition, water is a polar molecule and has a relative long relaxation time [28]. It has been suggested that with increasing bias voltage, water molecules cannot completely follow the change of external electric field, which causes the decrease of the PASiC resistance.

Fig. 8 illustrates variation of resistance versus voltage at different RH for a sensor having just been prepared and after storage for 4 months. As we can see in Fig. 8, a little deviation in the sensor resistance was observed between the first measurements (in January) and after 4 months, probably due to the little difference in the value of RH environments.

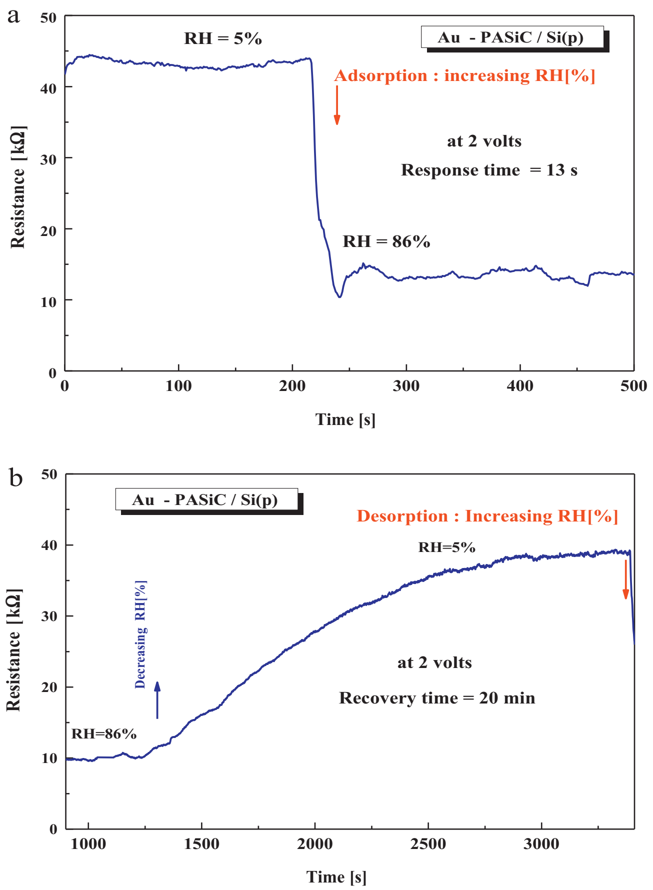

Fig. 9. Response/recovery time characteristics of Au–PASiC/Si(p) sensor.

3.3. Response time

Fig. 9 depicts the response/recovery curves of Au–PASiC/Si(p) structure for RH varying from 5 to 86% and from 86 to 5%. As it can be seen in Fig. 9(a) and (b) the calculated response and recovery times are found about 13 s and 20 min, respectively. Moreover, the recovery time is relatively longer than that of sensors based on traditional porous silicon (PSi) [6]. It is well known that, the capillary condensation and chemisorptions will occur in pores with a radius below the Kelvin radius [29], which is defined as:

$$\gamma_r = \frac{2\gamma M}{\rho RT \ln(P_s/P)} \tag{1}$$

γ: surface tension. P: vapour pressure. P_s: vapour pressure at saturation. M: molecular mass of water. ρ: density of water. R: gas constant 8.314. T: temperature test

This phenomenon will induce a recovery time. The Au–PASiC/(Si(p) sensor has a response time value in the same order of that of three sensors based on porous silicon (PSi), porous polysilicon (PPSi) and porous SiC (PSiC), with a response time in the order of a few seconds [6,30]. The observed different times between adsorption and desorption processes can be explained by the capillary condensation and chemisorption of water into the porous SiC structure.

The capillary condensation and chemisorption of water are the main reason for different times between adsorption and desorption processes. It is well known that desorption of the chemisorbed layer is not further affected by exposure to humidity once formed; it requires a higher energy compared with physisorption layer. Note that, the capillary condensation occurs when the pore size is equal or up to radius r_k given by the Kelvin equation (Eq. (1)), the willingness desorption is determined by a pore radius $2r_k$ [30,31]. Too small pore size goes against water molecule transmission and lengthens response time. With the changing of RH the times become longer gradually. As shown in Fig. 9, the recovery times are obviously longer than that of sensors based on traditional porous silicon [7]. In Ref. [7], porous silicon has the fastest response times among three sensors based on porous silicon, porous polysilicon and porous SiC, with the response and recovery times of the order of 2 min. The porous layer PASiC formed using HF:ETG shows a much more uniform distribution of pores. Larger pores are visible; furthermore, the surface appears slightly rough in the region of the larger pores. Wide distribution of pore sizes with a large percentage of macropores allowing a high sensitivity, and also a number of larger pores showing a fast response time. The sensitivity (S) can be expressed by the variation of the capacity of the sensor with Au–PASiC with RH, it is given by:

$$S = \frac{C_{RH} - C_{min}}{C_{min}} \times 100\% \tag{2}$$

and

$$C_{RH} = 0.089 \frac{S}{4d.RH}(\varepsilon_{SiC}) \left[1 + RH \frac{\varepsilon_{H_2O} - \varepsilon_{SiC}}{\varepsilon_{H_2O} + 2\varepsilon_{SiC}} \right] \tag{3}$$

S: sensor surface (0.5 mm^2). d: distance interdigital gold (1 mm). RH: Relative humidity [%]. ε_{SiC}: SiC dielectrical constant (9.7). ε_{H_2O}: water dielectrical constant (80).

Let us recall that variation of resistance and of capacity with RH can be attributed to the change of the average permittivity (ε_{H_2O}) of the sensitive layer as PASiC [32,33], caused by the water molecules (ε_{H_2O}) adsorbed in the pores. It is known, that the permittivity of water (ε_{H_2O}) is 80 while that of SiC (ε_{SiC}) is 9.7, respectively. A great difference between these permittivities allowed the possibility to detect various RH with a high sensitivity using like amorphous porous SiC sensitive material [34].

The sensitivity (S) depends closely on morphology and the structure of the sensitivity film (PASiC). The dependence of the sensitivity to the regard the signal applied (polarization of the sensor) can be explained by the behavior of polarization of the water molecules. The polar molecules need a long relative time of relaxation to follow the alternate electric field applied [29]. At high frequencies (1 MHz), it is difficult to catch up with the polarization of the water molecules compared to the alternative speed of the external field electric, which leads to the reduction in the permittivity and thus to the reduction in the capacity of device. Reduction in the contribution of the water molecules to the capacity involves a reduction in the sensitivity of the sensor Au–PASiC/Si(p) to the high frequencies. The adsorption of the water molecule on a full surface could be classified like chemical sorption and physical absorption, and the vapor condensation by the capillary action is produced under the conditions given by the equation of Kelvin.

In this work, we have not added the capacitance measurement; it is our future preoccupation, because there are many discussions. The new paper treats a study of the sensitivity versus the pore size (morphology pore) and as soon as it will be subjected to publication in Sensor and Actuators B: Chemical.

4. Conclusion

We have successfully fabricated a RH sensor using PASiC films with interdigital gold electrodes. As a characteristic of SiC material, the elaborated sensor based on SiC can be used in harsh environments. A macroporous PASiC film has been elaborated by anodization with a porosity value of around 60–65% and an average pore diameter of 90 nm, the Fourier transform infrared spectroscopy (FT-IR) revealed several vibrations bands, where the peaks centered at 616 and 765 cm^{-1} correspond to Si–C band for amorphous film by report to 810 cm^{-1} of the crystalline SiC film. Also, Raman spectra reveals a band centered at 480 cm^{-1} which arises from amorphous silicon; a relatively strong and broad band located at around 800 cm^{-1} is typical for amorphous SiC configuration, a weak peak at about 1410 cm^{-1}, seen only on etched a-$Si_{0.70}C_{0.30}$ films is due to sp^2 amorphous carbon. One can note that the etching of amorphous SiC films increases the amorphous character of the films. The elaborated Au–PASiC/Si(p) sensor structure remain hydrophobic at room temperature. The humidity behavior of Au–PASiC/Si(p) sensor was studied by measuring the response time and the changes of resistance versus RH and bias voltage, variation of resistance versus time caused by the water adsorption. It has been shown that the measured resistance of the structure highly depends on the applied bias voltage. Moreover, it has been demonstrated that the response signal against RH is linear for an applied potential of 2 V. The result is interesting and different to that obtained by several authors where a nonlinearity of the response signal is generally observed. The response time is found around 13 s. The preliminary humidity sensor measurements for Au–PASiC/Si(p) as a structure gave some promising results. The changes of electrical resistance in Au–PASiC/Si(p) sensors were remarkable. The effect of temperature on the resistance of the sensor with varying temperature is in progress in a future detailed paper.

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