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Abstract

Pd/SiC Schottky diodes detect hydrogen and hydrocarbons with high sensitivity. Variation of the diode temperature from 100°C to 200°C shows that the diode sensitivity to propylene is temperature dependent. Long-term heat treating at 425°C up to 140 hours is carried out to determine the effect of extended heat treating on the diode properties and gas sensitivity. The heat treating significantly affects the diode's capacitive characteristics, but the diode's current carrying characteristics are much more stable with a large response to hydrogen. Scanning Electron Microscopy and X-ray Spectrometry studies of the Pd surface after the heating show cluster formation and background regions with grain structure observed in both regions. The Pd and Si concentrations vary between grains. Auger Electron Spectroscopy depth profiles revealed that the heat treating promoted interdiffusion and reaction between the Pd and SiC that broadened the interface region. This work shows that Pd/SiC Schottky diodes have significant potential as high temperature gas sensors, but stabilization of the structure is necessary to insure their repeatability in long-term, high temperature applications.

INTRODUCTION

Hydrogen and hydrocarbon detection at elevated temperatures over a wide range of concentrations is important for a number of aeronautical and commercial applications. NASA Lewis Research Center and Case Western Reserve University (CWRU) have been developing high temperature hydrogen and hydrocarbon gas sensors using silicon carbide (SiC) as the semiconductor. The wide bandgap of SiC allows device operation at high temperatures. Recently, it has been shown that Schottky diodes composed of palladium (Pd) on SiC can sensitively detect hydrogen and hydrocarbon gases at 400°C (Hunter et al., 1995). In order for such a sensor to be of use in aeronautical and commercial applications, issues such as the dependence of sensor response on operating temperature and the effects of long-term high temperature operation must be addressed.

This paper discusses the electrical response and interfacial properties of Pd/6H-SiC Schottky diode gas sensors exposed to a variety of ambients and temperature conditions. First, the effect of operating temperature on sensor sensitivity to a hydrocarbon (propylene) is discussed. These studies suggest a range of possible operating temperatures above 200°C. The sensor then is heated to a temperature above this operating range for an extended period to determine the effects of long-term heat treating. The effects of this heat treatment are examined in two ways: 1) The characterization with time of the diode's electronic characteristics. 2) The examination of the surface and interfacial properties of the diode using Auger Electron Spectroscopy (AES), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Spectrometry (EDS). These studies show that although significant reaction occurs between the Pd and SiC, the diode is still operational as a sensitive gas sensor.

DEVICE FABRICATION AND TESTING

Diode sensors composed of Pd deposited on 6H-SiC were fabricated using the same procedure as discussed in Hunter et al., 1995. Approximately 400 angstroms (Å) of Pd were magnetron sputter deposited onto as-grown 6H-SiC epilayer surface and patterned by a lift-off technique into circular Pd Schottky patterns of diameter 200 μ m. A backside contact was formed by sputtering Al onto the bottom of the wafer. The samples were placed in a test chamber and mounted on a heated sample stage with a probing station. Sample contact was made using tungsten probes. The temperature of the heated sample

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stage was controlled from 100°C to 425°C. Computer controlled mass flow controllers supply gaseous hydrogen (H₂), helium (He), nitrogen (N₂), hydrocarbon containing mixtures, or air, either individually or as a mixture, to the test chamber.

The electrical properties of the Pd-SiC Schottky diodes were determined using capacitance and current measurements. The characterization of the diode's capacitance was conducted as follows: The response of the diode capacitance measured at 0 V with time (C-t) was used to characterize the time dependent behavior of the system upon exposure to various gases. The barrier height of the diode was determined using capacitance-voltage (C-V) measurements by finding the intercept on the voltage axis of the plot 1/C² vs V. Corresponding current-time (I-t) and current-voltage (I-V) measurements were also taken in separate tests. The forward voltage across the diode was held constant and the current was measured as a function of time for the I-t measurements. The forward voltage was chosen in order to maximize the diode response to the hydrogen-bearing gas and to minimize series resistance effects. The barrier height as measured by the current is determined by curve-fitting the exponential region of the I-V measurement and using the results of that fitting in the thermonic emission model (Sze, 1981).

TEMPERATURE DEPENDENCE OF SENSOR RESPONSE TO PROPYLENE

The ability of SiC electronics to function at high temperatures allows SiC diode or capacitor-based sensors to operate at temperatures at which hydrocarbons dissociate (Arbab, 1993). The dissociation of these hydrocarbons on the gate of the electronic device, which would not occur at lower temperatures, allows their detection by the electronic device (Baranzahi et al., 1995; Hunter et al., 1995). However, no studies have been done on the operating temperature of Pd/SiC Schottky diodes for the detection of hydrocarbons. We have studied the response of a Pd/SiC Schottky diode to one hydrocarbon, propylene, at a range of temperatures. Such studies help define the operating temperature of the sensor, and thus its long-term stability.

Figure 1 shows the zero bias capacitive response of the sensor to 360 ppm propylene (C_3H_6) at various temperatures. The sensor temperature is increased from 100 °C to 400°C in steps of 100°C and the response of the sensor is observed. At a given temperature, the sensor is exposed to air for 20 minutes, N_2 for 20 minutes, 360 ppm of propylene in N_2 for 20 minutes, N_2 for 10 minutes, and then 10 minutes of air. There are two points to note in the sensor behavior as shown in

Figure 1. First, the baseline capacitance in air does not change between 100°C and 200°C, but decreases to a lower value as the temperature is raised to 300°C and above. This decrease in capacitance as the sensor is heated above 200°C is consistent with the results reported in Hunter et al., 1995. Both results suggest the possibility that temperature dependent chemical reactions which affect the electronic properties of the diode may have an onset at temperatures above 200°C. Second, Figure 1 clearly shows that the magnitude of sensor response to 360 ppm propylene depends strongly on the operating temperature. The data can be understood by assuming that the reaction mechanism is based on the dissociation of propylene on the catalyst surface (Baranzahi et al., 1995; Hunter et al., 1995). A sensor operating temperature of 100 °C is too low for propylene to dissociate on the Pd surface, so the device does not respond at all. The three other curves for 200°C, 300°C, and 400°C show that elevating the temperature increases the sensor's response to propylene. The presence of propylene can be

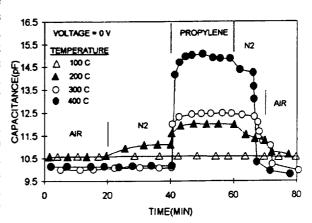


Figure 1. The temperature dependence of the zero bias capacitance to various gas mixtures.

detected at any of these temperatures with 200°C being the minimum operating temperature determined in this study.

EFFECTS OF LONG TERM HEAT TREATING

Electronic Properties

In order to examine the behavior of the sensor in long term operation, the sensor was heat treated at a temperature (425°C) significantly above the minimum operating temperature of 200°C suggested in the last section. An as-deposited diode was first operated at 100°C to establish the baseline electronic properties (Figure 2). The sensor was exposed to 20

minutes of air, 40 minutes in He, 40 minutes of 1000 ppm H₂ in He (He/H₂ mix), 10 minutes of He, and then 10 minutes of air. C-t measurements of the sensor's behavior were taken during the gas exposure. C-V measurements were taken in air and in He before the exposure to the He/H₂ mix, and while the diode was in the He/H₂ mix. In separate measurements, I-t and I-V data was also taken under the same conditions of temperature and gas exposure (Figure 3). After the baseline condition was established, the diode was heat treated at 425°C in air for periods of at least 13 hours at a time. The diode temperature was then decreased to 100°C and at least 1 hour elapsed before measurements were taken. The diode was exposed once after heating to the He/H₂ mix to assure reproducible behavior before characterization of the diode occurred (Hunter et al., 1995). The characterization of the diode was carried out in the same manner used to establish the baseline: C-t and C-V, or I-t and I-V, properties are measured during the timed exposure to air, He, He/H₂ mix, He, and air. This cycle of heating followed by diode characterization at 100°C and was repeated several times until the total time of exposure at 425°C in air was 140 hours. The heating period was usually near 17 hours but an extended heat period at 425°C of 58 hours was performed to see the effect of continuous heating on the diode.

The diode's capacitive properties are strongly affected by the heat treating. Figure 2 shows the diode's C-t responses to the different gas ambients both before and after heat treating at 425 °C for 140 hours. There are two different scales in this figure corresponding to before and after heating. The C-t response before heating shows no capacitance change in the He environment with a large change of capacitance (near 3pF) upon exposure to the He/H₂ mix. This change of capacitance corresponds to a change in barrier height of 550 mV as measured by the C-V data. After the He/H₂ mix exposure, recovery

of the capacitance in He is slow with a much more rapid recovery in air. After heating, the baseline capacitance in air is two orders of magnitude less than that before heating. There is no measurable change in the capacitance upon exposure to He or the He/H₂ mix. The corresponding C-V curve yields a value of the barrier height using the thermonic emission model greater than the 3.0 eV bandgap of SiC. This value for the barrier height is clearly nonphysical. It is likely that the capacitance in the after curve is dominated by noise: this explains the lack of response to the He/H₂ mix and the nonphysical barrier heights.

It should be noted that the diode continued to respond in the capacitive mode with reasonable values for the barrier height until after the extended heating period (total heating time = 125 hours). The capacitance as measured after 125 hours was near 0.3 pF but recovered to 6.6 pF after exposure to the He/H₂ mix. Further heating at 425°C until 140 hours left the capacitance at near 0.2 pF. This capacitance did not recover to higher values even after several He/H₂ mix exposures. Therefore, after 140 hours at 425°C in air, the measured diode capacitance was likely dominated by noise and the diode no longer responded as a capacitive gas sensor.

In contrast, the diode's current response to hydrogen was still significant even after heating. In Figure 3, the current at 0.7 V on a logarithmic scale before and after heating is shown. Before heating, the current shows no significant change in the oxygen deficient He environment while the after heating the current increases from the air baseline nearly two orders of magnitude upon exposure to He. Before heating, the current changes from the baseline in air (near 10-8A) by nearly four orders of magnitude in response to the He/H₂ mix. After heating, the current change to same change in environment is about three orders from the baseline in air (near 10-10 A) and

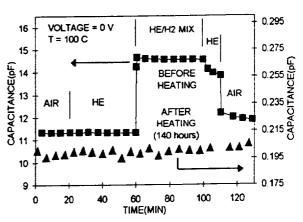


Figure 2. Capacitance at zero bias vs time at 100C before (■) and after (▲) heat treating (140 hours).

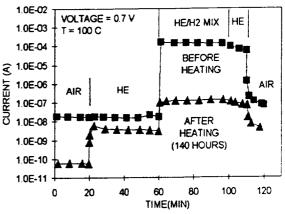


Figure 3. Current at 0.7 V vs time at 100°C before (■) and after (▲) heat treating (140 hours).

about one order from the level in the He environment. In both cases, the current in the He/H₂ mix is significantly larger than that in air. As with the capacitance, the recovery of the diode's current in air towards baseline is much more rapid than that

in He. Therefore, after 140 hours at 425°C in air, the diode current still responded sensitively to the He/H₂ mix and thus the diode still operated as a hydrogen sensor.

The corresponding before and after heating I-V curves are shown in Figure 4. It can be seen that the magnitude and slope of the I-V curves change significantly with heating. corresponding ideality constant in air derived from this curve increased from near 1.1 before heating to near 1.8 after heating. The sensitivity of the diode decreases with heating: a maximum difference between the air and He/H, mix curves is on the order of 105 before heating and on the order of 103 after heating. The barrier heights derived from the I-V curves (Sze, 1981) in air, in the He/H, mix, and the difference between these two values (Air-Mix difference) are shown as a function of heating time in Figure 5. The barrier heights in air and in the He/H, mix decrease after the initial heating and then increase again by the end of the heat treatments. In contrast, the Air-Mix Difference increases initially but then remains relatively constant throughout the heating period. As found for the shorter heating periods examined in Hunter et al., 1995, this data suggests that there are elements of the sensor behavior that are not changed by heating for the periods studied. Nonetheless, significant changes in the sensor behavior occur with heating. In order to determine the physical reason for these electronic changes, the surface and interface properties of the sensor were examined

Surface and Interface Properties

The surface and interface of both an as-desposited (control) sample and the heat treated (annealed) sample discussed in the section above were studied by Auger Electron Spectroscopy (AES). The AES system used for this work is a PHI-590

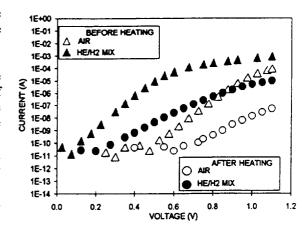


Figure 4. Current vs voltage curves at 100°C before and after heating in air and in the He/H₂ mix.

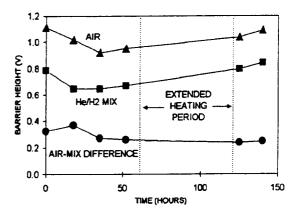


Figure 5. I-V Barrier height vs heating time at 425°C for air (▲), He/H₂ Mix (■) and the difference (●).

scanning AES system with a single pass CMA. An Argon ion beam with 4 KeV beam energy was used for surface sputtering to obtain AES depth profiles. The current density of the sputtering ion beam was 0.02 µA/mm² which corresponds to a sputtering rate on the order of 4 Å of Pd/minute (min). Surface sputtering and acquisition of AES spectrum were conducted periodically. The shape of Si LVV peaks were compared with the shape of Si peaks reported for systems composed of various monolayers of Pd on SiC (Bermudez, 1983).

The surface properties of the control and annealed sample differed significantly. The surface of the control sample contained some common surface contamination such as carbon and oxygen but was free of silicon (Si). The major components of the surface of the annealed sample were Pd, silicon oxide (SiO_x), and palladium silicides (Pd_xSi) with a total AES intensity ratio of Pd to Si of 35/65. Scanning Electron Microscopy (SEM) of the Pd region shows that before annealing the Pd surface was quite smooth. After annealing, SEM of the Pd region shows dense, bright, irregular shaped spots surrounded by a relatively darker background. The typical size of bright and dark regions are 0.1 µm with the area ratio of bright to dark regions being close to one. Further EDS studies indicated that Pd and Si concentrations vary considerably due to this grain structure. Some clusters with diameters from 0.3 to 2 µm were observed on top of the surface, but EDS results did not show any compositional difference between cluster and non-cluster regions. The Pd (Si) concentration was much higher (lower) in brighter grains than that in the darker grains. The SiO_x on the annealed surface may possibly be formed by either Si segregation from the interior of the Pd to the Pd surface or two dimensional diffusion of Si from the SiC surface surrounding the Pd region. In the surrounding SiC region, Si was detected in both oxides and carbide states. The mechanism for this SiO_x formation is still under investigation.

The AES depth profile of the control sample is shown in Figure 6A. The region below the surface of the control sample was predominately Pd with no other constituents until a sputtering time of t ~ 40 min. The silicide features dominated the

Si (LVV) peak at $t \sim 45$ min with the peak shape corresponding to that of 3.5 monolayers (ML) of Pd on SiC. In the region 45 min < t < 95 min, the silicides features decreased gradually with sputtering time. At the time of $t \sim 54$ min, SiC features became dominant and the shape of Si peak was very similar to that reported for 0.5 ML Pd on SiC (Bermudez, 1983). After the sputtering time corresponding to $t \sim 95$ min, the silicide features almost completely disappeared.

The AES data for the annealed sample suggests that the heat treating promoted the reaction between the Pd and SiC forming Pd_xSi. After a very brief sputtering (0.15 min), the silicide features noted on the surface disappeared and the Si peak presented only oxide features. With further sputtering, silicide features were present for a significant depth into the sample.

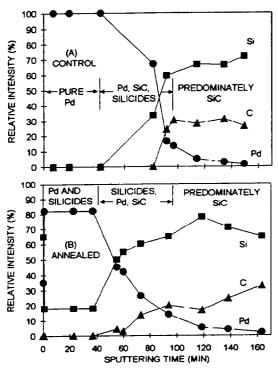


Figure 6. Relative AES intensities of Pd (\bullet), Si (\blacksquare), and C (\triangle) as a function of sputtering time for the control (A) and annealed sample (B).

As shown by Figure 6B, the relative intensity of the Si AES peak was near 18% in the upper part of Pd region (0.4 min < t < 30 min). Both the concentration and the chemical state of Si in this region were quite uniform. The Si peak shape in this region corresponded to the peak shape reported for a system of 3.5 ML Pd on SiC. The differential spectra of Si peaks at 73, 78.5, 84.5, 89, 93.5 eV indicate that the reaction products in this region are predominately Pd,Si. The relative intensity of the Pd AES peak decreases and the Si intensity increases in the region 45 min < t < 95 min. At t ~ 40 min, the shape of the Si peak corresponded to that of 3.5 ML Pd on SiC. With increasing sputtering time, the Si peak at 89 eV became stronger. At t ~ 75.5 min, the shape of Si peak corresponded to that of 1.3 ML Pd on SiC while around $t \sim 81$ min the shape of Si peak corresponded to that of 0.5 ML Pd on SiC. After t ~ 95 min, the only Si peak remaining corresponded to that of Si in SiC at 89 eV.

The interface region, which is defined as the transition region between the near surface region and the substrate, was broadened by the annealing. The majority of the Pd $_x$ Si was distributed in the region of 45 min < t < 95 min for both samples. However, the distribution and concentration of Pd $_x$ Si in the annealed and control samples were very different. This is reflected in the lower slope of the change of the relative intensity for both Pd and Si in the region 45 min < t < 95 min in the annealed sample, as well as the presence of Si throughout the sample (Figure 6B). Further, the concentration of Pd $_x$ Si in the interface region of the annealed sample was higher than that of

the control sample (not shown). Thus the interface region became broader because of the interdiffusion and reaction between Pd and SiC promoted by the heating. The dissociated carbon was always depleted where Pd_xSi formed with relatively rich Pd (45 min < t < 50 min for Figure 6A and 40 min < t < 60 min for Figure 6B). In the substrate region, the Si and C intensities changed slowly with sputtering time never matching the ratio of 1:1 as expected for SiC. This was likely due to preferential sputtering effects.

DISCUSSION AND SUMMARY

Long term operation of Pd/SiC diodes as gas sensors depends on the stability of the device structure at operating temperature. An operating temperature of 200°C has been shown in this work to be sufficient for detection of propylene but higher temperatures may be necessary for other hydrocarbons. Heating the sensor at temperatures significantly above 200°C for extended periods showed definite changes in the sensor properties. The sensor is still operational in the current mode after this heat treating although with decreased sensitivity. The capacitance of the sensor decreased significantly and was insensitive to changes in the hydrogen concentration.

Possible reasons for these changes may be found by examining the surface and interfacial properties of the diode. After heating, SiO_x and Pd_x Si formed on the surface that may have interfered with the probe contact for the capacitance measurements conducted at zero bias. The current may have tunneled through these barriers with forward bias and thus still been measurable. The changes with heating observed in the current response correspond to changes in the diode's surface

and interface. The formation of SiO_x and Pd_xSi on the surface may have interfered with the surface dissociation of hydrogen. Before heating, reactions at the Pd/SiC interface resulted in an interfacial region in which major reaction product is Pd_xSi. This interface contributed to the capacitive and current carrying characteristics initially observed for the diode. The interface broadened after heating with Pd_xSi present throughout the interface region. Since Pd_xSi behaves like a conductor (Rubloff, 1982), the electronic properties of Pd/SiC Schottky diodes are determined, especially after heat treating, largely by (Pd_xSi + Pd)/SiC interface rather than an ideal metal Pd/SiC interface. With this picture of the interface, it can be suggested that the macro-electronic properties of the device are determined by the superposition of all the (silicides + metal mixture)/SiC contacts constituting the interface. The heat treating at high temperature promoted the interdiffusion and chemical reaction between Pd and SiC, and thus changed the spatial distributions (the interface region was broadened) and chemical environment of these effective micro-contacts. It has been reported that the dissociated carbon found near the interface region is basically graphitic (Bermudez, 1983), but the effects of the dissociated carbon on the macro-electronic properties of the device are still not well understood.

It is suggested from these results that the heat treating at high temperature accelerated the interdiffusion and reaction process, and thus changed the spatial distributions and chemical environment of these effective microcontacts. The change in the surface may have decreased efficiency of the hydrogen dissociation. These surface and interfacial changes are significantly responsible for the changes in the sensor response. Nonetheless, it is extremely hopeful that even after these significant changes in diode's structure, the diode is a sensitive hydrogen sensor.

FUTURE PLANS

The future development of the Pd-based sensors using SiC will concentrate on stabilizing the sensor structure. Continued heat treating for even longer periods than discussed in this paper will be attempted to determine if changes in the sensor structure are minimized after a certain heating time. Another area of potential research is to place a thin diffusion barrier layer between the metal and the SiC to limit the interdiffusion.

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