Interface States in High-Temperature Gas Sensors Based on Silicon Carbide

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***Abstract*—Silicon carbide (SiC)-based metal-insulator-semiconductor devices are attractive for gas sensing in automotive exhausts and flue gases. The response of the devices to reducing gases has been assumed to be due to a reduced metal work function at the metal-oxide interface that shifts the flat band capacitance to lower voltages. We have discovered that high temperature (700 K) exposure to hydrogen results not only in the flat-band voltage occurring at a more negative bias than in oxygen, but also in the transition from accumulation (high capacitance) to inversion (low capacitance) occurring over a relatively narrow voltage range. In oxygen, this transition is broadened indicating the creation of a high density of interface states. We present a model of the hydrogen/oxygen response based on two independent phenomena: a chemically induced shift in the metal-semiconductor work function difference and the passivation/creation of charged states at the SiO_2-SiC interface that is much slower than the work function shift. We discuss the effect of these results on sensor design and the choice of operating point.**

***Index Terms*—Gas sensor, hydrogen, interface states, silicon carbide (SiC).**

I. Introduction

THERE is a need for gas sensors in the emissions control of automotive exhaust and flue gases, for both real time monitoring and feedback control. Typical operation conditions are high temperature, chemically reactive environments and low or varying oxygen concentrations. To extend the possible operation temperatures from 500 to above 1200 K, silicon carbide (SiC) has been used as the semiconductor instead of silicon, because the bandgap of SiC, around 3 eV, is much larger than the one for silicon, with 1.1 eV. In addition, SiC is chemically stable, making it well suited for sensing applications in harsh and reactive environments. Metal-insulator-silicon carbide (MISiC) structures have been used as sensors in these environments, with refractory metals as gates. Species that have been monitored with such MISiCs at high temperatures include hydrogen, hydrocarbons, nitrogen oxides, and fluorine containing gases [1]–[8].

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A critical parameter of any field-effect device like the MIS structure is the defect density at the interface of insulator and semiconductor. It should be low in order not to shield the semiconductor from the influence of the gate potential. SiC grows a thermal oxide, as silicon does, and its interface has a small defect density, but still about a decade larger than for silicon. In this article, we characterize the SiO_2-SiC interface during the exposure to oxidizing and reducing gases. We perform two different electrical measurements on the MIS structures: 1) capacitance-voltage (C-V) characterization by measuring the capacitance during bias voltage sweeps and 2) sensor characterization by recording the bias voltage required to hold the capacitance at a preset value during gas exchanges.

It has been assumed to date that the response of MISiCs with catalytic gates to hydrogen containing gases is due entirely to the chemically induced shift in the metal-insulator work function difference [1]. This model of sensor response is based on measurements performed on silicon based MIS structures at temperatures below 500 K [9]. We demonstrate that, at high temperatures (above 700 K) an additional mechanism, the reversible passivation/creation of charged states near the SiO_2-SiC interface contributes to the hydrogen sensitivity.

II. Experimental

Fig. 1 shows a schematic sketch of our MISiC sample. The SiC is n-type with a bulk-doping density of $\sim 5 \times 10^{18}$ cm^{-3}. A low-doped epi layer is grown on the Si-face with a doping density of $\sim 1.4 \times 10^{16}$ cm^{-3}. The SiC wafers are obtained commercially [10], either with or without a thermal oxide. The oxide is grown in dry or wet oxygen, followed by a wet anneal at 950 °C [11]. The samples are then sawed into 1 cm $\times$ 1 cm pieces. We measure the oxide thickness of each sample with spectroscopic ellipsometry, obtaining values between 45 and 47 nm. An accuracy of 0.3 nm is estimated from repeated measurements at different spots of the sample. For the ellipsometry modeling, we use the material constants from Palik [12].

Prior to metallization, we clean the samples in a solution of conc. NH_3, 30%-H_2O_2 and H_2O (1:1:6) at 75 °C for 20 min and then in a solution of conc. HCl, 30%-H_2O_2 and H_2O (1:1:7) at 75 °C for another 20 min. After being thoroughly rinsed and blown dry, metal dots are deposited on the front side of the samples through a shadow mask by sputtering at a temperature of 350 °C and an Ar pressure of 0.33 Pa. The dots consists of 100 nm Pt on top of 2 nm Ti (used as adhesive layer) and have several diameters between 200 and 1000 μm. Each dot is the gate of a MISiC. After covering the front with photoresist, the

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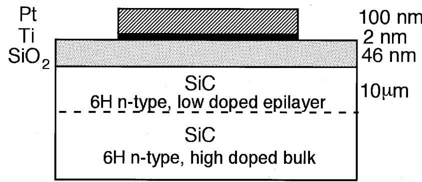

Fig. 1. Schematic of the MISiC device (not to scale). Pt is sputtered on a thermally oxidized SiC substrate with dot diameters from 200 to 1000 μm. The electrical contact to a Pt gate is made with a bonded gold wire (not shown).

oxide on the back is etched away with a buffered HF solution. The photoresist is removed in acetone, and the samples are attached with conducting paste [13] on thin alumina headers with gold pads.

First, we characterize all MISiCs, contacted with probe tips, by measuring their high frequency and quasi-static capacitance voltage (C-V) curves at room temperature. The precise doping density of the epi-layer is obtained from a $1/C^2$ analysis in depletion. We use a capacitance-measuring set-up [14] to measure simultaneously the high frequency capacitance at 1 MHz and the quasi-static capacitance while sweeping the voltage. The voltage is swept with rates between 0.02 and 0.43 V/s. Quasi-static curves are only possible up to a temperature of 500 K due to the leakage currents at higher temperatures. The electrical contact to the MISiCs is made by bonding 1 mil gold wire at 350 °C, with the gold pads on the alumina headers as the first bond and the MISiC gates as the second bond. The gold pads are in turn contacted by probe tips of a probe station or by other bond wires that are attached to fixed contacts in the furnace for gas measurements. The MISiCs are then characterized at temperatures from 300 to 900 K in different gases by C-V curves and by sensor measurements. For the sensor measurements, the capacitance is held constant to keep the Fermi level at the SiO_2-SiC interface fixed and to avoid charging and decharging of interface states. The gases used are nitrogen (purity 99.999%), 10% hydrogen in nitrogen (99.999%), and 1% oxygen in nitrogen (99.99%). The concentrations of the gases are chosen to reach saturated sensor signals, but to avoid explosive gas mixtures. The exposures of the MISiCs to hydrogen and oxygen are separated by short exposures to pure nitrogen, to avoid dangerous mixtures of hydrogen and oxygen in the sensor environment.

III. Results

In Fig. 2, we show the C-V curves of an n-type MISiC capacitor. For positive gate voltages, the device is in accumulation. Majority carriers (electrons) are attracted to the SiO_2-SiC interface, the SiC acts like a metal, and the measured capacitance has its maximal value equal to the capacitance determined by the thickness and dielectric constant of the oxide. As the voltage is decreased in a stepwise manner, the SiC near the interface is depleted of majority carriers, and the depleted region increases in thickness. The total capacitance decreases, because it includes the capacitance of the depleted region in series with the oxide capacitance. Shown in Fig. 2 are the C-V curves obtained in both hydrogen and oxygen. In switching from oxygen to hydrogen the curve is displaced to more negative voltages and has a steeper slope. During sensor measurements, the capacitance is held constant while changing the gas environment, and the corresponding gate voltage is recorded as the sensor signal. Therefore, the sensor response depends on the chosen capacitance set point, with larger responses for larger capacitances. We have indicated the measured gate voltages during two sensor measurements with symbols. At 180 pF the sensor response to a change from hydrogen to oxygen is 0.44 V, while at 80 pF the response is only 0.23 V.

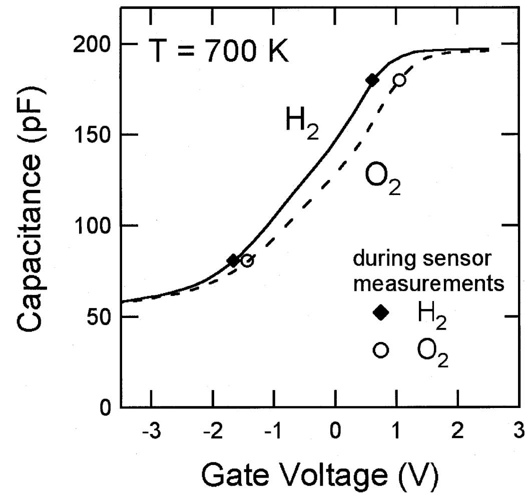

Fig. 2. High-frequency capacitance of a sample with a gate area of 2.5×10^{-7} m^2 and an oxide thickness of 46.5 nm, measured in two gas environments, 10% H_2 in N_2 and 1% O_2 in N_2. The gate voltage is swept from positive to negative voltages at 0.15 V/s, and the high frequency is 1 MHz. Also indicated are the gate voltages during two separate sensor measurements (see Fig. 3) where the capacitance was held at a constant value of 80 and 180 pF, respectively.

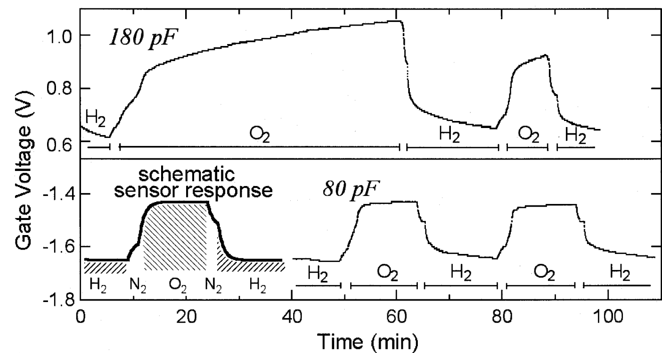

Fig. 3. Sensor measurements on the sample shown in Fig. 2 at 700 K, the capacitance is held constant and the gate voltage is recorded as sensor signal during the gas exchanges. The measurement sequence is shown schematically in the lower left curve: the sensor is exposed to 10% H_2 in N_2 and 1% O_2 in N_2, separated by a short exposure to pure nitrogen. We observe a reversible increase in O_2 and decrease in H_2. In the upper half of the figure, the capacitance is kept at 180 pF, in the lower half at 80 pF.

Fig. 3 shows the two sensor measurements at 180 and 80 pF, taken directly after each other. Each measurement consists of two cycles between hydrogen and oxygen, with short exposures to pure nitrogen in between. For comparison, the sensor is exposed to oxygen in both measurements at a time of around 80 min. Initially, both responses are fast with a similar increase of the gate voltage and a time constant of $\sim$ 1 min, but then the sensor at 180 pF continues to increase slowly with a time constant of $\sim$ 40 min. This makes the use of the sensor at

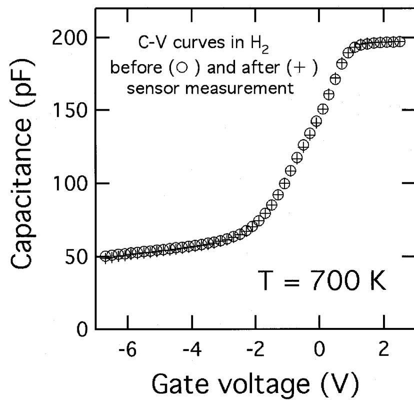

Fig. 4. C-V characteristic of the sensor shown in Fig. 2, showing that the effects of gas exposure are reversible. The device was allowed to equilibrate in a hydrogen environment for 15 min prior to each measurement. The initial C-V curve in H_2 is well reproduced after 4 h of cycling between O_2 and H_2 at 700 K.

180 pF impractical. The sensor response at 180 pF is 0.44 V, including the slow tail in oxygen. The total sensor response at 80 pF is only 0.23 V but reaches saturation faster. Note that the asymptotic values of the sensor signal from Fig. 3 in both hydrogen (diamond symbols) and oxygen (circle symbols) are overlaid on the original C-V curve in Fig. 2. The measured gate voltages between C-V characteristics and sensor measurements agree within 30 mV.

Fig. 4 demonstrates that the effect of gas exposure on the device's C-V characteristic is reversible. We show the C-V curves in hydrogen prior to making the sensor measurement of Fig. 3, and after 4 h of cycling between oxygen and hydrogen. The device was allowed to equilibrate for 15 min in hydrogen prior to each measurement. The difference between the values before and after are below 1% of the maximum capacitance value.

Additional support for the quality of the devices is the low defect density at the SiO_2-SiC interface. From simultaneous quasi-static and 1 MHz C-V curves at 500 K of the sample shown in Fig. 2, we have calculated a defect density at the SiO_2-SiC interface close to 10^{11} cm $^{-2}$ev^{-1} for $0.3 < \text{Ec-E} < 1.1$ eV. The defect density was obtained via the hi-low technique [15], by subtracting the quasi-static C-V curve, which includes the capacitance of the defects in series with the oxide capacitance, from the 1 MHz C-V characteristic, where the ac signal is too fast for the defects to follow. The defect densities of our devices [8] are comparable to state of the art oxides grown under similar conditions [16]. Within our measurement accuracy, the 1 MHz C-V characteristic in hydrogen at 700 K is what we would obtain for a MISiC device with no interface states.

We have observed these effects: 1) in samples at 800 K with Pt-gates evaporated directly on the oxide as opposed to a sputtered Pt/Ti gate [8]; 2) in 4H-SiC as well as in 6H-SiC capacitors; and 3) with gate oxides grown under different conditions [8]. In a few samples, the C-V curves in oxygen even crosses the C-V curve in hydrogen when the sensor was close to inversion [8].

IV. Discussion

At temperatures above 500 K, the device C-V curve is displaced toward negative voltages in hydrogen and toward positive voltages in oxygen. In addition there is a change in shape of the C-V characteristics between hydrogen and oxygen. The response of MISiC sensors to hydrogen containing gases has been attributed to date solely to the formation of a polarized layer at the metal-SiO_2 interface [1], due to the reduction of the metal-SiO_2 work function difference in hydrogen [9]. This "classic" sensor response mechanism accounts for only a portion of the observed sensor responses according to our model in Fig. 5.

Our model, explained in Fig. 5, shows the charge distribution in the sensor that we assume for different gas ambients. For an MISiC device biased at constant capacitance, the potentials in the SiC are constant, regardless of gas exposure. In a hydrogen ambient, the hydrogen is dissociating at the metal surface and diffusing relatively fast into the sensor. Diffusion times less than 5 μs in the platinum gate are calculated from [17] and times less than 0.5 ms in the SiO_2 layer are calculated from [18]. A polarized layer builds up at the metal-SiO_2 interface with the protons in the SiO_2 and the electrons in the metal. The dipole moment per hydrogen atom is larger than 2 Debye [19], indicating a separation of the positive and negative charge densities of the hydrogen. With the polarized layer present, the potential of the platinum decreases in hydrogen, according to Poisson's equation.

In an oxygen ambient, the hydrogen reacts with oxygen on the metal surface and is thereby removed from the sensor. The disappearance of the polarized layer at the metal-SiO_2 interface increases the potential of the platinum. The disappearance of the polarized layer, however, cannot be the only effect of oxygen, because this would lead only to a parallel shift of the C-V curve toward higher voltages and not to a shape change of the C-V curve in Fig. 2. The shape is influenced by the density of electronic states at the insulator semiconductor interface, because the states can partly screen the influence of the gate voltage on the semiconductor. As the interface state density increases, the transition between accumulation to inversion requires a larger change in gate voltage and the slope of the C-V curve in depletion decreases. In oxygen, the C-V curve has a smaller slope than in hydrogen, indicating a higher density of defects at the SiO_2-SiC interface. To explain the changing shape of the C-V curve, we assume that hydrogen can passivate defects at the interface as indicated in our model in Fig. 5. Such a passivation is generally believed to be the reason of the effectiveness of the last hydrogen anneal of silicon field-effect components that need low interface state densities [20]. In an oxygen ambient, hydrogen leaves these states and the states below the Fermi level get filled with electrons, as seen by the direction of the sensor response. A major difference between devices based on silicon and devices based on SiC is that the latter can operate at temperatures where hydrogen passivation or hydrogen removal takes place.

The model can explain the different sensor responses for different pre-set values of the capacitance, shown in Fig. 3. At a pre-set capacitance value of 180 pF, the Fermi level is closer to the conduction band than at a value of 80 pF. The interface state density in our samples is larger near the conduction band edge than at midgap [8], similar to that observed in silicon [21]. Therefore, at 180 pF, more defects take up an electron during oxygen exposure than at 80 pF. This makes the response

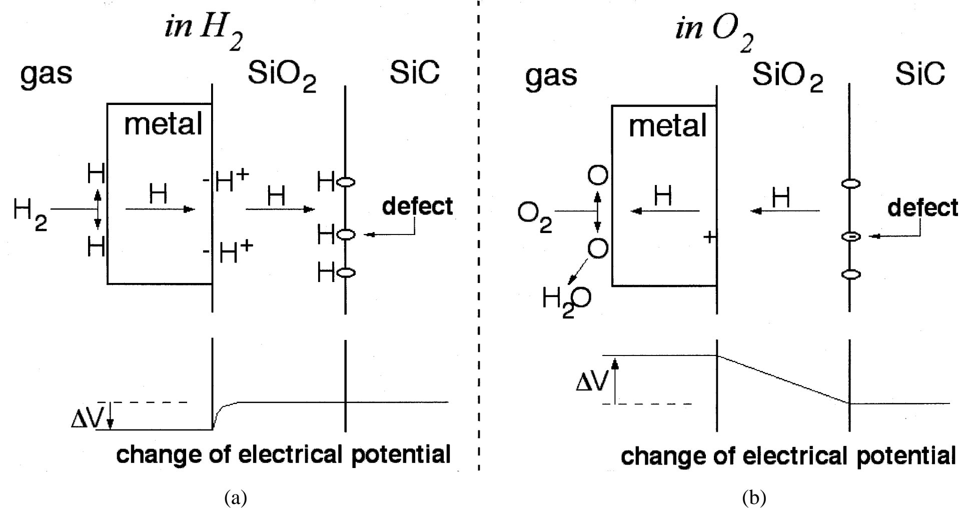

Fig. 5. Distribution of charges in the MISiC sensor during exposure to (left) H_2 and (right) O_2, respectively. When the sample capacitance is kept constant, the charge distribution and the potentials inside the SiC do not change. During H_2 exposure, hydrogen atoms and molecules diffuse into the sample. Some adsorb at the metal-SiO_2 interface with the protons in the SiO_2 and the electrons in the metal, other adsorb at the SiO_2-SiC interface where they passivate interface states and prevent them from charging. The charge distribution at the metal-SiO_2 interface decreases the potential of the metal, and, therefore, the gate voltage decreases in H_2. During O_2 exposure, the hydrogen in the sample is consumed at the metal surface. The states at the SiO_2-SiC interface can now be negatively charged, and the compensating positive charge is in the metal. The potential of the metal increases and, therefore, the gate voltage increases in O_2.

at 180 pF larger; however, the additional portion of it is also slower. The portion of the response due to the work function has the same magnitude and time constant for any capacitance value. We believe that the slow response of the defects is due to the slow desorption of hydrogen from defects it has been passivating. We cannot measure the fast component of the Pt-SiO_2 interface response to hydrogen, because the gas exchange time of our current measurement system is too slow. Other experiments have, however, shown response times in the millisecond range [22]. For most sensor measurements, the slow part of the sensor response is to be avoided.

The effect of the gases on the shape of the C-V curve is found to be reversible, as seen in the unchanged C-V curves before and after the sensor measurement. The interface state density of our devices is comparable to state-of-the-art material that is used for electronic applications. We conclude that the observed shape change is intrinsic to SiC field-effect structures. From our model in Fig. 5, we deduce that the sensor response in oxygen due to charged states at the SiO_2-SiC interface ΔV is proportional to the thickness t of the insulator, divided by its dielectric constant ε, and to the interface charge density $nq : \Delta V = nqt/\varepsilon$. Therefore, a thinner insulator in a MISiC device should reduce the influence of the interface states on the sensor signal.

The reduction of defect density at the SiO_2-SiC interface by hydrogen annealing at high temperatures has been suggested as a method of improving device characteristics at room temperatures [23]. An annealing at 1070 K reduced the density permanently, whereas annealing at 670 K had little or no effect. It has not been clarified yet which types of defects were measured. Since we observe an annealing effect during *in situ* C-V measurements at tempeatures of 700 K, we are probably observing a different type of defect. There have also been recent reports on the improvement in the room temperature interface state density of n-type 4H-SiC capacitors using forming gas ($N_2/5\%H^2$) annealing up to 1270 K [24].

For optimum sensor performance, what capacitance set point should be chosen? The choice of capacitance set-point determines the position of the Fermi level in the SiC bandgap at the SiO_2-SiC interface, and the position of the Fermi level determines which kind and how many defects will take up or release electrons during oxygen exposure. In the SiO_2-SiC system, the defect density decreases as the Fermi level is swept from the conduction band edge to midgap. When the Fermi level is near mid gap, the contribution due to interface states with long time constants are reduced and the work function change dominates the sensor response, as shown in Fig. 3. The precise value of the interface state density is very sensitive to oxidation conditions and post oxidation processing parameters [20]. In order to obtain repeatable results from independently fabricated devices, the effect of these states needs to be minimized. Therefore, we propose that the optimum bias point in terms of sensor response time and sensor to sensor repeatability is near midgap.

V. Conclusion

The response of MISiC sensors to oxidizing and reducing ambients at temperatures above 700 K is due to two independent phenomena: the familiar chemical modification of the metal-oxide work function difference and the creation/passivation of the interface states, an effect we have identified here for the first time. For n-type SiC devices, we have shown that the sensor response due to interface states, primarily in the upper half of the band gap, has a significantly larger time constant than the response due to change of the work function. For an MIS capacitance sensor operating in the constant capacitance mode, the capacitance set point determines both: 1) the magnitude of the sensor signal and 2) the relative contribution of each mechanism toward the sensor response. The optimum set point for MISiC sensors with respect to response time and sensor-to-sensor repeatability is close to midgap. These results are applicable to

field-effect SiC sensors in general, independent of sample design.

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