Stress and thermal characterization of 4H-SiC microelectromechanical structures

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Abstract—In this letter, the potential of single crystalline 4H-polytype silicon carbide (4H-SiC) based microelectromechanical structures as resistance thermometer for high temperature sensing were explored. A dopant-selective photoelectrochemical etching process was applied to release the sensing element - suspended microstructures on 4H-SiC substrate. Residual stress and stress gradient in the microstructure before and after release was examined by micro-Raman spectroscopy. Electrical resistance of the suspended microstructures at different temperatures were characterized and analyzed by a temperature-dependent electron mobility model.

Index Terms—Silicon carbide; microelectromechanical structure; micro-Raman; temperature sensing; resistance thermometer.

1. Introduction

Single crystalline 4H-SiC is a desirable material for electronic devices and microelectromechanical devices operating in hostile environments such as high temperature, high pressure and corrosion due to its high thermal robustness, large Young's modulus, radiation and chemical hardness, etc. 4H-SiC electronic devices as high temperature sensors have been studied, such as pn diode [1, 2], Schottky diode [3, 4], FET [5, 6], etc. The suspended microstructures in microelectromechanical devices also have great potential as the sensing element in resistance thermometer by monitoring the temperature-dependent resistance. By forming a thermally well-isolated air gap, the suspended structure reduces the thermal contact with surroundings and thermally conductive substrate, which concentrates heating more on the device. This advantageous design combined with the high temperature stability of 4H-SiC will ensure excellent

thermal sensitivity and enables a wide range of operation temperatures. 4H-SiC has homoepitaxial single crystalline SiC film grown on single crystalline SiC substrate. Due to the extreme chemical resistance of 4H-SiC, it is challenging to undercut the material to release microelectromechanical structures by conventional isotropic chemical etching process. A dopant-selective photoelectrochemical etching process has been developed [7, 8] to solve this challenge and fabricate suspended microstructures to form actuator and resonator devices. The residual stresses and stress gradients in the released structures can have a critical effect on the functionality and reliability of the devices, for example, device failure due to curling, buckling, or fracture at high temperatures especially. To exploit the full advantages of 4H-SiC based microelectromechanical devices, it is important to characterize and therefore optimize the photoelectrochemical etching process to release low-stress or stress-free suspended structures. Among the different techniques, micro-Raman spectroscopy has been shown to be effective to measure the residual stresses and map the stress gradients in Si microstructures [9, 10].

In this letter, the potential of 4H-SiC microelectromechanical devices was explored as resistance thermometer for high temperature sensing. The suspended clamped-clamped beams as the heating elements were released by the dopant selective photoelectrochemical etching process. Micro-Raman spectroscopy was applied to characterize the residual stress and stress gradient in the suspended microstructures. The resistances of the sensors at different temperatures were measured. Experimental data were compared with analytical results from a temperature-dependent mobility model.

2. Experimental

Suspended 4H-SiC microstructures were first patterned by conventional fabrication processes, and then released by photoelectrochemical etching. The starting material was an n-p homoepitaxial structure grown on a 4H-SiC n-type conductive substrate. The top n-layer was released to form the suspended resistor, and its thickness determines the thickness of the resistor. The middle p-type layer acted as the sacrificial layer in photoelectrochemical etching to release the microstructure, and also electrically isolated the suspended n-SiC resistor from the bottom n-SiC substrate. Layout of the microstructures was patterned by a reactive ion etching with etch mask formed by photolithography, nickel deposition and lift-off. Special care was taken to make sure the top n-SiC layer was etched through. After removing the etch mask by nickel etchant, the sample was cleaned thoroughly by $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ (1:1) solution, followed by another photolithography, nickel deposition and lift-off on the front of the wafer. A rapid thermal annealing process was carried out at 950°C for 60 sec in Ar ambient to transform nickel to Ohmic contacts. Photoelectrochemical etching was applied to release the top n-SiC layer to form suspended microstructures. The principle of dopant-selective photoelectrochemical etching of 4H-SiC has been explained elsewhere [7, 8]. The wafer was immersed in a dilute (5% by weight) potassium hydroxide (KOH) solution. The KOH solution was connected through a platinum wire to the cathode of a DC power supply. The backside of the wafer was isolated from KOH and connected to the anode of the DC power supply. A UV light source from a fluorescence microscope exposes the wafer surface. When a proper DC bias voltage was applied, the top n-SiC layer was intact while the underneath p-SiC sacrificial layer was etched, from which the top n-SiC layer was released. In this experiment, the DC voltage was kept at

0.5V~0.6V to keep the current between 50 mA and 60 mA. The higher current increases etching rate, but roughens the etched surface. The process flow is shown in Fig. 1.

Fig. 1. Fabrication process of 4H-SiC suspended microstructure. (a) Start material, (b) patterning and metal contact formation by photolithography, metal mask lift-off and RIE etching, (c) final suspended structure after photoelectrochemical etching.

3. Results and discussion

Fig. 2(a) shows the SEM image of a 50 μm -long, 5 μm -wide and 1 μm -thick clamped-clamped beam fabricated in this study. The image shows that photoelectrochemical etching did not leave obvious damage to the suspended structures. Micro-Raman measurements were performed using a LabRAM HR Raman confocal microscope by Horiba Scientific to characterize the stress or strain in the structures. The wavelength of the input laser was 442 nm with a spot size of 0.8 μm^2 . When a laser beam irradiated the sample, the scattered light with Raman signals were collected by a spectrometer in the system. The spectrometer measured the intensity of the Raman signal at different frequencies. The spectrum before the beam was released was used as the reference. When the residual stress or stain due to etching exists in the structures after release, the Raman spectrum displays a shift in frequency with respect to the reference spectrum. The spectra of the doubly clamped beam in Fig. 2(a) before and after release are shown in Fig. 2(b). Eleven locations on the suspended beam as shown in Fig. 2(a) were selected for micro-Raman measurements, with $x=0$ at the center. The transverse optical (TO) and longitudinal optical (LO) peaks of the Raman spectrum before release were at 775.98 cm^{-1} and 963.76 cm^{-1} . The Raman peaks after release was observed at the same wave numbers as shown in Fig. 2(b), which confirms that the beam was not subjected to residual stresses. The Raman intensity decreases after the structure was released. Since Raman intensity profile strongly depends on the stacking arrangements of the SiC polytypes [11], the decrease in intensity could be attributed to the removal of SiC material underneath the beam.

Fig. 2 (a) SEM image of a 50 μm -long, 5 μm -wide and 1 μm -thick clamped-clamped 4H-SiC beam with 11 locations for micro-Raman measurements; (b) Raman spectra of the beam before and after release.

Resistance-temperature measurements were performed on four suspended beams with length of 25 μm , 50 μm , 75 μm and 100 μm using a Signatone probe-station with a hot chuck and temperature controller. Fig. 3(a) shows the average values (markers) and standard deviations (error bars) of the resistances from multiple measurements on each device. With the n-type nitrogen-doped 4H-SiC suspended beams as the temperature sensing element, the sensor demonstrated a positive temperature coefficient, i.e., the resistance increases with an increasing environmental temperature. The nitrogen donor level in 4H-SiC is 60~70 meV [12] below the conduction band. Due to this low ionization energy, nitrogen donors tend to be fully ionized at room temperature [13]. Therefore, the positive temperature coefficient was not attributed to incomplete ionization of nitrogen donors, but instead to the temperature dependence of low-field electron mobility in 4H-SiC. The low-field electron mobility μ_n is a function of the doping concentration and temperature, with a widely used empirical expression as [14]:

$$\mu_n = \mu_{max} \left(\frac{T}{T_{ref}} \right)^{\alpha} \left(\frac{N}{N_{ref}} \right)^{\beta} \quad (1)$$

where N is the total doping concentration. μ_{max} , μ_{min} , N_{ref} , and α are fitting parameters, which are temperature-dependent and determined to be [15]:

$$\mu_{max} = 1000 \quad (\text{cm}^2/\text{Vs}), \quad (2)$$

$$\mu_{min} = 1000 \quad (\text{cm}^2/\text{Vs}), \quad (3)$$

$$\alpha = 0.76 \quad (\text{cm}^{-3}). \quad (4)$$

$\alpha=0.76$ and has no temperature dependence. Using doping concentration of $1 \times 10^{19} \text{ cm}^{-3}$ and geometry of 1 μm thickness and 5 μm width, resistances of the four suspended beams at different temperatures were calculated following Eq. (1~4) and plotted (lines) in Fig. 3(a). The measured and calculated data agree well. The temperature coefficient, or sensitivity of a resistance thermometer, is defined as $\frac{1}{R} \frac{\Delta R}{\Delta T}$, where $\Delta R = (R_T - R_r)$ and $\Delta T = T - T_r$. R_T is the sensor resistance at temperature T . R_r is the sensor resistance at reference temperature T_r , which is the room

temperature of 27°C in this study. Fig. 3(b) plots the calculated sensitivity versus temperature, with a sensitivity of $3823 \times 10^{-6} \Omega/(\Omega \cdot ^\circ\text{C})$ in the 0 to 100 °C range attained by suspended 4H-SiC beam sensor. This value is comparable to the temperature coefficient of some metals [16], such as platinum of $3925 \times 10^{-6} \Omega/(\Omega \cdot ^\circ\text{C})$, gold of $3950 \times 10^{-6} \Omega/(\Omega \cdot ^\circ\text{C})$, silver of $3950 \times 10^{-6} \Omega/(\Omega \cdot ^\circ\text{C})$, and copper of $4200 \times 10^{-6} \Omega/(\Omega \cdot ^\circ\text{C})$. It is noted that the temperature coefficient decreases with increase of temperature following a linear relationship of $= -0.8748 \times T + 3911.4$. This decrease in sensitivity was attributed to the less magnitude of changes in electron mobility (and therefore the resistivity) than the increase of temperature. The electron mobility values in Fig. 3(b) were calculated using Eq. (1~4).

Fig. 3 (a) Calculated (lines) and measured (markers) resistance data from four suspended beams as a function of temperature; (b) electron mobility calculated using Eq (1~4) and temperature coefficient (i.e. sensitivity) versus temperature.

4. Conclusion

Single crystalline 4H-SiC resistance thermometer with suspended microstructure as the sensing element was investigated. Micro-Raman spectroscopy was applied on the microstructure before and after release by photoelectrochemical etching. The results showed no shift in Raman peak positions, indicating that no residual stress or stress gradient existed in the suspended structure. Resistance-temperature measurements on doubly clamped beams showed a positive temperature coefficient. A good agreement was found among the measured resistance values and calculated resistance values based on a temperature-depended electron mobility model. A sensitivity of $3823 \times 10^{-6} \Omega/(\Omega \cdot ^\circ\text{C})$ in the 0 to 100 °C range was attained which is comparable to some metals. The sensitivity decreases with temperature following a linear function.

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