Pressure and Vibration Sensors Using Piezoresistive Effect of Polycrystalline Diamond Film

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Prototype sensors for the measurement of pressure and vibration were fabricated using the piezoresistive effect of boron-doped diamond. Undoped and boron-doped diamond were deposited on a Si substrate using the hot-filament chemical vapor deposition (CVD) method. The bulk micromachining technique was used to fabricate pressure and vibration sensors. The boron-doped diamond was deposited on an isolation layer of the undoped diamond film. Selective diamond deposition was carried out using a metal mask, and 30-µm-width patterning of the diamond was possible. The pressure sensor was evaluated in the ranges from room temperature to 250°C and from 0 to 0.07 MPa of applied pressure. The vibration sensor was evaluated in the ranges from room temperature to 180°C and from 1 to 50 G of acceleration.

1. Introduction

Using the piezoresistive effect of boron-doped diamond,¹ various sensors for high-temperature use can be realized. Although single crystalline diamond shows a high gauge factor such as 2000² compared to that of polycrystalline diamond film, 100,² the single crystalline diamond is too costly to be used in practical sensors because it requires a high pressure and a high temperature for manufacture. On the other hand, polycrystalline diamond can be formed by several chemical vapor deposition (CVD) methods, and it is sufficiently cheap for common use.

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Large-area depositions of such diamond film using microwave plasma CVD and hot-filament CVD, for wafers of up to several inches in size, have been achieved, and the cost advantage is clear.

In this work, we attempt to demonstrate the potentiality of polycrystalline diamond film for practical sensor use. There have been several works on the piezoresistive effect of boron-doped diamond films, but in most of these works, simple structures such as cantilever beams have been used as the test structure. Investigations of practical sensor chips involving packaging and evaluation of their high-temperature performance have been insufficient. Only a few investigations on diamond sensor structures have been carried out. We fabricated compatible sensor chips for commercial use, and evaluated these packaged sensors. Using micromachining technology for diamond sensor fabrication, several types of sensors will be realized. We applied a technique for the bulk micromachining of the silicon substrate and diamond film to the fabrication of pressure and vibration sensors.

2. Experimental

Diamond films were deposited on Si substrate chips of size 20×20×0.38 mm³, using the hot-filament CVD method under the conditions shown in Table 1. The boron was doped using a reaction gas containing H₃BO₃ during the deposition. The reaction gas was prepared in the following manner. Solution of H₃BO₃ to methanol (CH₃OH) was diluted to 1/3 with acetone ((CH₃)₂CO), and vaporized by hydrogen bubbling. The evaporated gas was mixed with the main hydrogen flow and introduced to the CVD chamber.

After boron-doped diamond deposition, the chip was heat treated at 400°C in air for 30 min to eliminate the surface conductive layer. After this treatment, the surface resistance increased above 100 MΩ. The diamond films were observed by SEM and analyzed by Raman spectroscopy.

Figure 1 shows the fabrication process of a diamond piezoresistor. In this experiment we did not use the diamond etching technique because a rough surface remained from the reactive ion etching. We used a selective diamond deposition method for obtaining undoped and boron-doped layers. First we used SiO₂ film as a mask, but the film disappeared under diamond deposition, and as a result, diamond was deposited on the exposed Si surface. Then,

<table>
<thead>
<tr>
<th></th>
<th>Undoped diamond</th>
<th>Boron-doped diamond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate temperature</td>
<td>900°C</td>
<td>900°C</td>
</tr>
<tr>
<td>Filament temperature</td>
<td>2500°C</td>
<td>2500°C</td>
</tr>
<tr>
<td>Filament-substrate distance</td>
<td>5 mm</td>
<td>5 mm</td>
</tr>
<tr>
<td>Gas composition (CH₄/H₂)</td>
<td>5.0%</td>
<td>2.2–3.7%</td>
</tr>
<tr>
<td>Pressure</td>
<td>4 kPa</td>
<td>4 kPa</td>
</tr>
<tr>
<td>Deposition time</td>
<td>60 min</td>
<td>60 min</td>
</tr>
<tr>
<td>Gas flow rate</td>
<td>300 sccm (CH₄+H₂)</td>
<td>H₂: 300 sccm (main), 38–76 sccm (bubbling)</td>
</tr>
</tbody>
</table>
we used the metals Ni, Ti and Cr for masking. These materials were deposited on the Si substrate by sputtering and were patterned by photolithography. In the test procedure of selective diamond deposition using a metal mask, Ni became the catalyzer, and graphite formed around the mask, while Ti and Cr showed good masking properties. After etching of the masking metal, a thin conductive layer remained on the Si substrate and diamond. This layer decreased the isolation resistance between the piezoresisters, and then we used a Murakami solution for etching of the layer. The Murakami solution (K$_3$[Fe(CN)$_6$] : KOH : H$_2$O=1 : 1 : 10) was used as the etching solution of carbides, but it did not eliminate the layer completely. Therefore, we then polished the chip surface using emery paper of 1000 grits, the layer could thus be eliminated completely, and a clean surface was obtained.

Figures 2(a) and 2(b) show the chip layouts for a pressure sensor and a vibration sensor, respectively. In the pressure sensor, there are 4 boron-doped diamond resistors on the undoped diamond layer. The black painted area shows the boron-doped layer. In order to avoid the problem of the uncovering of the metal layer at the step of the diamond layer edge, the boron-doped diamond was extended to the contact pads. The ohmic contact was formed by Ti/Au layers deposited on the pads. The dashed line represents the edge of the membrane, and a square diaphragm with a size of 2.0×2.0 mm$^2$ was formed by silicon dry etching from the back surface. Two of the four resistors are located at the edge of the diaphragm where the maximum tensile strain arises, and the resistors were 200 µm long and 30 µm wide. The two other resistors are located outside the diaphragm for use as references. The four resistors could be configured as a bridge circuit using a wire connection from outside the chip. However, only one resistor was used for the resistance measurement in this experiment. The
vibration sensor consisted of one or three beams which were connected with a mass at the center of the chip. The boron-doped diamond resistor was located at the root of the beam, and the length and width of the resistors were 400 $\mu$m and 30 $\mu$m, respectively. Figure 3 shows the measurement setup of the pressure sensor and the vibration sensor. Applied pressure to the pressure sensor was controlled using a valve on the vacuum pump. The vibration sensor was packaged and set on a top of the vibration exciter. The resistor of each sensor chip was supplied with a constant current and the voltage between the two terminals of the resistor was measured. The relative resistance change was calculated from the value of the differential voltage when pressure or acceleration was applied to the sensors.

3. Result and Discussion

3.1 Pressure sensor

Figure 4(a) shows a fabricated pressure sensor. The sensor chip was bonded onto the base, fixed in the can package, and connected to pins using a Au wire. Figure 5 shows the relative changes in resistance of two sensors as functions of pressure. Because the two sensors had different diaphragm thicknesses, they show linear resistance changes against applied pressure with two different gradients. Figure 6 shows the Arrhenius plot of six sensors. For the measurement, the sensors were placed in a small box where a heater was mounted, and no pressure was applied. Every resistance decreased with temperature, and the activation energy, $E_a$, was estimated to be 0.02–0.11 eV. These values were similar to values obtained in previous studies. Figure 7 shows $\Delta R/R$ as a function of temperature at the pressure of 0.07 MPa. The pressure sensitivity, $\Delta R/R$, strongly depends on the diaphragm thickness, and the thinnest diaphragm sample, No. 2181, shows the highest sensitivity. In the case of the square diaphragm, the radial strain $\sigma_r$ at the edge of the diaphragm is calculated as

$$\sigma_r = \frac{a^2}{h^2} P,$$  \hspace{1cm} (1)
Fig. 3. Measurement setups of pressure and vibration sensors.

(a) Measurement setup of pressure sensor. (b) Measurement setup of vibration sensor.

Fig. 4. Pictures of pressure and vibration sensors.

(a) Picture of pressure sensor. (b) Picture of vibration sensor.

Fig. 5. Relative change in resistance as a function of pressure.

Sample No. 2183 • (diaphragm thickness 30 µm)
Sample No. 2174 ■ (diaphragm thickness 53 µm)
Fig. 6. Arrhenius plot of piezoresistance.

Fig. 7. Relative change in resistance, $\Delta R/R$, as a function of temperature at 0.07 MPa of applied pressure.

where $a$ is the radius of the diaphragm, $P$ is the pressure difference, and $h$ is diaphragm thickness.

Gauge factor $K$, an index of the sensitivity of a piezoresistive sensor, is defined as the value of relative change in electrical resistance ($R/R_0$) divided by the applied strain $\varepsilon$, as follows.

$$K = \frac{\Delta R}{R_0} \frac{R}{\varepsilon}$$  

(2)
The gauge factor, $K$, for this type of pressure sensor was estimated using Eqs. (1) and (2). The effect of thermal expansion on the gauge factor was neglected. Figure 8 shows the gauge factors, $K$, of six sensors as functions of temperature. Sample No. 2180, with the highest $E_a = 0.11$ eV, shows the highest $K$ of 17.9, but it decreased with increasing temperature. Table 2 shows the fabrication conditions of the pressure sensor chips. There are three parameters for the sensor fabrication; they are the concentration of carbon to hydrogen, the thickness of the diaphragm, and the boron concentration in liquid solution. In our previous study, a low carbon concentration was observed to improve the gauge factor of the diamond film.\(^{(11)}\) Therefore, the concentration was made low in these experiments. In order to improve the sensitivity of the sensors, the thickness of the diaphragm was varied from 62 to 5 µm. However, in the case of 5 µm thickness, the diaphragm consisted of only a thin diamond film and the film was wrinkled.

Figure 9 shows the image of the fabricated pressure sensor chip according to SEM observation. The surface morphology of the boron-doped diamond resistor and its Raman spectrum are shown in the same figure. The film surface is revealed to be randomly oriented polycrystalline diamond, and its grain size is estimated as 2–3 µm. A diamond peak at 1333 cm$^{-1}$ is clearly observed from the Raman spectrum.

![Figure 8. Gauge factor as a function of temperature.](image)

Table 2
Fabrication conditions of pressure sensor chips.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Resistance (Ω)</th>
<th>Diaphragm thickness (µm)</th>
<th>C-O/H (%)</th>
<th>B/C (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2155</td>
<td>8860</td>
<td>50</td>
<td>3.0</td>
<td>1448</td>
</tr>
<tr>
<td>2174</td>
<td>66300</td>
<td>53</td>
<td>3.0</td>
<td>869</td>
</tr>
<tr>
<td>2175</td>
<td>14580</td>
<td>62</td>
<td>3.7</td>
<td>869</td>
</tr>
<tr>
<td>2180</td>
<td>108200</td>
<td>37</td>
<td>2.2</td>
<td>1448</td>
</tr>
<tr>
<td>2181</td>
<td>6450</td>
<td>20</td>
<td>3.0</td>
<td>2896</td>
</tr>
<tr>
<td>2183</td>
<td>10950</td>
<td>30</td>
<td>3.0</td>
<td>2896</td>
</tr>
</tbody>
</table>
3.2 The vibration sensor

Figure 4(b) shows the fabricated vibration sensor. The sensor chip was mounted in the stainless-steel housing using ceramics binders. Figure 10 shows the relative change in resistance of the resistor as a function of acceleration applied to the sensor chip (type1: one-beam-type vibration sensor). The sensitivity, $\Delta R/R$, was low, but it increased linearly with acceleration.

Figure 11 shows $\Delta R/R$ as a function of temperature under a constant acceleration of 30 G (type1). The sensitivity of the vibration sensor increased with temperature, but the sensitivities of all of the pressure sensors decreased with temperature. There was no significant difference in the conditions of the diamond deposition between the pressure sensors and the vibration sensors (see Tables 2 and 3). Therefore, this trend in the sensitivity was governed by factors other than the physical properties of the diamond film.

Figure 12 shows $\Delta R/R$ as a function of acceleration applied to the sensor chip having three beams (type 2). In order to improve the sensitivity, a small thickness of the beam, 10 $\mu$m, was employed. Owing to the thinness of the beam, the sensor could not be used under high acceleration. However, $\Delta R/R$ increased linearly with acceleration.

Figure 13 shows $\Delta R/R$ as a function of temperature under a constant acceleration of 2 G (type2). The trend of the sensitivity change with temperature is the same as that of a type 1 sensor. Type 2, the vibration sensor with three beams, shows high sensitivity in a low acceleration range. From Fig. 14, it is evident that the resonance frequency of this sensor is approximately 16 Hz. In the type 1 vibration sensor, clear resonance could not be found, and it showed a broad frequency response. The type 2 vibration sensor used three beams, because multiple beams stabilized the vibration of the mass and improved the strength of the sensor structure.

Figure 15 shows the Arrhenius plot of two sensors. The estimated activation energy, $E_a$,
Table 3
Fabrication conditions of vibration sensor chips.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Resistance (Ω)</th>
<th>Length of resistor (µm)</th>
<th>Beam thickness (µm)</th>
<th>Sensor type</th>
<th>C-O/H (%)</th>
<th>B/C (ppm)</th>
</tr>
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<tbody>
<tr>
<td>2161</td>
<td>28000</td>
<td>400</td>
<td>50</td>
<td>one beam</td>
<td>3.0</td>
<td>1448</td>
</tr>
<tr>
<td>2188</td>
<td>15000</td>
<td>400</td>
<td>10</td>
<td>three beams</td>
<td>3.7</td>
<td>869</td>
</tr>
<tr>
<td>2189</td>
<td>54000</td>
<td>800</td>
<td>10</td>
<td>three beams</td>
<td>3.7</td>
<td>869</td>
</tr>
</tbody>
</table>

Fig. 10 (left). Relative change in resistance, $\Delta R/R$, as a function of acceleration (Type 1, Sample No. 2161).

Fig. 11 (right). Relative change in resistance, $\Delta R/R$, as a function of temperature at 30 G of applied acceleration (Type 1, Sample No. 2161).

Fig. 12 (left). Relative change in resistance, $\Delta R/R$, as a function of acceleration (Type 2, Sample No. 2189).

Fig. 13 (right). Relative change in resistance, $\Delta R/R$, as a function of temperature at 2 G of applied acceleration (Type 2, Sample No. 2189).
was 0.05–0.26 eV. Table 3 shows the fabrication conditions of the vibration sensor chips. The beam thickness has a strong effect on the sensitivity and frequency response of the sensors. In this measurement setup, the current of the diamond gauge should be limited to below 0.2 mA. If the current went over this limit, the resistance of the diamond resistor was rapidly decreased and accurate measurement became difficult.

4. Conclusions

Prototype sensors were fabricated for the measurement of pressure and vibration using the piezoresistive effect of boron-doped diamond. The bulk micromachining technique was employed to realize the sensor chips. Diamond films were deposited on Si substrates using the hot-filament CVD method, and the diamond piezoresistors were fabricated by selective diamond deposition using metal masks. The maximum sensitivity of the pressure sensors

![Fig. 14. Relative change in resistance, $\Delta R/R$, as a function of acceleration frequency at 6 G of applied acceleration (Type 2, Sample No. 2188).](image1)

![Fig. 15. Arrhenius plot of piezoresistance.](image2)
was 0.19% of resistance change at the differential pressure of 0.07 MPa at room temperature. The sensitivity decreases with temperature, to become 0.06% at 250°C. The maximum sensitivity of the vibration sensors was 0.0029% of the resistance change at the acceleration of 2 G at room temperature, and increased with temperature to 0.0037% at 175°C.

**Acknowledgements**

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**References**