Technology of polycrystalline diamond thin films for microsystems applications

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Large area and uniform polycrystalline diamond (poly-C) thin films, with a thickness of approximately 1 μm, were grown and patterned on 4 in. oxidized Si wafers using IC compatible processes for microsystems applications. Uniform and reproducible seeding with a density of $2 \times 10^{10}/\text{cm}^2$ was achieved by spinning diamond powder loaded water on 4 in. wafers. Gas mixture of 1.5% methane in hydrogen was used in MPCVD system for diamond film growth with optimized pressure and microwave power. Thickness variation of less than 20% was achieved on the 4 in. area using 43 Torr pressure and 2.8 kW microwave power. Electron cyclotron resonance (ECR)-assisted microwave plasma reactive ion etch was carried out using SF$_6$/O$_2$/Ar gases to pattern the diamond films with an etch rate around 80 nm/min and less than 10% variation in etch rate over a 4 in. area. © 2005 American Vacuum Society. [DOI: 10.1116/1.1926290]

I. INTRODUCTION

The presence of $sp^3$ C–C bonds in the diamond lattice leads to its unique mechanical, chemical, optical and thermal properties not matched by any other known materials. Consequently, diamond becomes a unique material for a number of applications including microsystems and micro-electromechanical systems (MEMS), especially at high temperatures and in harsh environments. The difficulty to fabricate the $sp^3$ C–C bonds, which delayed the production of chemical vapor deposition (CVD) diamond until 1952–53, is now causing a delay in the development of a reliable diamond microfabrication technology that is compatible with conventional microsystems/MEMS technologies. Since there is no reliable large-area diamond hetero-epitaxial technology available today, a number of studies have focused on inexpensive polycrystalline diamond (poly-C) growth mainly over small size Si wafers (less than 2 in.). Although some studies used 4 in. Si wafers as substrates, most of the reported work has focused on small size substrates to demonstrate poly-C films for temperature sensors and heaters, piezoresistive sensors, gas/chemical sensors, radiation sensors, field emission and optoelectronics devices, MEMS packaging, and RFMEMS resonators. Most of these studies, although primarily motivated by industrial applications, were limited to laboratory scale devices with less emphasis on scaling, cost and reproducibility.

To develop a viable poly-C microfabrication technology, a number of different issues need to be addressed, particularly large area growth (3–6 in.), high seeding density ($10^8–10^{11}$ cm$^{-2}$), growth temperature (350–900 °C), diamond quality, surface roughness, $p$-type doping (0.001–100 Ω cm), dry etching, and contact resistance of metallization. Although most of these issues have been addressed by different researchers in laboratories around the world, they have never been addressed by a group of researchers in the same laboratory, particularly for applications in microsystems/MEMS area. This paper focuses on all of these issues to develop and optimize the poly-C technology for microsystems.

II. POLY-C MICROSYSTEMS TECHNOLOGY

The poly-C Microsystems technology development focuses on high-density diamond seeding, large area film growth, boron doping of poly-C, patterning through dry etching, Ohmic contacts and process integration. P-type 4 in. Si wafers with a 2 μm thick SiO$_2$ layer were mainly used as a substrate material in this study. The diamond growth conditions are shown in Table I.

A. Seeding

The CVD growth of poly-C films requires a pretreatment step to generate seeds (or nuclei) on the substrate before the growth begins. Surface abrasion, ultrasonic in diamond powder loaded solution, bias enhanced nucleation (BEN), spinning of diamond-powder-loaded photoresist (DPR), and spraying of diamond-loaded fluids are typically used for the pretreatment as shown in Table II.

In the present study, a new technique is developed for large area seeding with better control of uniformity and high density. A diamond powder loaded water (DPLW) solution is prepared by mixing of 25 carats of diamond powder, with an average particle size of 25 nm, in 1000 ml of deionized water and adding a suspension reagent. DPLW, particularly suitable for clean hydrophilic surfaces such as those of SiO$_2$ and Si$_3$N$_4$, is applied to the substrate using a regular spinner. Ultrasonication was used to enhance the uniformity of the suspended powder. The DPLW technique leads to a reproducible, nondestructive and uniform seeding with densities in the range of $10^8–10^{10}$ cm$^{-2}$ on SiO$_2$ which is not achievable by any of the previous seeding methods on a large area of SiO$_2$ or Si$_3$N$_4$ surface.

The oxidized Si wafers were RCA (Ref. 27) cleaned followed by an etching step of 10 s in 5% HF solution.
wafer were then spin-coated with DPLW using typically a spin speed of 3000 revolutions per minute (rpm) for 30 s. Different spin speeds were used to study the effect of spin speed on the seeding density. Multiple-spins were used to improve the seeding density and uniformity.

As shown in Table III, the DPLW was spun on the oxidized silicon wafers with spin speeds in the range of 1000–4000 rpm leading to seeding densities in the range of $3 \times 10^9$–$5 \times 10^{10} \text{ cm}^{-2}$, which was calculated statistically from a number of SEM pictures taken for each sample. It was found that low spin speed (1000 or 2000 rpm) gave density greater than $10^{10} \text{ cm}^{-2}$ but with some agglomeration of diamond particles that leaves some unseeded areas with density of $1.8 \times 10^9 \text{ cm}^{-2}$. High spin speed (3000 or 4000 rpm) can reduce the agglomeration but yields lower seeding density in the range of $4 \times 10^9 \text{ cm}^{-2}$.

Further experiments showed that multiple spins of DPLW (2–3 times) reduced the agglomeration while keeping the high seeding density as shown in Figs. 1 and 2. Figure 1(b) indicates uniformity over the whole wafer yielding a seeding density of $1.8 \times 10^{10} \text{ cm}^{-2}$ without agglomeration.

### B. Growth

Poly-C can be grown using a variety of CVD methods including microwave plasma CVD (MPCVD), hot filament CVD, radio frequency (RF) CVD and dc-arc jet CVD (Ref. 32) as seen in Table IV. However, MPCVD is the most widely used technique for electronic and optical applications because of its efficacy to produce high film quality, large substrate size, less contamination and better controllability.

For the results reported in this work, a bell jar type MPCVD chamber (Wavemat™ MPDR 313EHP) was used with a 9 in. chamber diameter and 5 in. quartz bell jar diameter. A 2.45 GHz, 5 kW microwave power supply (Sairem™, GMP60KSM) and a large chamber size ensured the uniformity of the plasma and the poly-C growth on 4 in. size substrates. The sample wafer was heated by the plasma and its temperature was monitored by a pyrometer. The detailed growth conditions are listed in the Table I. Trimethylboron (B(OCH_{3})_{3}, TMB) diluted in hydrogen (TMB/H_{2}=0.1% in volume ratio) was introduced during the poly-C growth environment for the in situ boron doping.

For the diamond growth in the MPCVD chamber, the radial decrease of plasma intensity causes the non-uniformity of film thickness on large area, which limits the possible applications of diamond thin films. Different growth parameters were tested as shown in Table I to optimize the film uniformity on 4 in. wafers. Figure 3 shows the AFM images of the poly-C film grown on 4 in. wafers using 2.4 kW of microwave power and 35 Torr pressure (the film thickness at the center of the wafer is 1.6 μm).

The thickness variation over the 4 in. area of the wafer is shown in Fig. 4 for three samples with comparable thicknesses, but grown at different pressures and microwave powers. The thickness of each sample is normalized to 1 for better comparison of the thickness variation along the radius.

### Table I. MPCVD poly-C growth parameters.

<table>
<thead>
<tr>
<th>Samples</th>
<th>#1</th>
<th>#2</th>
<th>#3</th>
</tr>
</thead>
<tbody>
<tr>
<td>H_{2}/CH_{4} flow (sccm)</td>
<td>200/3</td>
<td>200/3</td>
<td>200/3</td>
</tr>
<tr>
<td>Microwave power (kW)</td>
<td>1.6</td>
<td>2.4</td>
<td>2.8</td>
</tr>
<tr>
<td>Gas pressure (Torr)</td>
<td>22</td>
<td>35</td>
<td>43</td>
</tr>
<tr>
<td>Substrate temperature (°C)</td>
<td>520</td>
<td>700</td>
<td>780</td>
</tr>
<tr>
<td>Growth rate (μm/h)</td>
<td>0.1</td>
<td>0.2</td>
<td>0.3</td>
</tr>
</tbody>
</table>

### Table II. Comparison of different seeding methods for seeding density, substrate material, and surface morphology.

<table>
<thead>
<tr>
<th>Seeding method</th>
<th>Sonication /abrasion</th>
<th>BEN</th>
<th>DPR</th>
<th>Spray/paint</th>
<th>DW spin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Seeding density(cm⁻²)</td>
<td>Up to $10^{10}$</td>
<td>Up to $10^{11}$</td>
<td>$\sim 10^8$</td>
<td>Up to $10^{11}$</td>
<td>$10^9$–$10^{10}$</td>
</tr>
<tr>
<td>Substrate requirement</td>
<td>Most dielectric and metal</td>
<td>Conductive Si or metal</td>
<td>Most dielectric and metal</td>
<td>Most dielectric and metal</td>
<td>Hydrophilic surface like (SiO₂ and Si₃N₄)</td>
</tr>
<tr>
<td>Surface</td>
<td>Scratch surface, not good for thin film</td>
<td>No damage</td>
<td>No damage</td>
<td>No damage</td>
<td>No damage</td>
</tr>
<tr>
<td>Uniformity and controllability</td>
<td>Not uniform</td>
<td>Uniform and repeatable on whole wafer</td>
<td>Uniform and repeatable on whole wafer</td>
<td>Not uniform</td>
<td>Uniform and repeatable on whole wafer</td>
</tr>
</tbody>
</table>

### Table III. Relation between the diamond seeding densities on oxidized Si wafer and the DPLW spin speed.

<table>
<thead>
<tr>
<th>Spin speed (rpm)</th>
<th>1000</th>
<th>2000</th>
<th>3000</th>
<th>4000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nucleation density (cm⁻²)</td>
<td>$4 \times 10^{10}$</td>
<td>$1 \times 10^{10}$</td>
<td>$5 \times 10^9$</td>
<td>$3 \times 10^9$</td>
</tr>
</tbody>
</table>
The major reason for the thickness variation is that the non-uniform distribution of the plasma causes nonuniform substrate temperature and growth rate. At the low growth temperature of 520 °C (temperature at the center of the wafer in Table I, the film thickness shows the largest variation. The possible reason for this is that low microwave power and low pressure can only heat up the center part of the 4 in. wafer and have no appreciable diamond growth in the outer area with temperatures below 500 °C. As for some applications that low temperature growth of poly-C may be required, the plasma uniformity can be enhanced by using electron cyclotron resonance (ECR) at low growth pressures.

The sample grown at 780 °C reveals a thickness variation of less than 25%, which is comparable to that reported for 6 in. (Ref. 34) and 2.36 in. (60 mm) (Ref. 35) Si wafers using microwave power levels of 15–60 kW and 5 kW, respectively. However, the power consumption per unit area for samples used in the present study (0.056 kW/in.²) is substantially lower than that for 6 in. (0.53 kW/in.²) and for 2.36 in. (0.28 kW/in.²) wafers. This high efficiency was achieved by thermally insulating the sample from the substrate, and optimizing the microwave power and pressure.

C. Doping

Accurate control of the doping level and resistivity is particularly important for poly-C thin film applications in sensor area. Different boron containing compounds typically used for p-type doping are pure boron powder(36) boron trioxide (B₂O₃),37 diborane (B₂H₆),38,39 and trimethylboron.40 Although boron ion implantation has been used for diamond doping,41,42 its effectiveness has been limited by the implant damage which cannot be annealed due to low diffusion constant of boron in poly-C.43 In situ doping using trimethylboron (TMP) is chosen in the current work because 0.1% TMB diluted in hydrogen is safer than other boron compounds.40 It has been found that CVD diamond films grown in a hydrogen plasma have a thin hydrogenated surface layer, which becomes conductive after exposure to the atmosphere. Annealing at 600 °C can remove this conductive layer due to hydrogen desorption effect.44,45 In this work, the boron doping of poly-C film was investigated as a function of TMB/CH₄ gas ratio, growth temperature and postgrowth an-

<table>
<thead>
<tr>
<th>Methods</th>
<th>HFCVD</th>
<th>MPCVD</th>
<th>DC-arc Jet CVD</th>
<th>Combustion synthesis</th>
<th>RFCVD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Growth Rate</td>
<td>0.1–10</td>
<td>0.1–10</td>
<td>30–150</td>
<td>4–40</td>
<td>&lt;0.1</td>
</tr>
<tr>
<td>(μm/h)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Substrate</td>
<td>300–1000</td>
<td>300–1200</td>
<td>800–1100</td>
<td>600–1400</td>
<td>700–1200</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Growth Area</td>
<td>5–900</td>
<td>5–100</td>
<td>&lt;2</td>
<td>&lt;3</td>
<td>100</td>
</tr>
<tr>
<td>(cm²)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Advantages</td>
<td>Simple, large area</td>
<td>Quality, stability</td>
<td>High rate, good quality</td>
<td>Simple, high rate</td>
<td>High quality</td>
</tr>
<tr>
<td>Disadvantages</td>
<td>Contaminations, fragile filament</td>
<td>Rate</td>
<td>Contaminations, small area</td>
<td>Small area</td>
<td>Low rate, expensive</td>
</tr>
</tbody>
</table>

Typically, poly-C films with a thickness of 1 μm were grown at 700 °C with growth conditions described earlier. The poly-C films were annealed at 600 °C for 20 min at $1 \times 10^{-5}$ Torr to remove the surface layer by desorption.

The actual data consisting voltage $V$ and current $I$ measured using the 4-probe method yield the sheet resistance

$$R_{sh} = \frac{V}{I},$$

where $\xi$ is the geometric coefficient dependent on the sample area and shape.

If the thin film has only one conductive layer with known thickness $t$, the conductivity is given by

$$\sigma = \frac{1}{R_{sh}}. \quad (2)$$

Since the conductivity of the annealed film is determined only by the poly-C bulk layer, Eq. (2) becomes

$$\sigma_{\text{anneal}} = \frac{1}{R_{sh \text{ anneal}}} = \frac{1}{\rho_{\text{bulk}}} \frac{1}{R_{sh \text{ bulk}}}. \quad (3)$$

The as-grown poly-C film consists of two different conductive layers, the surface and bulk, with thicknesses of $t_{\text{surf}}$ and $t_{\text{bulk}}$, respectively. The total resistance, $R_{\text{as-grown}}$ of the parallel combination is given by

$$\frac{1}{R_{\text{as-grown}}} = \frac{1}{R_{\text{surf}}} + \frac{1}{R_{\text{bulk}}}, \quad (4)$$

where $R_{\text{surf}}$ and $R_{\text{bulk}}$ are resistances of the surface layer and bulk layer, respectively. Since $R_{\text{surf}} = R_{\text{sh}}(\xi) = \frac{1}{\rho_{\text{surf}}} = \frac{1}{\rho_{\text{as-grown}}} (t_{\text{surf}})$, Eq. (4) can be written in sheet resistance as

$$\frac{1}{R_{\text{sh \text{ as-grown}}}} = \frac{1}{R_{\text{sh \text{ surf}}}} + \frac{1}{R_{\text{sh \text{ bulk}}}}. \quad (5)$$

Using Eq. (2), the relationship between measured total sheet resistance and conductivity is given by

$$\frac{1}{R_{\text{sh \text{ as-grown}}}} = \sigma_{\text{surf}} t_{\text{surf}} + \sigma_{\text{bulk}} t_{\text{bulk}}, \quad (6)$$

According to Eq. (2), the conductivity of as-grown film, $\sigma_{\text{as-grown}}$, is given by

$$\sigma_{\text{as-grown}} = \frac{1}{R_{\text{sh \text{ as-grown}}}} = \frac{1}{R_{\text{sh \text{ as-grown}}} (t_{\text{bulk}} + t_{\text{surf}})^2}. \quad (7)$$

which using $t_{\text{surf}} \ll t_{\text{bulk}}$ and Eq. (6), becomes

$$\sigma_{\text{as-grown}} \approx \frac{1}{R_{\text{sh \text{ as-grown}}} t_{\text{bulk}}} = \sigma_{\text{surf}} t_{\text{surf}} + \sigma_{\text{bulk}}. \quad (8)$$

Now $\sigma_{\text{anneal}}$ and $\sigma_{\text{as-grown}}$, computed using Eqs. (3) and (8), are plotted in Fig. 5. The solid lines in Fig. 5 are exponential fits which yield:

$$\sigma_{\text{as-grown}} = 0.033 + 4.677 \times 10^{-4} \times \exp(264.5 \times x^{0.56}), \quad (9)$$

\[\text{Fig. 3. AFM images of the poly-C film grown at 700 °C on a 4 in. wafer.}\]

\[\text{Fig. 4. Radial distributions of normalized poly-C film thickness at different growth conditions and the Raman spectra of different samples.}\]

\[\text{Fig. 5. In situ and annealed conductivity of poly-C films grown at 700 °C with different doping levels.}\]
where $x$ is the TMB/CH$_4$ gas ratio. A comparison of Eqs. (9) and (10) reveal that the first term on the right-hand side of Eq. (9) relates to $\sigma_{\text{surf}}$, which, using Eq. (8), is given by

$$
\sigma_{\text{surf}} = 0.033(\Omega \, \text{cm})^{-1}.
$$

If the $t_{\text{surf}}$ and $t_{\text{bulk}}$ are 40 nm (Ref. 46) and 1 $\mu$m, respectively, Eq. (11) gives

$$
\sigma_{\text{surf}} = 0.825(\Omega \, \text{cm})^{-1},
$$

which suggests that the surface conductivity is independent of the doping concentrations. The corresponding surface sheet resistance is 330 k$\Omega$/$\square$.

For a $p$-type semiconductor, the bulk conductivity can be written as

$$
\sigma_{\text{bulk}} = q\mu_p = q\alpha N_A \mu_p \exp\left(\frac{E_A}{k T}\right),
$$

where $q$ is the charge of electron, $\mu$ is the hole concentration, $N_A$ is the concentration of acceptors, $\alpha$ is the activation coefficient, and $E_A$ is the acceptor activation energy. As the above discussion shows that $\sigma_{\text{anneal}}$ is equal to $\sigma_{\text{bulk}}$, Eqs. (10) and (13) lead to

$$
N_A = \frac{4.677 \times 10^{-4}}{q \mu_p} \exp\left(\frac{E_A}{k T} + 264.5 \times x^{0.56}\right).
$$

Equation (14) can be used to estimate the $N_A$ using TMB/CH$_4$ ratio if there is a weak dependence of $N_A$ on $x$ and $E_A$.

Figure 6 shows the poly-C film conductivities at different doping levels and different growth temperatures after anneal which can be used as experimental guidelines for doping estimation.

D. Patterning

To integrate poly-C devices in mostly Si-based microsystems, a patterning of poly-C films using techniques consistent with a typical Si processing is required. As the wet etching of poly-C is impossible, the only patterning techniques available are in situ patterning and dry etching. The in situ patterning techniques rely on selective seeding which has been achieved using different methods including diamond powder-loaded photoresist (DPR) employing standard lithography, selective seeding using SiO$_2$ as a mask, and direct spray writing. However, in all these techniques, the lateral growth at the pattern edges and, in some cases, growth in unwanted areas can pose serious problems for small feature sizes. The dry etching of poly-C, which uses different active gas species such as oxygen, argon, CF$_4$ and SF$_6$ with metal or SiO$_2$ masks, seems to be excellent choice for poly-C patterning. Most researches have used conventional reactive ion etching (RIE) methods where the gas species are excited by the rf power. Dry etching using ECR assisted microwave plasma at low substrate temperatures and pressures has led to very clean structures with small feature sizes and sharp edges.

The dry etching of poly-C film was carried out in an ECR microwave plasma chamber and the plasma was generated by a 2.45 GHz, 1.5 KW (ASTeX™) microwave power supply. An rf power of 100 W was coupled into the chamber to generate a negative bias in the range of ~50 V to ~200 V on the substrate of sample holder, which is critical to the etch rate. The O$_2$ and SF$_6$ (Ref. 52) were used as the reactive ion source for etching using a pressure in the range of 4–8 mTorr. With a rotary pump and turbomolecular pump, the achievable base vacuum is on the order of $10^{-7}$ Torr. Patterned Al films were used as a masking material for the selective dry etching of poly-C. The Al etch rate is approximately 1/13 of that of poly-C. The details of parameters for dry etching are listed in the Table V. A typical etch rate for poly-C was 80 nm/min with a variation of less than 7% over the 4 in. area of the wafer. Figure 7 shows SEM images of two poly-C structures prepared by the ECR plasma etching; a 1 $\mu$m thick finger structure with 2 $\mu$m minimum feature size and an intragrain $p$-type piezoresistor fabricated on an electrically-insulating 300-$\mu$m-thick large-grain freestanding poly-C plate.

### E. Ohmic contacts

Low-resistivity Ohmic contact between poly-C and metal thin films is essential for any diamond electronic device application. The type of metal contact on poly-C (Ohmic or rectifying) depends not only on the choice of metals but also on the doping concentration, annealing and other parameters. Titanium and chromium (with codeposited gold on top of them to prevent oxidation) are most widely used metals because they can form carbides with poly-C and yield good
conductivity after anneal. Werner et al. have demonstrated that the Al (with 1% Si) film also gave Ohmic contact with poly-C by forming silicon carbide. For Ti/Au, Cr/Au, and Al/Si films, a vacuum annealing at 400–600 °C for 20 min is enough to achieve stable Ohmic contacts. Figure 8 shows contact resistivity as a function of poly-C doping levels. This plot includes the results from this study and those reported by other researchers. Inset 1 of Fig. 8 shows the \( I-V \) curve of contact between poly-C and Ti/Au film at room temperature after annealing at 600 °C for 20 min. In the configuration of Kelvin Bridge, shown in the inset 2 of Fig. 8, a specific current \( I \) flows (shown in the figure by arrows) through the poly-C/metal contact area, leading to a voltage across the contact, which is measured between \( V1 \) and \( V2 \). The contact resistivity \( \rho_c \), computed by using the contact resistance \( R_c \) and the contact area \( A_c \) (the poly-C/metal overlay area in Fig. 8), is given by

\[
\rho_c = R_c \cdot A_c = \frac{V1 - V2}{I} \cdot A_c.
\]

**F. Microsystems process integration**

Integration is one of the important issues which limited the development and application of poly-C thin film sensors because of the special seeding and growth requirements. A common procedure in integrated circuits (IC) process is insulating the active layer (poly-Si or metals) with dielectric layers (such as SiO\(_2\)) both above and below it, and making electrical connections through the contact holes. Based on the design of poly-C piezoresistive position sensors, we developed a poly-C thin film fabrication process as shown in Fig. 9 to integrate the poly-C technology into standard IC fabrication process. The process insulates the micron-scale continuous poly-C films with dielectric layers both above and below it, and makes electrical connections through the contact holes such as those found in common IC processes. This method can protect the underlying materials during the...
diamond growth and dry etching procedures. The poly-C growth temperature can be varied between 400 °C and 900 °C depending on the materials used underneath the poly-C. Al (with 1% Si) was used as the interconnection layer which can give good Ohmic contact with poly-C after annealing.58 Figure 10 showed a poly-C piezo-resistive position sensor fabricated following the process depicted in Fig. 9. The poly-C film was heated to 425 °C for 20 min during LTO deposition, but the electrical conductivity of the fabricated position sensor is consistent with the unpatterned film.

III. CONCLUSIONS

An IC compatible poly-C thin films fabrication process was realized on a 4 in. oxidized Si wafer using the DPLW spin, high power MPCVD, and ECR-assisted reactive ion etching technologies. The new DPLW spin method can give a uniform seeding density of $2 \times 10^{10}$ cm$^{-2}$ over the 4 in. wafer. The optimized growth exhibited a power efficient large area growth with less than 25% thickness variation over the 4 in. wafer. ECR-assisted reactive ion etching with microwave plasma was carried out using SF$_6$/O$_2$/Ar gases with an etch rate of 80 nm/min and less than 7% variation over the 4 in. area.

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Fig. 10. SEM of the poly-C piezo-resistive sensor fabricated on a silicon probe with integration process described in Fig. 9.