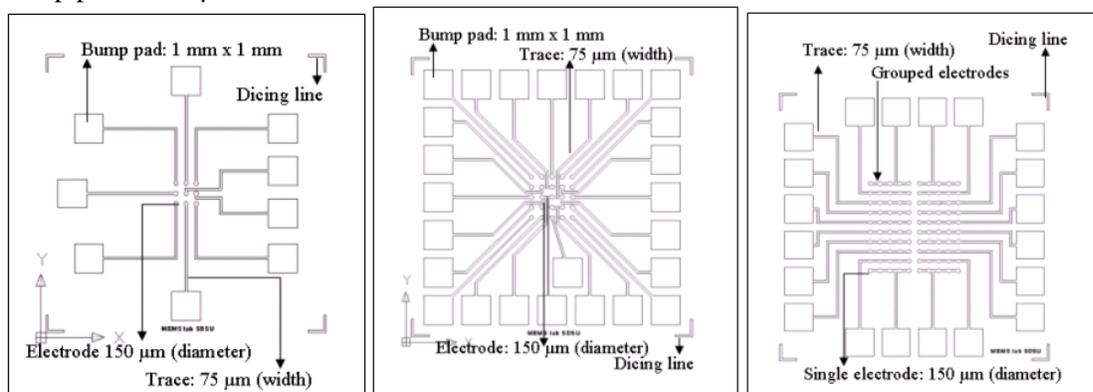


DESIGN AND MICROFABRICATION OF 3D CARBON MEMS ELECTRODE MICROARRAY

This chapter discusses the design of 3D C-MEMS electrode microarray and its microfabrication process. First the design starts with an active microarray design of electrodes that uses an electric field for the migration and accumulation of biological samples to achieve fast hybridization. Secondly, three dimensional electrodes (with a height of $200\ \mu\text{m}$) design is introduced instead of planar electrode design for efficient trapping. Lastly, C-MEMS technology is utilized for the microfabrication of a 3D electrode microarray due to carbon materials having attractive properties for design of electrodes. These properties include mechanical durability, electrical conductivity, and chemical stability. The use of carbon materials also enables the achievement of high aspect ratio features with cheap cost [7]. The 3D photoresist-derived carbon electrodes with a height of $200\ \mu\text{m}$ are obtained by using conventional photolithography followed by pyrolysis. Due to the shrinkage while undergoing pyrolysis [50], the photoresist layer for the electrode posts was spin-coated with a thickness of $220\ \mu\text{m}$ initially to obtain a final height of $200\ \mu\text{m}$.

1 CHIP DESIGN USING CAD

Three microarray designs that were drawn by CAD are presented in this thesis. First, a 3×3 design that has 9 test sites was designed as the simplest among three total designs. In order to increase the number of test sites that are related to work efficiency, slightly more complicated 5×5 and 10×10 designs were also designed. As the number of test sites increased, it became more difficult to achieve an efficient traces layout to connect the electrodes with the bump pads. Electrodes for the 3×3 and 5×5 microarrays were connected directly to bump pads so that they could be biased individually. However, for the 10×10 microarray, groups of five electrodes were bundled together per individual bump pad due to the complexity of the traces layout, as can be seen in Figure 4.1 (c). All the electrodes, bump pads, and traces are located on the same surface. The isolation of the electrodes site from the bump pads is achieved through proper packaging, as elaborated upon in more detail in Chapter 5. The die size of each design is 1 cm by 1 cm and the size of bump pads that surround the electrodes site and will be used to bias the electrodes is 1 mm by 1 mm. Two types of electrode shapes are used; a circular-shaped electrode and a diamond-shaped electrode. The electric fields generated at the edge of diamond-shaped electrodes are expected to be higher than the ones of circular-shaped electrodes. The diameter of each circular-shaped electrode and the width of each diamond-shaped electrode are both $150\ \mu\text{m}$ and the spacing between center to center points of each electrode is $350\ \mu\text{m}$. The width of traces that connect the electrodes and the bump pads is $75\ \mu\text{m}$.



(a) 3×3 C microarray design (b) 5×5 C microarray design (c) 10×10 C microarray design

Figure 1 Sketch of each microarray design with dimensions for electrodes (150 μm), traces (75 μm), and bump pads (1 mm x 1 mm).

2 CHIP POPULATION AND MASK DESIGN

Three basic microarray designs and three other alternate designs that have diamond-shaped electrodes with a width of 150 μm were arranged on a 4 inch (100 mm) silicon wafer. Each design is first placed in the middle section of the substrate and then the rest of the area is filled again with each design.

After populating the chip, the mask CAD file was built using AutoCad and sent to the printing company (CAD Art Services, Inc., Bandon, OR) to fabricate a transparent film mask to be used for photolithography. Two masks were needed in the 3D carbon electrode microarray fabrication process. The first mask was used for the electrical connections between electrodes and bump pads and the second mask was needed for electrode posts.

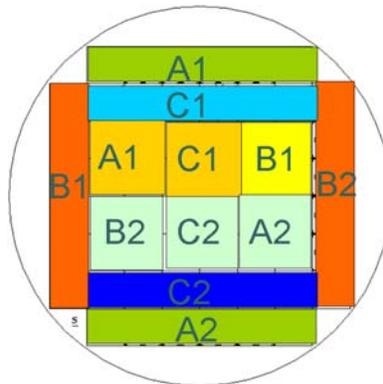


Figure 4.2 Schematic diagram of chip population (A1: 3 x 3C, A2: 3 x 3D, B1: 5 x 5C, B2: 5 x 5D, C1: 10 x 10C, C2: 10 x 10D, C: Circular shaped electrode, D: Diamond shaped electrode).

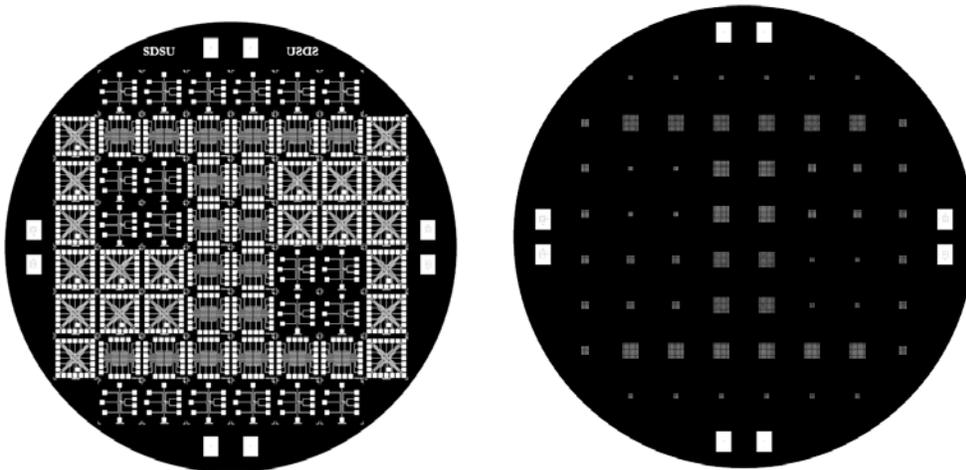


Figure 4.3 Mask 1 for electrical connections. Figure 4.4 Mask 2 for high-aspect-ratio electrode.

The SU-8 used for the electrical layer and electrode posts layer in this thesis is a negative photoresist. Therefore white areas which appeared on the masks (Figure 4.3 and Figure 4.4) will be cross-linked after they are exposed to UV light and will remain after the SU-8 development process.

Since two masks are used, alignment marks are crucial for the mask alignment. Eight white rectangles at each mask (Figure 4.3 and Figure 4.4) contain the alignment marks shown below (Figure 4.5).

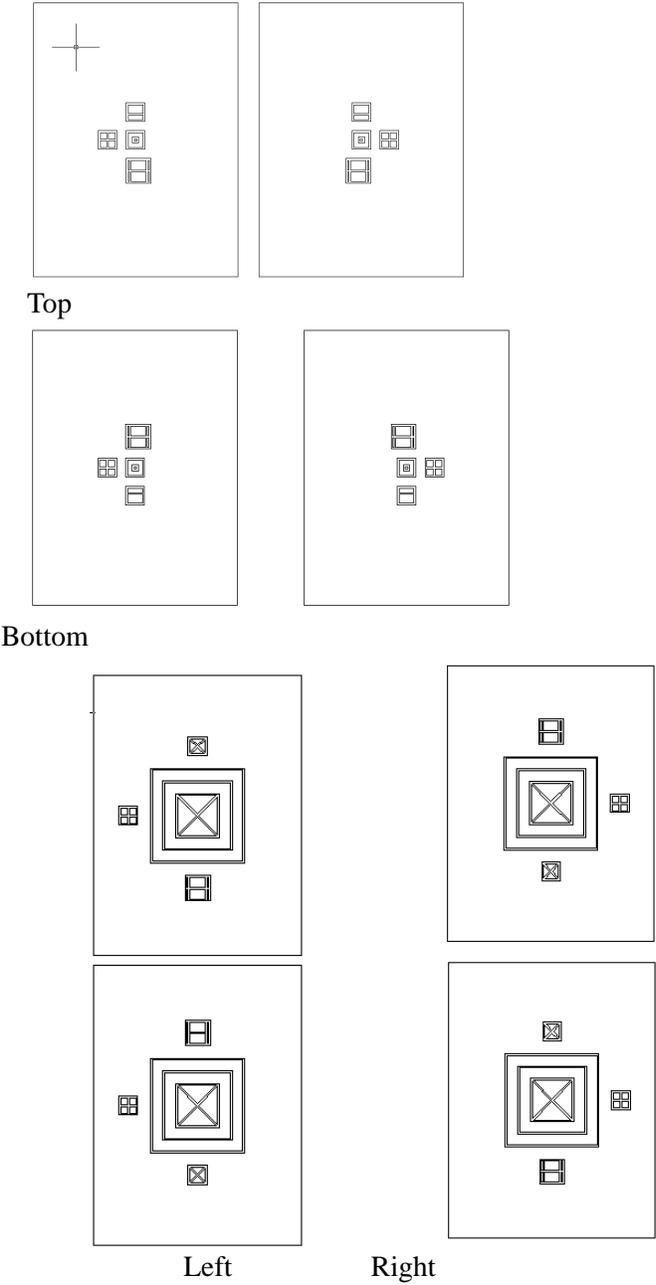


Figure 4.5 Sketch of alignment marks for mask alignment.

3 CHIP FABRICATION PROCESS

C-MEMS technology is utilized for the microfabrication process since carbon materials have attractive properties for design of electrodes that have mechanical durability, electrical conductivity, and chemical stability [7, 49]. It also enables the achievement of high-aspect-ratio features with cheap cost.

The 3D carbon MEMS electrode microarrays were fabricated using the facility at UCI-INRF in collaboration with the Madou Research Group (Genis). 3D carbon MEMS electrode microarray fabrication starts with a clean (100) silicon wafer which has a 0.5 μm thick silicon dioxide layer for the insulation (Figure 4.7).

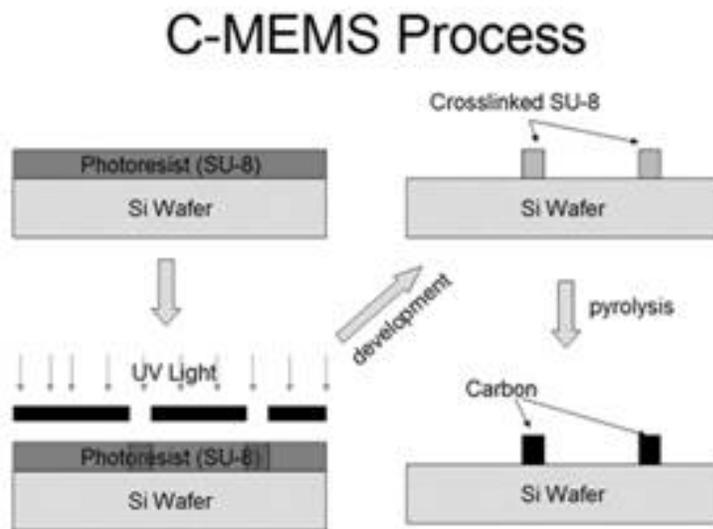


Figure 4.6 Schematic diagram of CMEMS process [71].

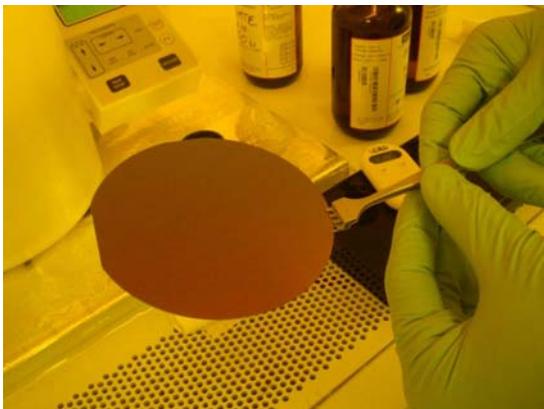


Figure 4.7 Clean wafer with 0.5 μm oxide layer washed with acetone.

The clean wafer is then spin-coated with a 10 μm thickness of SU-8-10 for 35 seconds at 3000 rpm before going through the soft-bake process for 7 minutes at 95 $^{\circ}\text{C}$ in the oven.



Figure 4.8 Spinner used for spin-coating (made by Laurell technologies).

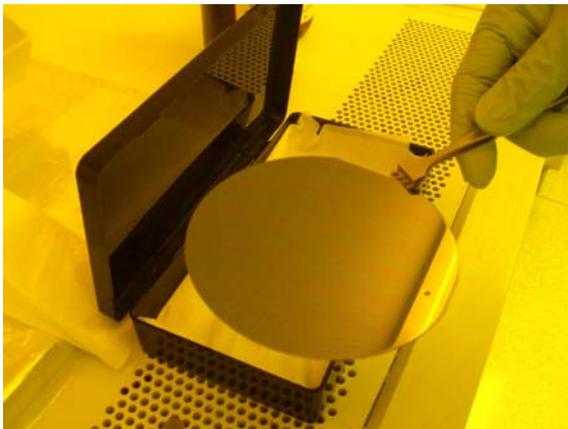


Figure 4.9 Wafer spin-coated with SU-8-10 with a thickness of 10 μm .



Figure 4.10 Oven used for soft-bake process (made by BLUE M).

The soft-baked wafer is then exposed to UV light using the first mask on top of it with an energy of 300 mJ/cm^2 . Among the three different exposure modes (contact, proximity, or projection), proximity mode was utilized here. In proximity lithography, the mask does not touch the wafer. The mask is placed and fixed on top of the glass and the bottom of the glass contacts with the wafer, as seen in Figure 4.12.



Figure 4.11 UV light machine made by Oriel.

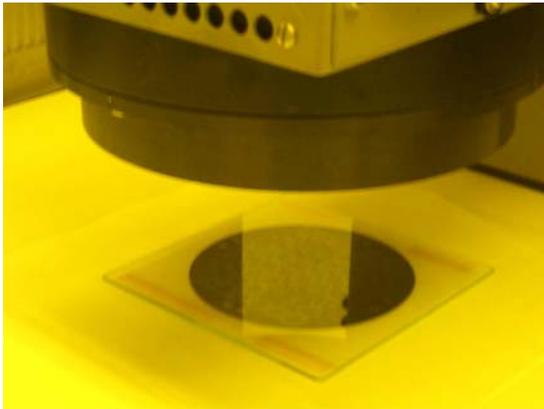


Figure 4.12 UV exposure process in proximity mode.

For obtaining a reasonable cross-link, the wafer undergoes a PEB (Post Expose Bake) process for 4 minutes at 95 °C on the leveled hot plate. After a 10-minute cooling off period, the wafer is developed by using SU-8 developer (Figure 4.13) and then rinsed off by isopropanol to get rid of the residues.



Figure 4.13 Developing process using SU-8 developer.

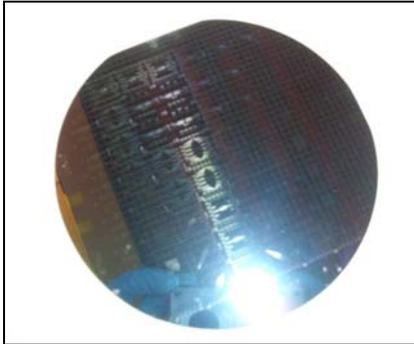


Figure 4.14 Electrical connection patterned wafer after the first lithography.

The processes above are for the electrical connections between electrodes and bump pads. Similar processes should be done to give electrodes with high-aspect-ratio structures. The wafer shown in Figure 4.14 is spin-coated again using SU-8-100 (more viscous than SU-8-10) for 35 seconds at 1600 rpm with a thickness of 220 μm (Figure 4.15).

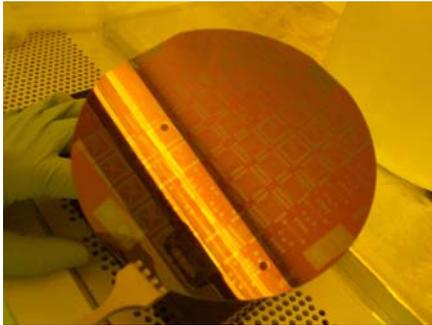


Figure 4.15 Wafer after the second spin-coat.

The SU-8-100 spin-coated wafer is then soft-baked for 1.5 hours at 95 $^{\circ}\text{C}$ on the leveled hot plate (Figure 4.19). After a 1.5 hour long soft-bake, a second lithography process follows. This time the mask aligner is needed for both alignment of the wafer with the second mask and subsequent UV exposure (Figure 4.16). Alignment marks play a very significant role for the alignment. The second mask is placed in the left drawer (Figure 4.17) and the wafer is placed in the front middle drawer. Alignment is carried out by matching the alignment marks on the mask with the ones patterned on the wafer (Figure 4.18). Once alignment is done, the wafer is exposed to UV light with an energy of 700 mJ/cm^2 .

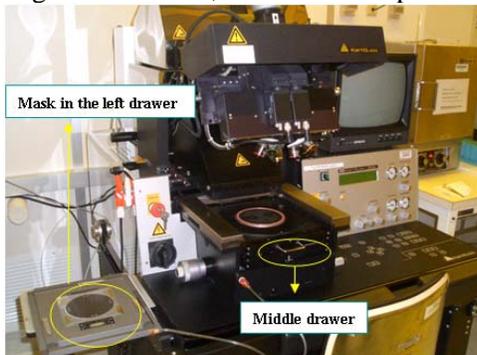


Figure 4.16 Karl Suss MA6 Mask Aligner (MA6-BSA) is used for both mask alignment and UV exposure during second lithography.

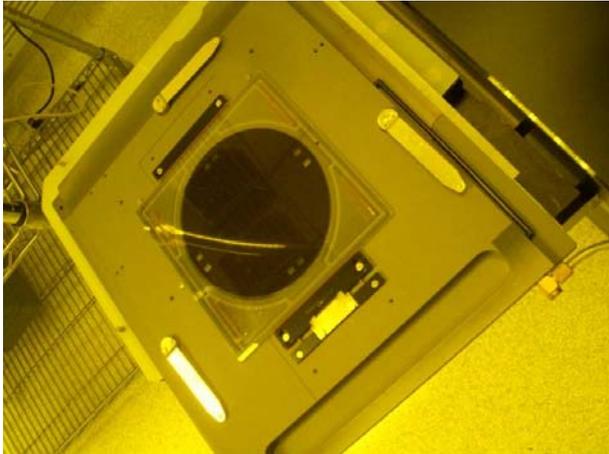


Figure 4.17 Mask placed in the left drawer (mask alignment I).



Figure 4.18 Alignment marks matching process (mask alignment process II).

The wafer goes through a 20-minute PEB @ 95 °C on the hot plate (Figure 4.19) and then is developed for 30 minutes (Figure 4.20).



Figure 4.19 Leveled hot plate (95 °C) used for PEB (Courtesy of Madou's Lab at UCI).

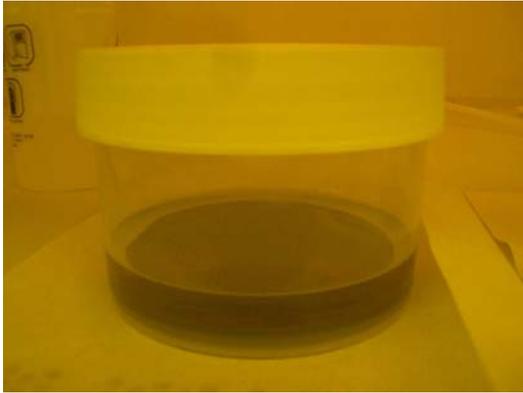


Figure 4.20 Second developing process for 30 minutes.

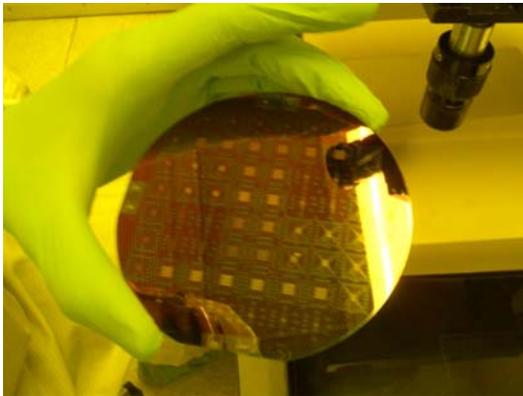
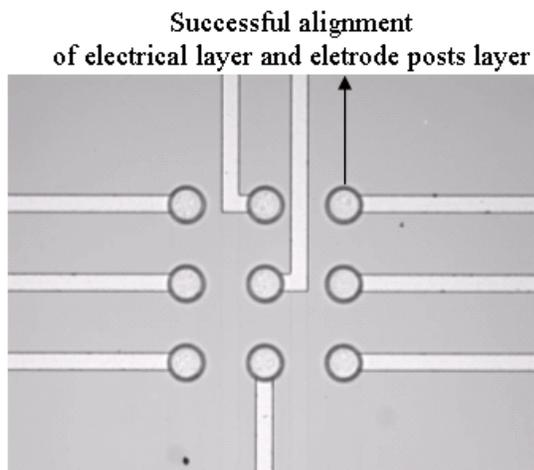
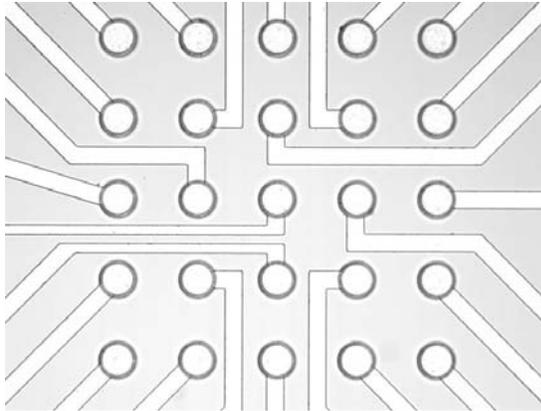


Figure 4.21 Completed wafer before pyrolysis.

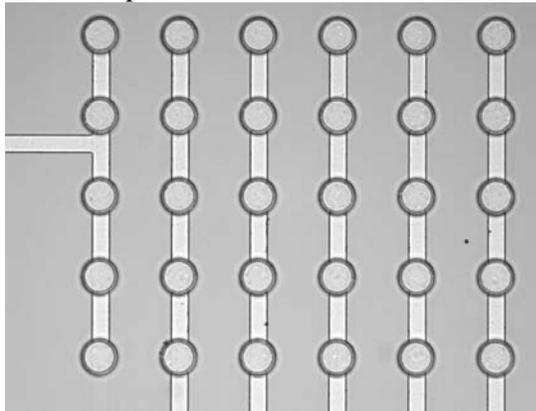
The pictures of each chip design shown in Figure 4.22 were taken before pyrolysis using an electronic microscope (5x magnification). These images show that the electrical layer and electrode posts layer are well-aligned. Thick-lined circles indicate where alignment occurred.



3 x 3C chip



5 x 5C chip



10 x 10C chip

Figure 4.22 Electronic microscope pictures with 5x magnification of each chip design before pyrolysis.

The next step is pyrolyzing the wafer in the furnace (Figure 4.23) according to a specified heating schedule (Figure 4.24). The heating schedule has a multitude of steps in order to prevent the peel-off phenomenon which can occur during pyrolysis. Since pyrolysis is decomposition of organic materials by heating in the absence of oxygen, nitrogen gas flow is provided during the pyrolysis process to prevent the presence of oxygen. The pyrolysis process is carried out for about 10 hours.



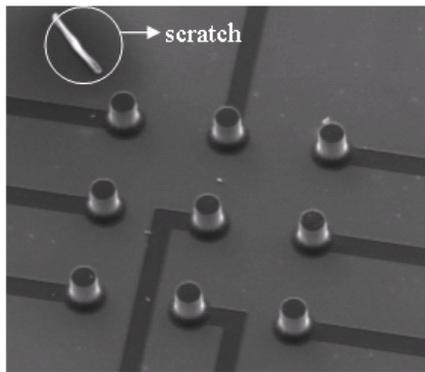
Figure 4.23 High temperature vacuum/inert gas atmosphere furnace made by RD Webb Company.



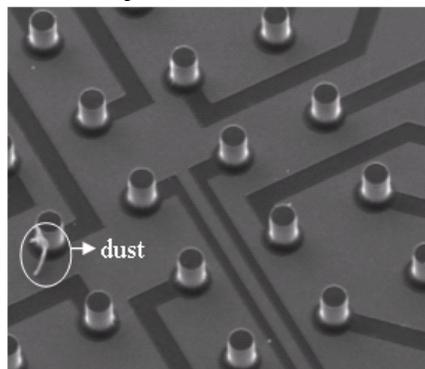
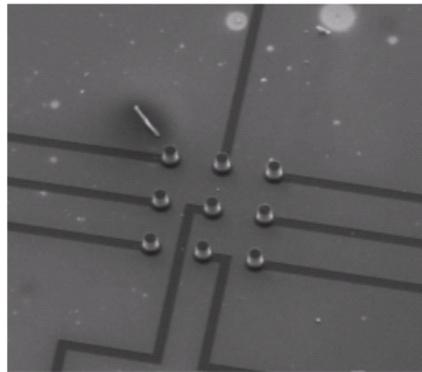
Temperature	Time
20 °C	90 min.
700 °C	90 min.
900 °C	60 min.
1000 °C	60 min.
1000 °C	60 min.
900 °C	90 min.
700 °C	90 min.
20 °C	

Figure 4.24 Heating programmer and multi-step heating schedule for the prevention of peel-off during pyrolysis.

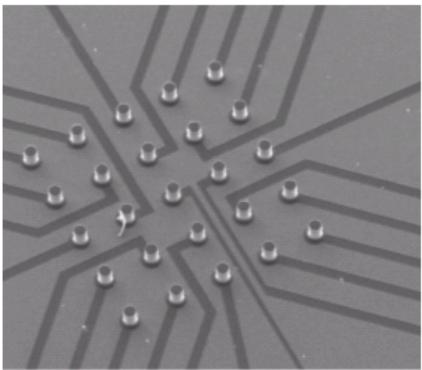
SEM (scanning electron microscope) images of each chip design were also taken after pyrolysis. A scratch is shown in the 3 x 3C microarray chip and some dust debris is on the 5 x 5C chip in the figure.

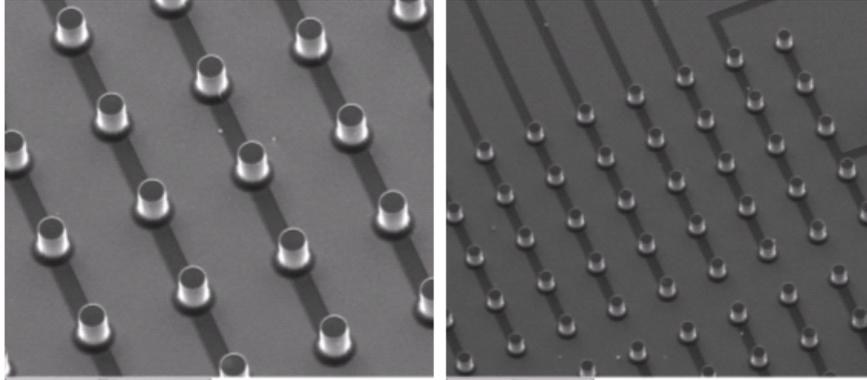


3 x 3C chip



5 x 5C chip





10 x 10C chip

Figure 4.25 SEM pictures of each chip design after pyrolysis.

A summarized table of the C-MEMS fabrication process and a comparison of Dr. Kassegne's 3D electrode microarray group to Dr. Tay's group is provided in Table 4.1 and Table 4.2, respectively.

Table 4.1 Carbon MEMS microfabrication process

Process	Equipment	Chemical used	Thickness	Note
Clean wafer	-	Acetone/ methanol	-	-
Grow oxide	Oven	-	0.5 μm	
First lithography	Spinner	SU-8-10	10 μm	35 sec. @ 3000 rpm
Spin-coating	Oven			7 min. @ 95 °C
Soft-bake	UV exposure machine			Use Mask 1 with an energy of 300 mJ/cm^2
UV exposure	Leveled hot plate			4 min. @ 95 °C
PEB	Wet bench	SU-8 developer /isopropanol		
Develop	Spinner	SU-8-100	220 μm	35 sec. @ 1600 rpm
Second lithography	Oven			1.5 h @ 95 °C
Spin-coating	Mask aligner			Use Mask 2 with the energy of 700 mJ/cm^2
Soft-bake	Leveled hot plate			20 min. @ 95 °C
Alignment & UV exposure	Wet bench	SU-8 developer /isopropanol		30 min.
PEB	Furnace	Nitrogen gas		Pyrolyze according to Heating schedule
Develop				
Pyrolysis				