## SINGLE-MATERIAL MEMS USING POLYCYRSTALLINE DIAMOND

By

Zongliang Cao

## A DISSERTATION

Submitted to Michigan State University in partial fulfillment of the requirements for the degree of

## DOCTOR OF PHILOSOPHY

Electrical and Computer Engineering

2011

#### ABSTRACT

#### SINGLE-MATERIAL MEMS USING POLYCYRSTALLINE DIAMOND

#### By

#### Zongliang Cao

Diamond, due to its unique mechanical, thermal, chemical and electrical properties, is an excellent material for Microelectromechanical Systems (MEMS). Furthermore, due to its large band gap of 5.5 eV, diamond offers the possibility of making MEMS structures out of a single material by varying the doping level to achieve the semiconducting, metallic and insulating (undoped) properties needed in a typical MEMS structure. Such single-material MEMS (SMM) can alleviate problems of complicated multilayer devices, such as thermal mismatch, adhesion, inter-layer diffusion, contact resistance.

Since polycrystalline diamond (poly-C) is inexpensive and retains many of the unique properties of single-crystal diamond, SMM technology based on poly-C has been developed in this research work. Moreover, poly-C can be layered to perform a number of functions, whereas a complex stack of materials would otherwise be required. Consequently, due to poly-C's high etching selectivity to most other materials, the SMM fabrication process developed in the current work can reduce the number of fabrication masks by a factor of 1.5 - 2 as compared to a conventional MEMS process.

The development of SMM technology faces three major challenges; (a) production of highly conducting and highly insulating films, (b) the development of dry etching techniques for multilayer poly-C structures without any damage to the sacrificial layers necessary to produce multilayer structures and (c) the surface micromachining technology of building multilayer SMM structures containing insulating, semiconducting and highly-conducting layers. All of these challenges and other associated poly-C micromachining technologies are discussed in depth. A number of complex poly-C SMM structures were fabricated using Si or  $SiO_2$  as a sacrificial layer to address the initial SMM issues. Additionally, the surface micromachining processes of SMM devices and SMM thin film packaging have been developed.

In this research, the design, fabrication and testing of novel poly-C RFMEMS resonators with piezoresistive detection, a potential application for SMM technology, are developed and presented for the first time. These resonator uses undoped poly-C with a resistivity above  $10^9 \,$   $\Omega \cdot cm$  as an insulating layer as well as a structural layer. Lightly boron-doped poly-C with a resistivity of 9  $\Omega \cdot cm$  is used as a semiconducting material with piezoresistive properties. Highly doped poly-C with a resistivity on the order of  $10^{-3} \, \Omega \cdot cm$  is used as interconnect to reduce the total parasitic resistance in signal transmission. SMM system integration has been achieved for a RFMEMS application with piezoelectric actuation and piezoresistive detection.

#### ACKNOWLEDGEMENTS

The author would like to express his deep appreciation to my thesis advisor and mentor, Dr. Dean M. Aslam, for his encouragement, guidance and support throughout this research. Additional thanks are extended to Dr. Donnie K. Reinhard, Dr. Terence Brown and Dr. Brian F. Feeny for their valuable discussions and academic advices.

The author would like to acknowledge all the members of Dr. Aslam's research group, particularly Dr. Hoyin Chan, Sean Hatch, Mike Varney and Sedat Gur for their assistance and helpful discussions. The author is also thankful to staffs and friends of the WIMS center at University of Michigan for their great help and friendship, particularly Jay Yoong Cho and Seowyuen Yee.

Last but not least, the author wishes to thank my whole family for their patience, understanding and support during this study. Greatest thanks to my wife Leting Zhao for her sacrifice.

This work was supported primarily by the Engineering Research Centers Program of the National Science Foundation under Award Number EEC-9986866.

### **TABLE OF CONTENTS**

LIST OF TABLES	vii
LIST OF FIGURES	viii
Chapter 1 Research Motivation and Goals 1.1 Introduction 1.2 Objective of This Work 1.3 Dissertation Organization	1 
Chapter 2 Diamond MEMS Technologies 2.1 Introduction 2.2 Forms of Diamond 2.3 Unexpected Electrical Conductivity of Undoped Poly-C	
<ul> <li>2.4 Patterning of Poly-C</li> <li>2.5 Diamond Piezoresistive Sensors</li> <li>2.6 Diamond RFMEMS</li> <li>2.7 BioMEMS</li> <li>2.7.1 Microfluidic channel</li> </ul>	
<ul><li>2.7.2 Neural Probes</li><li>2.8 Diamond Packaging</li></ul>	
<ul> <li>Chapter 3 Experimental Design and Setup</li></ul>	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
<ul> <li>Chapter 4 Single-Material MEMS Poly-C Film Technologies</li></ul>	49 49 49
<ul> <li>4.2.1 Poly-C growth with the presence of O<sub>2</sub></li></ul>	50 53 56 57 62

4.4 Characterization of Residual Stress	63
4.4.1 Substrate curvature measurement	
4.4.2 Characterization using micro-mechanical structure	
4.5 Multilayer Single-Material MEMS Fabrication Technologies	
4.5.1 Achievement of conformal poly-C coating	
4.5.2 Optimization of dry etching of poly-C	72
4.5.3 Surface micromachining of poly-C structures and devices	80
4.6 Single-Material MEMS Examples	
4.6.1 SMM capacitive RFMEMS	
4.6.2 SMM integrated field emission devices	
4.6.3 SMM pieozoresistive sensors	
Chapter 5 Single-Material MEMS Piezoresistive Resonators for RFMEMS	
5.1 Introduction	
5.2 Sensor Design	
5.3 Sensor Fabrication	
5.4 Sensor Characterization	101
5.4.1 Electrical conductivity and contact resistivity characterization	101
5.4.2 Displacement sensitivity and gauge factor characterization	106
5.5 Measurement of Frequency Response	107
Chapter 6 Single-Material MEMS Thin Film Packaging	114
6.1 Introduction	114
6.2 Process Design and Fabrication	115
6.3 Release and Sealing Using Porous Diamond	119
6.4 Evaluation of Poly-C Hermeticiy	122
Chapter 7 System Integration	124
Chapter 8 CONCLUSIONS AND FUTURE RESEARCH	127
8.1 Summary of Contributions	127
8.2 Future Research	128
APPENDICES	129
APPENDIX A	130
APPENDIX B	135
APPENDIX C	137
APPENDIX D	141
APPENDIX E	142
APPENDIX F	145
APPENDIX G	146
APPENDIX H	147
APPENDIX I	150
BIBLIOGRAPHY	156

#### LIST OF TABLES

Table 2-1 Comparison of diamond properties with other semiconductors	. 10
Table 2-2 Properties of various forms of diamond [2] [20] [23-27]	. 14
Table 2-3 The reported room temperature quality factor $Q$ 's at resonant frequencies $f_0$ 's flexural resonators	s of . 24
Table 3-1 Design values of micro strain gauge	. 39
Table 4-1 The processing parameters in various steps used in this study	. 51
Table 4-2 The measured resistivities of samples A-D	. 54
Table 4-3 Electrical resistivity, doping concentration for various TMB ratios	. 58
Table 4-4 Residual stresses for $CH_4/H_2$ of 0.5%, 1.0% and 2.0%	. 66
Table 4-5 Three-step etching conditions of poly-C	. 77
Table 5-1 Analytical, simulation results of k and $\varepsilon_{PZR}$	. 97
Table A-1 Gauge factors of poly-C reported by different groups	133
Table H-1 Values of x, y, $f(x,y)$ , $g(x,y)$ , k and $\varepsilon_{PZR}$	149

### LIST OF FIGURES

Figure 1-1 Concept diagram of SMM. For interpretation of the references to color in this and all other figures, the reader is referred to the electronic version of this dissertation
Figure 1-2 Overview of single-material MEMS using polycrystalline diamond
Figure 2-1 SEM images of (a) microcrystalline diamond (MCD) [3], (b) nanocrystalline diamond (NCD) [17], (c) ultrananocrystalline diamond (UNCD) [17] and (d) diamond-like carbon (DLC) [18]
Figure 2-2 Raman spectrum of (a) microcrystalline diamond (MCD) [21], (b) nanocrystalline diamond (NCD) [17], (c) ultrananocrystalline diamond (UNCD) [17], (d) diamond-like carbon (DLC) including a-C and ta-C [137]
Figure 2-3 Comparison of the highest gauge factors achieved in previous work at room temperature between poly-C and other metals and semiconductors. [88]
Figure 2-4 Gauge factors of poly-C at room temperature in literatures [89-104] [106] [180] 20
Figure 2-5 (a) Cantilever test structure to characterize the gauge factor [99], (b) a pressure sensor using <i>p</i> -type poly-C piezoresistor [109], (c) diamond acceleration sensor (left) and diamond pressure sensor (right) [99], (d) position sensor made of <i>p</i> -type poly-C in cochlear implant probe [180].
Figure 2-6 (a) Schematic cross-setion of diamond microwave relay (b) SEM-micrographs of microwave relay structures (beam length: 2500 $\mu$ m); left: coplanar line pattern deposited on the substrate and oriented orthogonally to the cantilever: right: complanar line deposited onto the beam surface and oriented along the cantilever. [142] [143]
Figure 2-7 SEM of microfluidic channels with 4 µm tunnels. [146]
Figure 2-8 Poly-C probe used for electrochemically detecting neurotransmitters. [157]
Figure 2-9 Completed sealed poly-C packaging. [158]
Figure 3-1 Cross-sectional view of micro-fabricated 4-point probe structure
Figure 3-2 Top view of micro-fabricated Van der Pauw structure
Figure 3-3 Schematic diagram of micro-fabricated Kelvin bridge structure used to measure the contact resistivity between doped poly-C ( $p$ poly-C) and metal with or without highly doped interlayer ( $p$ + poly-C)

Figure 3-4 Schematic diagram of micro-fabricated structure of linear Transmission line model (TLM) pattern used to characterize the contact resistivity of doped poly-C
Figure 3-5 A MEMS design used to characterize the electrical resistivity of undoped poly-C 37
Figure 3-6 Micro strain gauge used to measure the internal stress in poly-C thin film. Test beam, slope beam and indicator beam are released
Figure 3-7 Schematic diagram of the measurement setup to characterize the gauge factor $(GF)$ 40
Figure 3-8 Schematic diagram of testing sample mounted on the piezoelectric actuator
Figure 3-9 Schematic diagram of actuation of piezoelectric actuator
Figure 3-10 Schematic diagram of the driving circuit
Figure 3-11 Characterization of the driving circuit by oscilloscope
Figure 3-12 Schematic diagram of piezoresistive detection circuit
Figure 3-13 The frequency response of the detection circuit
Figure 4-1 Characteristics of the poly-C film seeded by 5% DPLW spin method. (a) front surface of the film; (b) side view of the film; (c) AFM image of the film; (d) Raman spectrum of the film. 50
Figure 4-2 Growth rate of poly-C with the presence of $O_2$ versus ratio of $O_2$ to $CH_4$
Figure 4-3 <i>I-V</i> plot of a 0.68-µm-thick undoped poly-C grown with O <sub>2</sub> present based on high ultra-high nucleation density
Figure 4-4 (a) SEM images of the fabricated structure with four metal lines in 4-point-probe configuration and (b) measurement result
Figure 4-5 (a) Optical image and concept diagram of the sample for resistivity measurement (b) resistivity of doped poly-C as a function of TMB ratio
Figure 4-6 Electrical resistances ( $k\Omega$ ) of a 600-800 nm thick boron-doped poly-C layer versus reciprocal temperature (K)
Figure 4-7 (a) Kelvin Bridge Structure used to measure the contact resistivity between $p$ poly-C (piezoresistive layer) and metal with or without $p$ + poly-C interlayer; (b) Plot of contact resistivity as a function of the resistivity of piezoresistive layer with the inset of $I$ - $V$ plot by Semiconductor Analyzer indicating excellent ohmic contact

Figure 4-9 Reading from micro strain gauge indicating a tensile stress of 26-36 MPa......67

Figure 4-14 Schematic diagram of 3-step etching of poly-C......77

Figure 4-15 (a) The SEM image of smooth etched surface using multi-step recipes in Tab.	le I; (b)
a dot of 5 $\mu m$ diameter was designed to study the etching of side wall; (c) smooth side	wall is
achieved when poly-C was etched in O2 plasma and H2 plasma at 10 mTorr	80

Figure 4-17 Left: Photoresist 9260 patterned on top of rough poly-C surface using optimized photolithography parameters; Right: an Al ribbon was patterned successfully via lift-off step. . 82

Figure 4-18 Released cantilevers using the surface micromachining techniques	
--	--

Figure 4-20 Inspection of (a) lateral gap and (b) vertical gap using fabricated poly-C structures.
Figure 4-21 poly-C beams with widths varying from 200 nm – 2 μm inspected by (a) SEM and (b) optical microscopy
Figure 4-22 SEM images of fabricated capacitive SMM RFMEMS and the fabrication process flow
Figure 4-23 (a) The concept diagram and SEM picture of fabricated IFED; (b) diamond needles created in O <sub>2</sub> plasma by ICP
Figure 4-24 Field emission measurement result with vacuum as insulator
Figure 4-25 SEM images of SMM piezoresistive sensors
Figure 4-26 Cross-sections of the fabrication sequence
Figure 4-27 Relative change of resistance vs. cantilever tip deflection
Figure 5-1 Schematic diagram of piezoresistive resonator
Figure 5-2 (a) stress contour of piezoresistive resonator, (b) tip deflection and (c) average strain in piezoresistors vs. applied force at the tip
Figure 5-3 Fabrication process flow of SMM piezoresistive resonator
Figure 5-4 SEM images of fabricated piezoresistive resonator 101
Figure 5-5 SEM images of fabricated Van Der Pauw structure used to measure the electrical resistivity of doped poly-C
Figure 5-6 Electrical resistivity of piezoresistive layer versus temperature
Figure 5-7 (a) SEM image of transmission line model used to characterize the contact resistance (b) The total resistance between two neighboring metal pads as a function of spacing between pads is plotted for four different contact lengths
Figure 5-8 $\Delta R/R$ vs. (a) cantilever tip deflection and (b) microstrain in piezoresistive sensor. 106
Figure 5-9 Concept diagram of measurement setup for piezoresistive detection and piezoelectric actuation. 108
Figure 5-10 Piezoresistive detection and piezoelectric actuation boards with wire-bonded sample shown at the bottom left

Figure 5-11 The measured frequency response in air for resonator with length $l = 430 \mu m$ when power supplied to Wheastone bridge is (a) 24 V and (b) 0 V
Figure 5-12 (a) experimental and (b) simulation results of frequency response in air for resonator with beam length $l = 630 \ \mu\text{m}$ ; (c) experimental and (d) simulation results with beam length $l = 430 \ \mu\text{m}$ .
Figure 6-1 SMM package concept diagram and the SEM picture of the structures 116
Figure 6-2 General fabrication process flow of SMM thin film package 118
Figure 6-3 (a) Creation of porous diamond by RIE of diamond (b) Release the structure by using HF to penetrate the porous diamond
Figure 6-4 Poly-C package having porous diamond as release holes before being soaked in HF
Figure 6-5 Poly-C package having porous diamond as release holes after being soaked in HF for (a) 10 minutes and (b) 30 minutes
Figure 6-6 Porous diamond grows into continuous poly-C film in MPCVD 122
Figure 6-7 Sample chips with poly-C layer of (a) 2.62 μm (b) 2.92 μm after 2 days soak in HF.
Figure 7-1 Design of system integration
Figure 7-2 Frequency response in air for a resonator with beam length of 430 µm measured using the system integration setup in Figure 7-1
Figure A-1 Simplified Schematic of valence bands split-off model (a) no stress, (b) under tensile stress, and (c) under compressive stress
Figure B-1 (a) Schematic diagram of temperature control system and (b) sample mounting on the heater
Figure C-1 Schematic diagram of seeding process (a) Si has hydrophobic surface after removing the native oxide by diluted hydrofluoric acid (HF). Separate water drops can be observed after taking the wafer out from deionized (DI) water. (b) Super-hydrophilic surface is achieved after Oc plasma transmost. A thin water layer forms on the surface after taking the wafer out from DI
water. (c) Several drops of DPLW are put on silicon wafer immediately after $O_2$ plasma treatment. (d) Wafer is spun. Uniform and high seeding density is achieved across the wafer. 137

Figure C-2 For the case of using 0.5% DPLW, (a) seeding density of  $10^9$  cm<sup>-2</sup> was achieved and (b) 8 hours are needed to form continuous poly-C thin film. For the case of using 5% DPLW, (c)

seeding density above $10^{11}$ cm <sup>-2</sup> was achieved and (d) less than 20 minutes are need continuous film.	eded to form
Figure D-1 Measurement setup to characterize the electrical resistivity of undoped p	ooly-C films. 141
Figure F-1 Labview program used to measure the change of the resistance of p sensor as function of cantilever tip deflection.	iezoresistive 145
Figure G-1 Labview program used to measure frequency response	146
Figure G-2 The block diagram of the programming	
Figure H-1 Top and cross-sectional view of piezoresistive resonator	
Figure H-2 (a) $f(x,y)$ and (b) $g(x,y)$ as a function of x and y	

# **Chapter 1**

## **Research Motivation and Goals**

#### **1.1 Introduction**

The development of methods for the production of miniaturized mechanical components and devices in Si is a natural outgrowth of the Si surface machining methods that have been developed for the production of microcircuits. However, Si has its material limitations [1]. In particular, it has relatively low values for its Young's modulus and its fracture toughness; against both itself and many other materials, it has a comparatively large coefficient of friction (COF) and high rate of wear, such that Si-based MEMS exhibit high energy loss due to high friction. In addition, Si-based MEMS devices that involve extensive rolling or sliding motion typically wear out in a very short time [2] [3] [4]. The friction problem can be exacerbated by capillary adhesion in humid environments [5] [6] or by low operation temperature and heat dissipation due to the small energy band gap [3]. Clearly, Si is not an appropriate material for some MEMS devices, even for room temperature applications. Si MEMS components coated with Si<sub>3</sub>N<sub>4</sub> or SiC exhibit better behavior than pure Si in room-temperature MEMS applications, but they also have materials and fabrication limitations [7] [8].

Researchers continue to seek new materials for use in MEMS. Metals, semiconductors and polymers are being studied for their suitability. Among these candidate materials, diamond is believed to be amongst the most promising materials for MEMS applications. Usually polycrystalline diamond (poly-C) is used for MEMS applications instead of single crystal diamond (SCD) because it not only retains many of the properties of single crystal diamond but is also inexpensive and can be deposited on a wide range of substrates with reasonable deposition rates and good uniformity, making it suitable for very large-scale production. Two distinct forms of poly-C are microcrystalline diamond (MCD) and ultrananocrystalline diamond (UNCD), grown in H rich and H poor environments, and having grain sizes in the ranges of 0.5-10  $\mu$ m and 3-5 nm, respectively [2]. The MCD with grain sizes in the nanometer range, which increase with film thickness is called nanocrystalline diamond (NCD) [9].

Diamond possesses a number of excellent properties that Si and other materials lack. These include generally reduced COF and increased resistance to wear, both particularly useful for microsystems containing components with sliding interfaces. Poly-C also has a very high Young's modulus, high tensile and fracture strength, and is suitable for high frequency MEMS devices. It is also chemically inert and stable in air up to 600 °C, making it an attractive material for operation at above 300 °C, where conventional Si devices cannot operate [3]. It has excellent thermal conductivity (on the contrary UNCD has a low thermal conductivity which makes it an ideal thermal insulation layer) and a low coefficient of thermal expansion (CTE) which make it useful in developing lab-on-chip devices, packages or substrates with better heat dissipation. It can also be used as bimorph thermal actuators with large displacements. Diamond is biocompatible, mechanically strong and tribologically effective, and as such is an excellent coating material for implantable medical devices. Diamond-based micro-mechanical devices can be integrated with potential diamond electronics on the same substrate for high temperature applications using diamond.

#### **1.2** Objective of This Work



Figure 1-1 Concept diagram of SMM. For interpretation of the references to color in this and all other figures, the reader is referred to the electronic version of this dissertation.

The focus of this work is on developing the technology to build single-material MEMS (SMM) using poly-C and demonstrate SMM concept. The concept diagram of SMM is shown in Figure 1-1. It consists of diamond MEMS and circuits encapsulated inside a diamond package. Everything will be built on a diamond substrate. Metals are only used for long interconnects or bonding pads. The development of the technology required for diamond MEMS devices, interconnects and thin film packaging is reported in this work.

An overview of the work is illustrated in Figure 1-2. The study of SMM poly-C technology starts from the fundamental research of characterization and improvements of basic poly-C deposition techniques to achieve highly insulating and conducting poly-C thin films. Fundamental research also includes the achievement of poly-C conformal coating and dry

etching of poly-C. Then enabling technologies have been developed to build free-standing SMM devices and SMM thin film packaging with multiple poly-C layers. SMM system integration was achieved for a RFMEMS application with piezoelectric actuation and piezoresistive detection. This work involves following specific goals:

Fundamental studies to characterize MEMS quality poly-C thin film

- Highly insulating undoped poly-C growth and characterization;
- > Characterization of highly conducting and semiconducting poly-C thin film;
- Achievement of conformal poly-C coating;
- > Optimization of dry etching of multilayer poly-C structures;

#### Enabling technologies

- > Fabrication process development for SMM structures in micrometer & nanometer scales;
- Development of SMM examples: non-tested structures and functional structures;

#### SMM System integration

- SMM system components: study the piezoelectric actuation and piezoresistive detection for RFMEMS resonators;
- $\blacktriangleright$  Measure the resonant frequency and quality factor Q of piezoresistive resonators;

Successful accomplishment of these goals leads to the following unique contributions:

- Achievement of highly insulating poly-C in a reactor also used for doped poly-C growth.
- Optimization of dry etching of multilayer poly-C structures without causing damage to the underlying layer
- Development of multilayer SMM structures using poly-C
- First fabrication of SMM thin film packaging with rapid release and sealing using porous diamond in the release-hole region.

- First fabrication and testing of SMM piezoresistive RFMEMS resonators.
- First achievement of SMM system integration.



Figure 1-2 Overview of single-material MEMS using polycrystalline diamond.

### **1.3 Dissertation Organization**

Chapter 2 presents an overview of current diamond MEMS technologies. In chapter 3, some characterization methodologies and experimental setups are described. In chapter 4, important technologies needed for building multilayer SMM structures are demonstrated. Typical surface micromaching steps in poly-C microfabrication are provided, leading to the successful fabrication of multilayer SMM devices reported at the end of chapter 4. In chapter 5, SMM piezoresistive resonators for RFMEMS applications are persented as a typical SMM example. The design, fabrication and testing of the device were performed. The frequency response was achieved using piezoelectric actuation and piezoresistive detection. The RFMEMS resonator needs high-vacuum packaging to improve its performance. Chapter 6 discusses the fabrication technology for a SMM thin film packaging process. SMM system integration is explored in Chapter 7 to measure the frequency response of piezoresistive resonator using MSP430 microcontroller. Chapter 8 presents conclusions of this study and considers possible future directions.

# **Chapter 2**

## **Diamond MEMS Technologies**

### 2.1 Introduction

This chapter presents an overview of current diamond MEMS technologies. First, different forms of diamond thin films, typically used in MEMS, and their growth mechanism and properties are briefly reviewed in section 2.2. The cause of the low electrical resistivity of undoped poly-C is discussed in section 2.3, along with the role of O<sub>2</sub> in increasing its resistivity. Contemporary patterning methods for poly-C films, and their challenges, are reviewed in section 2.4. Diamond piezoresistive sensors, perhaps being the most widely used diamond sensors, are then discussed in section 2.5. At the end of this chapter, state-of-the-art diamond RFMEMS, BioMEMS and thin film packaging results are briefly reviewed.

### 2.2 Forms of Diamond

There are several types of diamonds for different applications, including natural diamond, synthetic industrial diamond, chemical vapor deposition (CVD) polycrystalline diamond (poly-C) and diamond-like carbon (DLC). The properties of natural diamond are summarized and compared with other semiconductor materials in Table 2-1. Synthetic industrial diamond is produced by the High Pressure High Temperature (HPHT) synthesis process. Both natural diamond and HPHT diamond are single-crystalline (SCD). Considering the high cost and difficulty of processing, these two types of diamonds are very limited in MEMS applications.

However, CVD poly-crystalline diamond (poly-C) can still provide remarkable quality for MEMS application with relatively low fabrication cost.



Figure 2-1 SEM images of (a) microcrystalline diamond (MCD) [3], (b) nanocrystalline diamond (NCD) [17], (c) ultrananocrystalline diamond (UNCD) [17] and (d) diamond-like carbon (DLC) [18].

Properties	Si	GaAs	SiC	Natural Diamond
Density (g/cm <sup>3</sup> )	2.329	5.317	3.216	3.52
Melting point (°C)	1412	1240	2540	3827
Hardness (GPa)	8.5	7	24.8	100
Young's modulus (GPa)	130-180	85	700	1050-1200
Poisson's ratio	0.22-0.24	0.31-0.32		0.1-0.21
Lattice constant (Å)	5.43	5.65	4.36	3.57
Band Gap (eV)	1.12	1.42	3.0	5.45
Carrier mobility Electron $(cm^2/V \cdot s)$	1450	8500	400	1800-2000
Hole (cm <sup>2</sup> /V·s)	500	400	50	1600-2100
Dielectric constant	11.7	10.9	10	5.7
Breakdown voltage (x10 <sup>6</sup> V/cm)	0.37-0.5	0.6	2-3	4-20
Intrinsic resistivity ( $\Omega \cdot cm$ )	1x10 <sup>3</sup>	1x10 <sup>8</sup>		1x10 <sup>16</sup>
Thermal conductivity (W/cm·K)	1.5	0.5	5	20
Thermal expansion coef. $(x10^{-6}/^{\circ}C)$	2.6	5.9	4.7	1.1

Table 2-1 Comparison of diamond properties with other semiconductors

Three types of poly-C thin films have been synthesized and systematically studied, which exhibit different microstructure, surface morphology, and properties. Standard poly-C thin film deposition methods, based on microwave plasma chemical vapor deposition (MPCVD), hot filament chemical vapor deposition (HFCVD) or any other CVD methods, involve hydrogen-rich chemistry (CH<sub>4</sub>/H<sub>2</sub>=0.1-4%) [10] [11] [12], which results in microcrystalline diamond (MCD) (typically 0.5-10 µm grains) with columnar structures as shown in Figure 2-1 (a) [3] and nanocrystalline diamond (NCD) (typically 50-100 nm grains) in Figure 2-1 (b) [17]. NCD is usually defined as a small grain size poly-C with a very high nucleation density to allow very thin coalesced films as demonstrated by Philip *et al.* [13] and Sekaric *et al.* [14], and not the cauliflower type grown with high CH<sub>4</sub> levels or under conditions designed to reduce atomic hydrogen and thus increase re-nucleation [15] [16]. The MCD and NCD film surface coarsens with thickness. Correspondingly, they exhibit a rough, highly faceted morphology with root mean square (RMS) roughness typically ~10% of the film thickness. In contrast to the growth process for MCD and NCD films, ultrananocrystalline diamond (UNCD) films growth uses an argon-rich chemistry (typically Ar (99%)/CH<sub>4</sub> (1%)) with no hydrogen added [2] [4]. As a result, UNCD is a fundamentally different material. Grains of UNCD (typically 2-5 nm) are much smaller than those of MCD and NCD films as shown in Figure 2-1 (c) [17]. Unlike other poly-C, UNCD can be grown to any thickness with no appreciable increase in roughness due to the high re-nucleation rate resulting from its plasma chemistry [17].

In addition to poly-C, DLC is also exploited in MEMS. DLC is a metastable, amorphous carbon material which may contain microcrystalline phase of diamond. The SEM image of DLC is shown in Figure 2-1 (d) [18]. It contains a mixture of  $sp^2$  and  $sp^3$  carbon phases. DLC films in

general are very smooth and can be deposited at low temperatures. Various methods used in deposition have led to a wide range of DLC films. It can be deposited by plasma-enhanced CVD (PECVD), ion beam deposition, sputtering from a graphite target or laser ablation of graphite [3]. Based on different source gases and/or deposition methods, DLC can be either hydrogen free (a-C or ta-C) or hydrogenated (a-C:H or ta-C:H). Usually, both a-C and a-C:H represent a structure with a high sp<sup>2</sup>/sp<sup>3</sup> ratio. However, using high plasma density sputtering and plasma-enhanced deposition procedures films can be grown possessing a high fraction of sp<sup>3</sup> bonding. In order to distinguish them from sp<sup>2</sup> DLC, tetrahedral amorphous carbon (ta-C) was suggested by McKenzie [19] for high sp<sup>3</sup> amorphous carbon and ta-C:H was referred to hydrogenated tetrahedral amorphous carbon by Weiler [20].



Figure 2-2 Raman spectrum of (a) microcrystalline diamond (MCD) [21], (b) nanocrystalline diamond (NCD) [17], (c) ultrananocrystalline diamond (UNCD) [17], (d) diamond-like carbon (DLC) including a-C and ta-C [137].

	SCD	Density (g/cm <sup>-3</sup> ) 3.52	Hardness (Gpa) 100	Young's modulus (GPa) 1050-1200	sp <sup>3</sup> (%) 100	H (at%) <0.1	Gap (eV) 5.45	Surface roughness
MC ~(	CD (grain size 0.5-10 μm)	3.52	70-100	800-1200	~100	<1	5.45	400 nm – 1 μm
NCD	(grain size 50- 100 nm)		30-75	800-1020	>50	<1	2-4.7	50-100 nm
UNG	CD (grain size ~2-5 nm)	3.50	88-98	916-980	95- 98	<1	5.4- 5.65	20-40 nm
	ta-C (evaporated)	1.9-2.0	2-5	757			0.4- 0.7	5-100 nm
	ta-C (MSIB)	3.0	30-130		90±5	<9	0.5- 1.5	
DLC	ta-C:H	2.9	61		75	22-28		
	a-C:H (hard)	1.6-2.2	10-20	300	30- 60	10-40	0.8- 1.7	1-30 nm
	a-C:H (soft)	0.9-1.6	<5		50- 80	40-65	1.6-4	1.50 mm

Table 2-2 Properties of various forms of diamond [2] [20] [23-27]

Raman spectroscopy is one of the principle characterization tools to investigate diamond film quality. Good quality diamond shows a high-intensity peak at 1332 cm<sup>-1</sup> in the Raman scan with a very small Full Width at Half Maximum (FWHM) value as shown in Figure 2-2 (a) [21]. The broad band approximately centered at 1550 cm<sup>-1</sup> is attributed to three bands: the D and G peaks of polycrystalline graphite at around 1345 cm<sup>-1</sup> and 1560 cm<sup>-1</sup>, and a low intensity band centered approximately at 1470 cm<sup>-1</sup> attributed to a tetrahedrally bonded diamond precursor [22]. The Raman spectrum of NCD, UNCD and DLC are shown in Figure 2-2 (b-d) [17] [137], respectively. MCD has the highest quality indicated by its high intensity of sp<sup>3</sup> peak and amorphous carbon has the lowest quality as indicated by the almost invisible sp<sup>3</sup> peak. A comparison of basic properties of various forms of diamond is given in Table 2-2 [2] [20] [23] [24] [25] [26] [27].

## 2.3 Unexpected Electrical Conductivity of Undoped Poly-C

The electrical resistivity of natural diamond is approximately  $10^{14}$ - $10^{16} \Omega \cdot \text{cm}$  [28] [29] [30]. Due to its high electrical resistivity, diamond is a good candidate for electrical insulation in MEMS applications. But after certain surface treatment is done to remove the poly-C surface conduction as demonstrated previously, undoped poly-C films still show a resistivity of a few orders of magnitude lower than natural diamonds. The conduction mechanism in undoped poly-C has been extensively studied [31] [32] [33] [34] [35] [36] [37] [38] [39] [40] [41] [42] [43] [44]. The conduction of undoped poly-C can be attributed to the impurities or traps in diamond and the grain boundary regions. After comparing the quality of diamond grown under different CH<sub>4</sub>/H<sub>2</sub> ratio with SEM and Raman, Kulkarni *et al.* attributed the low resistivity on the order of  $10-10^{6} \ \Omega$ -cm to the grain boundary regions [36] [37]. Lee applied Cu electroplating to diamond film showing that the grain boundaries within the films were the main conduction path in undoped poly-C [41]. The conduction mechanism due to impurities had also been investigated by deriving the activation energy based on the change of the electrical conductivity as a function of the temperature. Usually, boron corresponds to the activation energy about 0.3-0.4 eV. Nitrogen corresponds to the activation energy about 1.4-1.6 eV. The electrical conductivity as a function of inverse temperature in [35] suggests that electrically active boron and nitrogen incorporation is correlated with the film grown along (111) and (100) directions, respectively. Wang, *et al.* [40] also reported activation energy of 1.68 eV which was attributed to Si trapped in a vacancy in the diamond lattice. It is speculated that the possible source of boron contamination in their films could be the Si wafer which is heavily doped with boron. Stoner *et al.* found that hydrogen trap can also be an origin for conduction of undoped poly-C with activation energies less than 0.6 eV [44]. The real cause for low resistivity of undoped poly-C still needs further study.

## 2.4 Patterning of Poly-C

For integrating poly-C devices into Si-based microsystems, patterning of poly-C films using techniques consistent with silicon processing is required. As wet etching poly-C is impractical, the commonly used patterning techniques are in-situ patterning, selective dry etching. Laser beam [45] and synchrotron excited radiation [46] were also reported as an optional way to etch poly-C.

Selective deposition is achieved by selective nucleation or by masking the areas where growth is not desirable. SiO<sub>2</sub> was successfully used as a masking layer by Masood *et al.* [47], Roppel *et* 

*al.* [48] and Davidson *et al.* [49]. Patterned amorphous Si masking was also reported [50]. Hirabayashi et al. first nucleated the Si substrate by ultrasonic treatment, then used a photoresist mask for etching Si to 60-70 nm using an Ar+ ion beam [51] or reactive ion etching [50]. A simple selective nucleation technique, which consists of spinning a layer of photoresist premixed with diamond powder and lithographically patterning it, was developed by researchers at Michigan State University [47].

However, in all of these in-situ techniques, the lateral growth at the pattern edges and, in some cases, growth on unwanted areas can pose serious problems for small feature sizes (< 10  $\mu$ m). Consequently, selective dry etching is a better choice for poly-C patterning. Selective etching of poly-C with SiO<sub>2</sub> or Si<sub>3</sub>N<sub>4</sub> as a mask, was performed at atmospheric pressure, in oxygen environment at 700 °C, in a rapid thermal processor [47]. Dry etching technique using conventional dry etching tools has become the primary approach for poly-C patterning in recent years, such as ion beam etching (IBE) [52] [53], reactive ion etching (RIE) [54] [55] [56] [57] [58] [59] [60] [61] [62] [63] [64] [65] [66] [67] [68] [69] [70] [71] [72] [73] [74], electron cyclotron resonance etching (ECR) [75] [76] [77] [78] [79] [80] [81] and inductively coupled plasma etching (ICP) [82] [83] [84] [85].

RIE of poly-C has been extensively studied for the realization of micro- and nano-structures. Generally,  $O_2$  is used to etch diamond through the generation of CO and  $CO_2$  byproducts [86]. However, diamond column structures (also called needles or whiskers) which are usually created in pure  $O_2$  plasma etching unintentionally [59] [60] [61] [70] [72] [73] [74]. These column structures are generated simultaneously at the beginning of the etching process along grain boundaries due to the difference in chemical composition of grains and grain boundaries. The columns on the grains are attributed to the intrinsic defects in the crystals and unintentionally redeposited micro-masks. The origin of the columns on grains and grain boundaries were also explained as the difference of crystal planes in [74] for grains have (100) plane and grain boundaries have (111) plane. These diamond column structures can even be formed without a metal coating existing on the sample [73] [74].

It was suggested that a small amount of  $CF_4$  in  $O_2$  can increase the etch rate by avoiding the formation of diamond column structures [55] [65], however, fluorine (F) in plasma etching causes damage to Si substrates or other underlying layers such as SiO<sub>2</sub> or Si<sub>3</sub>N<sub>4</sub> during the patterning of poly-C [55]. As a result, a new etching recipe is needed to preserve the underlying layer after the removal of poly-C, while maintaining a reasonable etch rate and aspect ratio.

#### 2.5 Diamond Piezoresistive Sensors

Piezoresistance is a change in resistivity of a material under the influence of an externally applied stress or strain. The piezoresistive property of semiconductors such as doped diamond under uniaxial stress can be explained by the valence-band split-off model which is discussed in APPENDIX A. The strain will change the concentration of heavy and light holes, so the strain can change the conductivity of the *p*-type semiconductor. The piezoresistivity of a material is normally quantified by the gauge factor (*GF*). The *GF* of a material is defined as the fractional change of resistance ( $\Delta R/R$ ) per unit strain  $\varepsilon$  which is the figure of merit for piezoresistive material and expressed by equation (2.1).

$$GF = (\Delta R/R)/\varepsilon \tag{2.1}$$

The piezoresistive effect in silicon MEMS is widely used for pressure, force and acceleration sensors. The most prominent example is piezoresistive acceleration sensors for airbags in cars. Commercially available sensors cannot operate at high temperatures because of pn-insulation of the piezoresistors. Since the first report of piezoresistivity in SCD (single crystalline diamond) and poly-C by researchers at Michigan State in 1992 [87], there has been enormous interest in the utilization of p-type poly-C prepared by chemical vapor deposition (CVD) as a material for sensor applications due to its high GF as shown in Figure 2-3 [88] and its potential application at high temperatures. A number of studies have focused on the piezoresistive effect of poly-C [89] [90] [91] [92] [93] [94] [95] [96] [97] [98] [99] [100] [101] [102] [103] [104] [106] [180]. The GF is usually characterized by measuring the change in resistance of the piezoresistor by deflecting the structural material which is usually made of a cantilever or diaphragm made of either Si, stainless steel or undoped poly-C. The reported GFs of poly-C as a function of electrical resistivity are given in Figure 2-4 with details given in APPENDIX A. GFs are found to increase with the increase of resistivity and are mainly in the range of 5 to 100 at room temperature.



Figure 2-3 Comparison of the highest gauge factors achieved in previous work at room temperature between poly-C and other metals and semiconductors. [88]



Figure 2-4 Gauge factors of poly-C at room temperature in literatures [89-104] [106] [180].



Figure 2-5 (a) Cantilever test structure to characterize the gauge factor [99], (b) a pressure sensor using *p*-type poly-C piezoresistor [109], (c) diamond acceleration sensor (left) and diamond pressure sensor (right) [99], (d) position sensor made of *p*-type poly-C in cochlear implant probe [180].

Figure 2-5 (a) shows a cantilever made of undoped poly-C was used as a structural material to characterize the *GF* of *p*-type poly-C piezoresistor [99]. Among the applications of poly-C piezoresistive sensors, pressure sensors have been studied the most [105] [106] [107] [108] [109]. Figure 2-5 (b) and (c) show pressure sensors and an accelerometer from the work in [109] and [99]. A position sensor using poly-C was developed for application in cochlear implant probe [180] as shown in Figure 2-5 (d). RFMEMS resonators with piezoresistive detection are also being developed [110] [111]. DLC also has piezoresistive effect. Pressure sensors and force

sensors made of DLC have also been reported [112] [113] [114] [115] [116]. Piezoresistive detection, which is receptive to device scaling without decreasing the detection sensitivity, can help be improved by (a) adjusting the piezoresistor dimensions and (b) increasing the mobility of holes by using intra-grain piezoresistor.

#### 2.6 Diamond RFMEMS

Radio frequency micro electro mechanical systems (RFMEMS) can yield on-chip micromechanical resonators with ultra-high quality factors over 10,000 at GHz frequencies in both vacuum and air making them excellent candidates for broadband wireless communications [117]. So far, polycrystalline silicon has been the preferred material for RFMEMS resonators, having been demonstrated with measured quality factor O's above 8400 at a frequency of 50.35 MHz [118] for free-free beam design, 2650 at a frequency of 1.156 GHz [119] for radial disk design, and 2800 at a frequency of 1.52 GHz for extensional wine-glass design [120]. While already impressive, such an achievement could be greatly amplified if Q's >10,000 were achievable at the same GHz frequencies and in the same tiny sizes. In order to further extend frequencies, the use of alternative structural materials with higher acoustic velocities ( $\sqrt{E/\rho}$ , E is Young's modulus and  $\rho$  is density) than poly-silicon such as silicon carbide [121] [122] and diamond, has been explored. Among the currently available set of thin-film materials, single crystal diamond offers the largest acoustic velocity on the order of 18,076 m/s [123]. This is to be compared with the 8,024 m/s of single crystal silicon [124] and 11,500 m/s of silicon carbide [125], which are 2.25X and 1.57X smaller, respectively. Given that resonance frequency is generally proportional to acoustic velocity, diamond potentially provides higher micromechanical resonator frequencies. In addition, the electrical and mechanical properties of polycrystalline silicon begin to rapidly degrade at temperatures above 350 °C, making it increasingly unsuitable for high temperature applications.

Fabrication and testing of diamond-based RFMEMS resonators have been reported in the literature. They have been made of SCD [126], MCD [127] [128] [129] [130], NCD [14] [131] [132] [133] [134] [135], UNCD [4] [136] and DLC [137] [138]. Reported quality factor Q's at resonant frequencies  $f_0$ 's of diamond-based RFMEMS resonators are given in Table 2-3. The dissipation (affecting Q) is mainly due to the relaxation of large number of defects in the bulk or the surface of the film. Although SCD seems to be the best potential material for high Qachievement, SCD fabrication technology is not developed as well as other types of diamond material. As a result, poly-C is the most feasible candidate to achieve higher quality factor. Sepúlveda *et al.* [129] reported a quality factor Q for poly-C (grain size ~300 nm) cantilevers in the range of 4,000-165,000, which includes values higher than the O's observed for UNCD cantilevers with comparable dimensions [138]. They suggested that, as the film percentage occupied by the nucleation layer containing fine grained diamond is increased, the quality factor Q of the resonators is reduced. This is consistent with the idea that the higher dissipation in the UNCD or DLC is mainly due to the presence of a higher proportion of grain boundaries and defects. Low quality factor O of NCD resonators in Table 2-3 can be attributed to the fact that as the resonator dimensions become smaller, dissipation due to surface effects also play a role. This conclusion is supported by the work of Imboden et al [139].
	Quality factor	Measured Actuation		
Resonator design	Q	frequency $f_0$	Method	Mater.
Cantilever [126]	510	~230 KHz	Piezoelectric	SCD
Doubly-clamped beam [140]	6,225	3.022 MHz	Electrostatic	MCD
Combdrive [130]	36,460	27.352 KHz	Electrostatic	MCD
Cantilever [128]	15,260	384.9 KHz	Piezoelectric	MCD
Cantilever [129]	116,000	8-50 KHz	Piezoelectric	MCD
Doubly-clamped paddle [14]	2,400-3,500	6-30 MHz	Piezoelectric	NCD
Mesh membrane [14]	3,000	8-20 MHz	Piezoelectric	NCD
Disk resonator [141]	11,555	1.51 GHz	Electrostatic	NCD
Doubly-clamped beam [133]	~3,000	14-157 MHz	Magnetomotive	NCD
Tuning fork [134]	8,000	37.0 MHz	Laser	NCD
Square paddle (pillared) [134]	1,500	14.77 MHz	Laser	NCD
Squarepaddle(freelysuspended) [134]	3,000	13.85 MHz	Laser	NCD
Ring resonator [134]	5,000	40.18 MHz	Laser	NCD
Antenna structure [135]	23,200	630.6 MHz	Magnetomotive	NCD
Harp structure [132]	600-2,400	17-66 MHz	Magnetomotive	NCD
Cantilever [4]	11,460	11 KHz	Piezoelectric	UNCD
Cantilever [136]	5,000-16,000	12-35 KHz	Piezoelectric	UNCD

Table 2-3 The reported room temperature Q's at resonant frequencies  $f_0$ 's of flexural resonators



Diamond Gate Contact (p<sup>+</sup>-Diamond)

(a)





Figure 2-6 (a) Schematic cross-setion of diamond microwave relay (b) SEM-micrographs of microwave relay structures (beam length: 2500  $\mu$ m); left: coplanar line pattern deposited on the substrate and oriented orthogonally to the cantilever: right: complanar line deposited onto the beam surface and oriented along the cantilever. [142] [143]

A classical electrostatically driven, cantilever-beam-structure switch made of poly-C is shown in Figure 2-6 [142] [143]. RFMEMS switches have great potential in RF communications because of their high isolation and low power consumption. The main part is a freestanding poly-C cantilever that can be deflected electrostatically by applying a voltage between the cantilever gate contact and the substrate gate contact. All essential parts of the switch are made of diamond except the silicon substrate. Thus, heat generated caused by the power loss in the signal contacts can be effectively dissipated over the cantilever and the substrate. Also, no sticking caused by the melting of metal contacts or formation of insulating oxide layers on the signal contacts can occur. An on-state attenuation of ~3 dB and an off-state attenuation of -23 dB at 10 GHz were reported in [143]. The results indicate that for operation in air a diamond microswitch exhibits a maximum frequency of operation approximately eight times higher than a silicon microswitch of identical geometry [144].

### 2.7 BioMEMS

- 5 μm 20 μm
- 2.7.1 Microfluidic channel

Figure 2-7 SEM of microfluidic channels with 4 µm tunnels. [146]

Heat dissipation is an increasingly important challenge for the electronics industry. Miniaturized coolers using a liquid passing through microfluidic channels represent one of the most advanced technologies used to extract thermal energy from microelectronics circuits such as CPUs. Microfluidic elements are also an essential part of lab-on-a-chip systems to perform complex chemical and biochemical synthesis, as well as analytical tasks. Ramesham, *et al.* used anisotropic chemical etching of silicon and selective growth of diamond to form poly-C microfluidic channels of 17- $\mu$ m wide [145]. This resulted in channels with diamond forming the top half of the structure and silicon the bottom half. Such channels may not be useful in many microfluidic applications due to the relative chemical reactivity and thermal instability of silicon compared to diamond. Guillaudeu, *et al.* developed free-standing all-diamond fluidic channels using Si as a mold as shown in Figure 2-7 [146]. This process is simpler but only narrow width can be designed to close the trench in a proper time by additional poly-C growth.

#### 2.7.2 Neural Probes

Many research groups are developing unique processes for creating neural probes. However, silicon can be an undesirable probe material as, without modification, it has been found to have poor flexibility, solubility in water, and can induce undesirable glial responses [147]. Many groups have solved this problem through coating silicon with biocompatible materials. As an alternative to coating silicon, undoped diamond has been explored as a substrate material due to its structural properties and high biocompatibility [148]. The optical transparency of the diamond substrate is important for *in vitro* experiment because it allows the electrode's position on the probe to be easily located under a microscope. Boron-doped poly-C sensors and electrodes were integrated in Si cochlear microprobes for the first time [149]. Boron-doped poly-C electrodes have also been shown to be effective for the detection of neurotransmitters [150]. Chan *et al.* [151] [152] [153] developed a process for fabricating neural probes using undoped poly-C as the structural material and boron-doped poly-C as the electrode material. Non-diamond materials are

used for interconnects and electrical insulation layers. This probe has been used to measure electrical neural activity. In addition, the comparatively wide potential window (the reported values range from 1.4 to 4 V) in an aqueous environment [154] [155], low double layer capacitance (ranging from 5 to 40  $\mu$ F/cm<sup>2</sup>) [156], chemical inertness and stability, and resistance to fouling of boron doped diamond make it an excellent site material for electrochemically detecting neurotransmitters in vitro as shown in Figure 2-8 [157].



Figure 2-8 Poly-C probe used for electrochemically detecting neurotransmitters. [157]

## 2.8 Diamond Packaging



Figure 2-9 Completed sealed poly-C packaging. [158]

Although MEMS packaging can take advantage of mature packaging techniques from the semiconductor IC industry, MEMS packaging is still complicated due to the diversity of applications. Poly-C has emerged as a novel material for MEMS packaging [158] [159]. The potential of poly-C as a thin film packaging material was explored in view of its excellent mechanical strength, electrical properties, chemical stability and thermal management as a heat sink. A poly-C thin film packaging process was developed to encapsulate poly-C cantilever resonators [159] integrated with highly doped poly-C interconnects [158], as shown in Figure 2-9. The efficacy of the encapsulation process was evaluated by measuring the resonator frequency and quality factor before and after the packaging process, indicating the poly-C packaging can be integrated into conventional MEMS fabrication without affecting the yields of the device. The

fluidic hermeticity was also tested by an HF soak test. The encapsulated cantilevers are suitable for applications in harsh environments and are able to withstand chemical and mechanical attack and high temperatures. A technology using an all-diamond package for wireless-integrated Microsystems with boron-doped diamond as built-in interconnects was also developed [160] and demonstrated the ability to achieve an outstanding level of heat dissipation.

# **Chapter 3**

# **Experimental Design and Setup**

### 3.1 Introduction

First, the designs of MEMS-based characterization structures are demonstrated. These structures are widely used in this work. The advantage of using these designs is that it can be integrated with the designer's own chip design very conveniently without taking up too much space. Then the experimental setup and the equations that are used to characterize the gauge factor (GF) of piezoresisive poly-C film are discussed. Finally the design and characterization of the circuits that are used to measure the frequency response of piezoresistive RFMEMS resonators in Chapter 5 are discussed.

## 3.2 Design of MEMS-Based Characterization Structures

#### 3.2.1 Electrical resistivity characterization of doped poly-C

### Four-point probe MEMS structure

The purpose of the 4-point probe is to measure the resistivity of any semiconductor material to exclude the influence from contact resistivity. A micro-fabricated structure with a four-point probe configuration is used to characterize the electrical resistivity of doped poly-C in this work as shown by the schematic diagram in Figure 3-1. Doped poly-C resistors were fabricated on an insulating layer (thermal SiO<sub>2</sub> or undoped poly-C) with a length of 410  $\mu$ m and a width of 20, 40 and 60  $\mu$ m first and then metal interconnects (either Cr/Au 50/500 nm or Ti/Au 50/500 nm) were patterned in a four-point probe measurement setup. A current of 10  $\mu$ A was applied to the two

bottom electrodes, while the voltage drop was measured across the two top electrodes. With 10  $\mu$ A applied current, the resistive heating is less than 0.35 mW, which leads to a negligible temperature rise. The thickness of the doped poly-C resistor was measured by a Dektak 6M surface profilometer. Current is driven through the outer two electrodes while voltage is sensed on the inner electrodes. The resistivity is calculated based on the current source and sensed voltage as expressed in equation (3.1).

$$\rho = \frac{V}{I} \cdot \frac{wt}{L} \tag{3.1}$$

where I is the applied current, V is the voltage drop, and L, w and t are the length, width and thickness of the doped poly-C resistor, respectively.



Figure 3-1 Cross-sectional view of micro-fabricated 4-point probe structure.

### Van der Pauw MEMS Structure

A micro-fabricated Van Der Pauw structure is also used to measure the electrical resistivity of doped poly-C. The top view is shown in Figure 3-2 with the same cross-sectional view as in Figure 3-1. The square membrane is made of doped poly-C with a size of 400x400  $\mu$ m<sup>2</sup>. First 10  $\mu$ A of current was applied to two contacts, while the voltage drop was measured across the two

remaining metal pads. The same procedure was then repeated with perpendicularly oriented contacts as well as by switching measurement polarity. When the structure is symmetrical, the electrical resistivity can be expressed by equation (3.2).



Figure 3-2 Top view of micro-fabricated Van der Pauw structure.

### 3.2.2 Contact resistivity characterization

### Kelvin bridge MEMS structure

A micro-fabricated Kelvin bridge structure is used to characterize the contact resistivity as shown by Figure 3-3. A given current I flows from the metal layer to the doped poly-C layer through the contact area. The generated voltage difference V is created between the other two electrodes. The contact resistivity can be calculated by

$$\rho_C = R_C \cdot A = (V/I) \cdot A \tag{3.3}$$

where  $\rho_c$  is the contact resistivity in  $\Omega \cdot cm^2$ ,  $R_c$  ( $\Omega$ ) is the contact resistance, and A (cm<sup>2</sup>) is the size of the contact area. Several values of contact areas used in this work are 100, 400, 900, 1600, 2500 and 10000 (10<sup>-8</sup> cm<sup>2</sup>).



Figure 3-3 Schematic diagram of micro-fabricated Kelvin bridge structure used to measure the contact resistivity between doped poly-C (p poly-C) and metal with or without highly doped interlayer (p+ poly-C).

#### transmission line model MEMS structure

A transmission line model (TLM) was also used to study the contact resistivity as shown in Figure 3-4. Metal contacts to the mesa of doped poly-C were fabricated on top of the insulating layer which could be SiO<sub>2</sub> or undoped poly-C. The total resistance value  $R_T$  (obtained by V/I) can be expressed by [161]

$$R_T = R_S \frac{d + 2L_T}{Z} \tag{3.4}$$

for rectangular geometry, where  $R_S$  is the sheet resistance of the semiconductor layer and d is the spacing. d has values of 6, 10, 15, 25, 30 and 35 µm. Z is the length of the rectangular pads, i.e. contact length. In this study, the contact length was designed for four series, e.g. 150, 300, 450 and 600 µm, in order to compare the effect of contact length on contact resistivity. The transfer length  $L_T$  is the averaged portion of the contact width H (H=200 µm in this study) which current actually flows and can be expressed by

$$L_T = \sqrt{\rho_c / R_S} \tag{3.5}$$

where  $\rho_c$  is contact resistivity in  $\Omega \cdot cm^2$ . Fitting the  $R_T$  value of different spacing d to the appropriate formula (equation (3.4)) yields the free parameters  $R_S$  and  $L_T$ , and  $\rho_c$  can be calculated from equation (3.5).



Figure 3-4 Schematic diagram of micro-fabricated structure of linear Transmission line model (TLM) pattern used to characterize the contact resistivity of doped poly-C



3.2.3 Electrical resistivity characterization of undoped poly-C

Figure 3-5 A MEMS design used to characterize the electrical resistivity of undoped poly-C

Typically, the four-point probe resistivity arrangement, based on four needle-like contacts having a very small contact area (4 x  $10^2 \ \mu m^2$ ), is not usable for resistivity measurements on undoped poly-C because the measured current is too low to be measured. In the present study, using a special design as shown in Figure 3-5, the contact area for each of the four metal contacts was increased to 6.58 x  $10^7 \ \mu m^2$ , and the inter-probe spacing was decreased to 4  $\mu m$ . The five orders of magnitude increase in contact area and the small length to width ratio of the resistor makes it possible to measure the high resistivity of undoped poly-C up to  $10^{17} \ \Omega$ ·cm when a Semiconductor Analyzer (Agilent 6514C) is used. In addition, convenient measurement can even

be performed only using the inner two electrodes. For example, when the resistivity of a 1- $\mu$ mthick undoped poly-C film is 10<sup>12</sup>  $\Omega$ ·cm, a resistance of approximately 24 M $\Omega$  would be expected, which is well below the 200 M $\Omega$  measurement range of a typical hand-held digital multimeter (MASTECH MY-65).



3.2.4 Residual stress characterization

Figure 3-6 Micro strain gauge used to measure the internal stress in poly-C thin film. Test beam, slope beam and indicator beam are released.

A micro strain gauge (from [162]) is used to evaluate the internal film stress as shown in Figure 3-6. Design values are given in Table 3-1. The design utilizes a lever arm structure to mechanically amplify strains caused by stress. Residual stress, either tensile or compressive, generates a small displacement  $\delta_{tb}$  in the "test beam" (beam elongates or contracts). This displacement in turn generates an angular deflection along the "slope beam", which then generates a much larger displacement  $\delta_v$  at the tip of the indicator beam that can be read visually via optical microscope. Via this structure, the strain in the "test beam" is effectively amplified to a larger displacement. The mechanical gain factor is given by:  $\delta_{\nu}/\delta_{tb}=3L_{ib}/(2L_{sb})=37.5$ . The residual stress can be represented in equation (3.6).

$$\sigma_0 = \frac{\delta_{tb}E}{37.5 \times L_{tb}} \tag{3.6}$$

Table 3-1 Design	values of micro strain	gauge

Symbol	Element	Value (µm)	Symbol	Element	Value (µm)
L <sub>tb</sub>	Length of the test beam	500	W <sub>tb</sub>	Width of the test beam	30
L <sub>sb</sub>	Length of the slope beam	20	W <sub>sb</sub>	Width of the slope beam	3
L <sub>ib</sub>	Length of the indicator beam	500	W <sub>ib</sub>	Width of the indicator beam	3
h	Thickness of thin film	~3			



### **3.3** Measurement of Gauge Factor by Beam Bending Method

Figure 3-7 Schematic diagram of the measurement setup to characterize the gauge factor (GF)

The measurement setup to characterize the GF of doped poly-C is shown in Figure 3-7. An undoped poly-C cantilever with doped poly-C at the anchor end was micro-fabricated. The tip of the cantilever beam was pressed down using a micromanipulator. Another two micromanipulators were located on the two metal contacts at the two ends of doped poly-C in order to read the change of the resistance by multi-meter by every certain amount of change of the tip deflection. A Labview program is made to facilitate the measurement which is discussed in APPENDIX F.

The GF can be computed using the equation below.

$$GF = \frac{\Delta R/R}{\varepsilon}; \ \varepsilon = \frac{3h}{2L^3} \left( L - \frac{l_1}{2} \right) \delta$$
(3.7)

where  $\Delta R/R$  is the relative resistance change of doped poly-C resistor,  $\varepsilon$  is the longitudinal average strain in doped poly-C, L and h are the length and thickness of undoped poly-C cantilever, respectively.  $l_1$  is the length of doped poly-C resistor and  $\delta$  is the tip deflection of the cantilever. In equation (3.7), the thickness of doped poly-C resistor is assumed to be zero and its effect on the cantilever deformation is also ignored.

## 3.4 Piezoresistive Resonator Testing

In order to achieve the frequency response of the piezoresistive resonator in the study presented in Chapter 5, a piezoelectric actuation setup, a driver circuit to actuate the piezoelectric sheet and a detection circuit to detect the piezoresistive response are needed.



### 3.4.1 Piezoelectric actuation setup

Figure 3-8 Schematic diagram of testing sample mounted on the piezoelectric actuator

The schematic for the testing sample mounted on the piezoelectric actuator is shown in Figure 3-8. A single-layer piezoceramic sheet (PSI-5A4E, from Piezo System, INC.) with a thickness of 0.267 mm is used as piezoelectric actuator. For piezoelectric material, if a voltage is applied across it, the electrical field that developed would cause it to deform. For the rectangular piezoelectric actuator used in this study as shown in Figure 3-9, when a voltage is applied longitudinally, a displacement  $\Delta L$  occurs and can be closely approximated by:

$$\Delta L = \varepsilon \cdot L_0 \approx \pm E \cdot d_{33} \cdot L_0 \approx \pm V \cdot d_{33} \tag{3.8}$$

where  $\Delta L$  is the change in length (m),  $\varepsilon$  is the strain, E is the electrical field strength (V/m) and  $L_0$  is the length of actuator (m). The term  $d_{33}$  is the piezoelectric coefficient (m/V), where the first subscript identifies the axis of the field and the second subscript identifies the axis of displacement. In order to generate a large enough vibration amplitude, a high voltage has to be applied.



Figure 3-9 Schematic diagram of actuation of piezoelectric actuator

The use of the spacers underneath the piezoelectric sheet is optional; they are included here to decrease energy loss due to ultrasonic radiation to the board underneath. Silver paint was used to bond the spacer to the piezoceramic sheet since the interface between the two sheets requires electrical conduction. Super glue was used to bond the actuator to the silicon die and also hold

the spacers in place on the board. It is important to ground the Si substrate. Since the actuator can be regarded as a single capacitor (~10 nF), when it is driven by an AC voltage, charge and discharge of the capacitor occur. This effect can induce unwanted current in signal transmission lines on the testing sample. With Si substrate grounded, the induced current can be greatly reduced.

### 3.4.2 Driver circuit for piezoelectric sheet



Figure 3-10 Schematic diagram of the driving circuit

When the actuator is driven by a periodic voltage source whose frequency is below or above the resonant frequency of the actuator, it can be modeled by a single capacitor. Therefore, the impedance presented to the driving source  $Z_{load}$  is, to a good approximation, simply:

$$Z_{\text{load}} = 1/(2\pi \cdot f \cdot C_{\text{PA}}) \tag{3.9}$$

where f is the frequency of driving source and  $C_{PA}$  is the equivalent capacitance of the piezoelectric actuator.  $C_{PA}$  of the piezoelectric sheet used in this work is 10 nF. A high voltage results in high current due to the relative large capacitance of the actuator. Since the actuator is driven at high voltage (targeted peak-to-peak  $V_{AC}$  is 48 Volts) with relatively high frequency (10-100 kHz), the driving circuit must allow high speed or high slew rate. However, to combine the high power and high speed, the choices of suitable amplifiers with cost less than \$100 shrink very fast. A power amplifier PA78 together with its evaluation board EK60 is used in this work to build the inexpensive driver circuit as shown by the schematic diagram in Figure 3-10. The circuit can deliver current up to 10 A with a slew rate of 300 V/µs where the current is limited by the two power MOSFETs at the output and the slew rate is controlled by the power amplifier PA78. The driving circuit was characterized when the input is a sine wave or square wave as shown in Figure 3-11. A square wave is used to simulate the high-frequency pulses generated by the microcontroller. The circuit shows a gain of 17.5-18 dB at 40 kHz. The gain keeps constant when the driving frequency is less than 100 kHz.



Figure 3-11 Characterization of the driving circuit by oscilloscope

### 3.4.3 Piezoresistive detection circuit



Figure 3-12 Schematic diagram of piezoresistive detection circuit

Figure 3-12 illustrates the circuit design and configuration for measuring the frequency response of a resonator using piezoresistive detection. A Wheastone bridge is used to convert the change of the resistance in the piezoresistors to a small AC voltage.  $R_{PZR}$  in the Wheastone bridge is the total resistance of a piezoresistive sensor on the testing sample whose resistance changes when the resonator is vibrating.  $R_1$  and  $R_2$  are the variable resistors that are used to null the output voltage from the Wheastone bridge ( $V_A$ - $V_B$ ) when resonator is not vibrating.  $R_{REF}$  is the reference resistor that is identical in dimensions and normal resistance to  $R_{PZR}$ , but is located on the substrate to cancel out the voltage due to the  $R_{PZR}$ .  $R_{REF}$  also provides a first-order cancellation of thermal and electromagnetic disturbances that may occur to  $R_{PZR}$  (assuming  $R_{PZR}$  and  $R_{REF}$  experience the same disturbance). The Wheastone bridge is used since this

configuration has maximum sensitivity of the output voltage,  $V_{\rm A}$ - $V_{\rm B}$ , over the varied resistance. A constant 24 V<sub>dc</sub> voltage, achieved by voltage regulation, is used as a stable and low-noise input source for the piezoresistive sensor. When  $R_{\rm PZR} / R_1 = R_{\rm REF} / R_2$ , the output voltage from the Wheastone bridge can be expressed in (3.11)

$$V_{\rm A} - V_{\rm B} \approx \frac{\Delta R \cdot R_2}{\left(R_{\rm PZR} + R_1\right) \cdot \left(R_{\rm REF} + R_2\right)} V_{\rm in} \tag{3.10}$$

where  $\Delta R$  is the resistance change of the piezoresistor when resonator is vibrating. When  $R_{PZR} = R_{REF} = R$ , equation (3.10) can be simplified as given below

$$V_{\rm A} - V_{\rm B} \approx \frac{\Delta R}{4R} V_{\rm in} \tag{3.11}$$

Since  $V_{\text{in}} = 24 \text{ V}$ ,  $V_{\text{A}} - V_{\text{B}} \approx 6\Delta R/R$ .



Figure 3-13 The frequency response of the detection circuit

An instrumentation amplifier (IN103) is used to amplify the small AC signal with a gain of 40 dB. Next, two active filters are used to achieve a fourth order Butterworth high pass filter with corner frequency of 9 kHz and gain of 0 dB. The Butterworth filter type is selected because it provides the flattest pass-band response as compared to Bessel and Chebyshev filter types. The low-noise opamp (OPA128) before the output is used to further amplify the signal with a gain of 20 dB. Since the total gain of the circuit after the Wheastone bridge in Figure 3-12 is 60 dB, the output voltage from the circuit  $V_{\text{out}}$  is equal to  $6000\Delta R/R$ . The frequency response of the detection circuit is characterized by plotting  $V_{\text{out}}$  as a function of frequency. A flat pass-band between 10-100 kHz is clearly seen. The actual gain in the pass-band is 58.8 dB.

# **Chapter 4**

# **Single-Material MEMS Poly-C Film Technologies**

## 4.1 Introduction

In order to build single-material MEMS (SMM) structures and devices, the achievement of MEMS-quality poly-C thin films is essential. Major challenges are addressed in this chapter including the achievement of highly insulating poly-C thin film, conformal poly-C coating, dry etching of poly-C and the high-resolution patterning of poly-C film with high surface roughness. The electrical properties characterization and the residual stress characterization of poly-C thin films are also discussed in this chapter to complete the study on poly-C film used in SMM.

# 4.2 Achievement of Highly Insulating Poly-C Thin Film

Insulating poly-C layers are important for SMM devices. When conducting transmission lines are fabricated on top of undoped poly-C in SMM devices, crosstalk can be a problem depending upon spacing between the neighboring lines. However, it is a challenge to achieve a high electrical resistivity of undoped poly-C above  $10^6 \Omega \cdot \text{cm}$  in a growth system which is also used for doped poly-C due to unintentional boron doping. Since O<sub>2</sub> can suppress the boron incorporation into poly-C film as discussed in section 2.3, O<sub>2</sub> is used in this work to increase the resistivity of undoped poly-C from ~ $10^3 \Omega \cdot \text{cm}$  to above  $10^9 \Omega \cdot \text{cm}$ .

### 4.2.1 Poly-C growth with the presence of O<sub>2</sub>



Figure 4-1 Characteristics of the poly-C film seeded by 5% DPLW spin method. (a) front surface of the film; (b) side view of the film; (c) AFM image of the film; (d) Raman spectrum of the film.

CVD poly-C films (MCD and NCD) require a pretreatment step to generate seeds (or nuclei) on the substrate before growth begins. Ultra-high seeding density is required for highly insulating thin film, because a low nucleation density or small variation in seeding density could

create a non-covered area in the poly-C thin film, such as pin-holes, leading to current leakage through the non-covered area. The seeding procedure used to achieve ultra-high seeding density of approximately  $10^{11}$  cm<sup>-2</sup> is discussed in APPENDIX A.

	Gas components	Microwave Power	Pressure	Time
Step 1	H <sub>2</sub>	2.3 kW	10 Torr	10 min
Step 2	CH <sub>4</sub> :H <sub>2</sub> =3:200	2.3 kW	40 Torr	30 min
Step 3	O <sub>2</sub> :CH <sub>4</sub> :H <sub>2</sub> =0.5:2:200	2.3 kW	40 Torr	

Table 4-1 The processing parameters in various steps used in this study

The processing parameters for various steps used for insulating poly-C growth in MPCVD are given in Table 4-1. Etching in H<sub>2</sub> plasma is performed first to remove silicon dioxide and also impurities on the substrate and substrate holder. Relatively low pressure is applied here which results in a larger plasma size. This step is crucial because it provides a well-defined and reproducible substrate surface for poly-C growth. Also, a stable temperature is achieved across the substrate surface, which can help the control of poly-C growth. Next, the sample is exposed to a rich CH<sub>4</sub> in H<sub>2</sub> plasma in order to form SiC and a diamond nuclei layer. In this process, diamond powders are chemically bonded to each other and are bonded to the silicon substrate. It is also suggested that oxygen should be introduced only after the nucleation stage because the addition of O<sub>2</sub> can suppress diamond nucleation and result in a decrease of nucleation density [163]. Finally, O<sub>2</sub> is introduced to improve the diamond quality primarily by preferential sp<sup>2</sup> etching and the suppression of boron or silicon incorporation in the grown film. The  $CH_4$  to  $H_2$  ratio is also lowered relative to step 2 leading to higher diamond quality.

As-grown poly-C film was characterized by SEM, AFM (atomic force microscope) and Raman spectroscopy (excitation wavelength of 532 nm, laser spot size of  $< 2 \mu m$ ). The poly-C film was grown on a substrate having a seeding density of  $10^{11}$  cm<sup>-2</sup>. The film shows wellfaceted grains. The average grain size is about 0.7 µm as observed by SEM imaging, as shown in Figure 4-1 (a). The thickness is inspected by taking an SEM image of the sample's cross-section as shown in the Figure 4-1 (b). As the ratio of  $O_2/CH_4$  varies from 0-75%, the growth rate is computed as the sample's thickness divided by its growth time as shown in Figure 4-2. The highest growth rate occurs when the O<sub>2</sub> to CH<sub>4</sub> ratio is 25%, and no poly-C is grown when the O<sub>2</sub> to CH<sub>4</sub> ratio is higher than 75%. Measurement of surface roughness can be achieved by AFM imaging as shown in Figure 4-1 (c). An average arithmetic surface roughness ranges from 30-100 nm for a poly-C layer having a thickness of 2-6 µm. Raman spectroscopy is a well-known method to investigate the diamond film quality. A sharp, narrow and high intensity peak observed at 1332.9-1333.5 cm<sup>-1</sup> with an absence of appreciable amorphous carbon signature around 1350 cm<sup>-1</sup> and 1560 cm<sup>-1</sup> indicates good quality of poly-C, as shown in Figure 4-1 (d) when the film's thickness ranges from 2-6  $\mu$ m. The peak shift away from 1332 cm<sup>-1</sup> is most likely caused by the residual stress. The full width at half-maximum (FWHM) is 7.8-8.4 cm<sup>-1</sup>. The narrow FWHM is comparable to the reported FWHM in [21], also indicating good poly-C quality.



Figure 4-2 Growth rate of poly-C with the presence of O<sub>2</sub> versus ratio of O<sub>2</sub> to CH<sub>4</sub>.

### 4.2.2 Characterization of electrical resistivity

Throughout the study of films grown under different conditions, it was observed that different processes performed before the undoped poly-C growth can affect the film resistivity. The measured resistivities of samples A-D grown with/without  $O_2$  presence and with different process histories prior to growth are given in Table 4-2. All measurements were performed after the hydrogenated layer was removed by  $O_2$  plasma etching in a plasma asher as discussed in APPENDIX C. It should be noted that only n-type Si wafers are used for undoped poly-C growth, because it was suspected that boron can diffuse out of *p*-type Si wafers and unintentionally incorporate into undoped poly-C films [157].

Sample	Process history of MPCVD chamber	Sample growth with O <sub>2</sub> ?	Resistivity $\rho$ ( $\Omega$ ·cm)
А	Doped poly-C growth	No	$10^2 - 10^3$
В	Undoped poly-C growth without O <sub>2</sub>	No	$10^4 - 10^5$
	for over 100 hours		
С	Doped poly-C growth	Yes	$10^{5} - 10^{6}$
D	Undoped poly-C growth with O <sub>2</sub>	Yes	$10^9 - 10^{11}$
	for over 100 hours		

Table 4-2 The measured resistivities of samples A-D



Figure 4-3 I-V plot of a 0.68-µm-thick undoped poly-C grown with O<sub>2</sub> present based on high ultra-high nucleation density.



Figure 4-4 (a) SEM images of the fabricated structure with four metal lines in 4-point-probe configuration and (b) measurement result.

The results in Table 4-2 indicate that undoped poly-C resistivity is relatively low if it is grown after doped poly-C growth. This is due to the fact that boron can stick on the dome or the substrate holder, and act as a dopant for the next undoped poly-C sample. It is also observed

from Table 4-2 that the addition of O<sub>2</sub> during growth can increase the electrical resistivity of undoped poly-C significantly. So in order to achieve a highly insulating poly-C layer, a sample must be grown with O<sub>2</sub> present after a long period of undoped poly-C growth with O<sub>2</sub>. A 0.68µm-thick undoped poly-C film based on ultra-high nucleation density is grown under this condition. The measurement result is shown in Figure 4-3 with the measurement setup discussed in APPENDIX D. This result corresponds to an electrical resistivity of ~2·10<sup>10</sup>  $\Omega$ ·cm. Such high resistivity is not necessary for most SMM applications. The undoped poly-C layers used in this work usually has a relatively lower resistivity. The resistivity is generally characterized using the on-chip MEMS design discussed in section 3.2.3. The fabricated structure is shown in Figure 4-4 (a) and a typical *I-V* plot of 3-µm-thick undoped poly-C film is shown in Figure 4-4 (b) corresponding to an electrical resistivity of ~10<sup>9</sup>  $\Omega$ ·cm.

### 4.3 Characterization of Semiconducting and Metallic Poly-C

To study the efficacy of poly-C as a semiconductor and as a conductor, *in situ* doping of poly-C was achieved by introducing trimethylboron (B(CH<sub>3</sub>)<sub>3</sub>, TMB), diluted in hydrogen (TMB/H<sub>2</sub>=0.098%), during the poly-C growth. The doping concentration is controlled by changing the TMB ratio while keeping the ratio of  $CH_4/H_2$  constant to make the poly-C semiconducting or metallic.



### 4.3.1 Characterization of electrical resistivity

Figure 4-5 (a) Optical image and concept diagram of the sample for resistivity measurement (b) resistivity of doped poly-C as a function of TMB ratio.

Doped poly-C resistivity as a function of TMB ratio is performed using a 4-point probe configuration as demonstrated in section 3.2. Doped poly-C resistors were patterned on thermal  $SiO_2$  with a length of 410 µm and a width of 20, 40 and 60 µm. Then Cr/Au interconnects were

patterned in a four-point probe measurement setup as shown in Figure 4-5 (a). Cr/Au is chosen because it is resistant to hydrofluoric acid (HF), which is commonly used in SMM to remove sacrificial SiO<sub>2</sub> layers. The measurement of doped poly-C resistivity as a function of TMB ratio (0-1 ppm) is shown in Figure 4-5 (b). 1000 ppm O<sub>2</sub> is added to increase the electrical resistivity by 10-100 times to allow for more resistivity control.

TMB (ppm)	Resistivity $\rho$ ( $\Omega$ ·cm)	Doping concentration $N_A$ (cm <sup>-3</sup> )
0.010	58.145	2.949x10 <sup>19</sup>
0.025	43.608	$3.492 \times 10^{19}$
0.050	21.823	4.045 x10 <sup>19</sup>
0.100	7.016	5.071x10 <sup>19</sup>
1.000	0.010	>1.7 x 10 <sup>20</sup>

Table 4-3 Electrical resistivity, doping concentration for various TMB ratios

The activation energy can be inferred by plotting the change of resistance of doped poly-C resistors as a function of temperature. The temperature-control setup is demonstrated in APPENDIX B. The resistance can be read directly from a digital multimeter. The resistance R is dependent on the activation energy  $E_a$  (eV) and absolute temperature T (K) which is rewritten from section 2.3 as shown below

$$R = C \exp(E_a/kT) \tag{4.1}$$

where  $E_a$  is the activation energy, k is the Boltzmann constant and T is temperature in K. Since  $\ln R = \ln C - E_a/kT$ , a straight line in a plot of  $\ln R$  vs. 1/T is expected. A linear fit was used to find the slope, where slope  $= E_a/kT$  is the fit parameter. As a result,  $E_a$  can be calculated. Then, the doping concentration  $N_A$  cm<sup>-3</sup> can be derived using equation (4.2) [164].

$$E_a = 0.37 - 6.7 \times 10^{-8} N_A^{1/3} \text{ eV}$$
(4.2)


Figure 4-6 Electrical resistances ( $k\Omega$ ) of a 600-800 nm thick boron-doped poly-C layer versus reciprocal temperature (K).



Figure 4-7 (a) Kelvin Bridge Structure used to measure the contact resistivity between p poly-C (piezoresistive layer) and metal with or without p+ poly-C interlayer; (b) Plot of contact resistivity as a function of the resistivity of piezoresistive layer with the inset of I-V plot by Semiconductor Analyzer indicating excellent ohmic contact.

The change of resistance of doped poly-C resistor versus reciprocal temperature was plotted in Figure 4-6 for five typical TMB ratios. The sample was placed in a box during the measurement to avoid light-induced photo-current. The activation energy  $E_a$  is computed using the fit parameter of the slope.  $N_A$  based on the known value of activation energy  $E_a$  is then computed.  $N_A$ 's for typical TMB ratios were listed in Table 4-3. The resistance of highly doped poly-C remains nearly constant when the temperature increases, indicating zero activation energy which corresponds to a doping concentration greater than 1.7 x 10<sup>20</sup> cm<sup>-3</sup>.

#### 4.3.2 Characterization of contact resistivity

Kelvin Bridge (KB) structures are used for contact resistance measurements between doped poly-C and Cr/Au. The detection scheme is demonstrated in section 3.2.2. The fabricated structure is shown in Figure 4-7 (a). The *I-V* curve was plotted with a semiconductor analyzer (where a p+ poly-C interlayer was sandwiched in between the p (lightly-doped) poly-C layer) indicating a good ohmic contact (even without annealing), as shown by the inset of Figure 4-7 (b). This is probably due to the partial carbide formation by heat treatment during sample preparation and during the lift-off process [165], or the high roughness of the poly-C film [166]. The contact resistivity between p-type poly-C (piezoresistive layer) and Cr/Au with or without a p+ poly-C interlayer as a function of the resistivity of p-type poly-C layer is plotted in Figure 4-7 (b). The result, as measured by a Kelvin Bridge structure, shows that the p+ poly-C interlayer can reduce the contact resistivity to 30% -45% of the original values. This result is very important for piezoresistive sensor designs.

# 4.4 Characterization of Residual Stress

The study of residual stress minimization is essential, because unacceptably high residual stress may cause buckling, warping, or other damages in released SMM structures. Residual stresses in thin films are typically measured by the curvature method, XRD-based methods and Raman peak-shift methods [167]. Film stress can also be monitored and characterized by micromechanical structures. The curvature method and micromechanical structures are used in the current study.

#### 4.4.1 Substrate curvature measurement

A general uni-axial residual stress field in a thin film can be represented by equation (4.3).

$$\sigma_{total} = \sigma_0 + \sigma_1 \frac{y}{h/2} \tag{4.3}$$

where  $y \in (-h/2, h/2)$  is the coordinate across the thickness h, with an origin chosen at the film's midplane.  $\sigma_0$  is the mean stress and  $\sigma_1$  is the stress gradient. The stress gradient is caused by localized effects including atomic diffusion through h to the film/substrate interface, interstitial or substitutional defects and atomic peening [168].  $\sigma_0$  can be calculated based on the Stoney's equation as given in equation (4.4):

$$\sigma_0 = \frac{E_s d_s^2}{6R(1 - \nu_s)h}$$
(4.4)

where  $E_s$ ,  $v_s$  and  $d_s$  are the Young's modulus, Poisson ratio and thickness of the substrate, and h is the thickness of the thin film. Values of  $\sigma_0$  are determined through measurement of R (radius of curvature).

Three poly-C samples grown with  $CH_4$  to  $H_2$  ratio of 0.5%, 1% and 2%, were used for this study to evaluate the stress range. All the samples were grown on a 2" circular silicon substrate with an average thickness of 275  $\mu$ m. A Dektak stylus profilometer was used to measure the curvature of the wafer. Since all the samples have a concave top surface (indicating tensile stress), the measurements were taken on the backside. A scan length of 3 cm was run along the diameter of sample and 6 points were measured every 10  $\mu$ m as shown in Figure 4-8 (a). The sample with CH<sub>4</sub> to H<sub>2</sub> ratio of 1.0% is given as an example here. The data was smoothed in Matlab, and subsequently fit to a quadratic as shown in Figure 4-8 (b). Equation (4.5) was then used to calculate the curvature.

$$\kappa = \frac{1}{R} = \frac{|y''(x)|}{\left(1 + [y'(x)]^2\right)^{3/2}}$$
(4.5)

where  $\kappa$  is the curvature, R is the radius of curvatusre, x is the scan direction and y is normal to the scan direction. Since bending is not perfectly circular, the curvature varies with x as shown in Figure 4-8 (c). An average curvature was used to compute the residual stress. In this study, the substrate thickness  $d_s$  is 275 µm. Young's modulus  $E_s$  and Poisson ratio  $v_s$  are 165 GPa and 0.27 for silicon, respectively. The film thickness h was measured by profilometer after poly-C was patterned. Finally, the residual stress for these three samples were computed and given in . All stresses are tensile and below 100 MPa. Stresses increase with CH<sub>4</sub> concentration (become more tensile when CH<sub>4</sub> concentration is high). These stress values are smaller or similar to the reported stress values of poly-C films used for MEMS. For example, NCD film has a stress of 180-390 MPa in [169], MCD has a stress approximately 500 MPa in [170] and UNCD has a stress of 50-100 MPa in [171].

 $CH_4/H_2$  Thickness h Curvature  $\kappa$  Curvature Radius R Stress Strain 0.6535 m<sup>-1</sup> 0.5% 4±0.4 µm 153.0 cm 47±4.7 MPa ~0.00482% 1.3495 m<sup>-1</sup> 1.0% 6±0.6 µm 74.1 cm 64±6.4 MPa ~0.00656% 1.5329 m<sup>-1</sup> 6±0.8 μm 2.0% 65.2 cm 73±7.3 MPa ~0.00752%

Table 4-4 Residual stresses for  $CH_4/H_2$  of 0.5%, 1.0% and 2.0%



Figure 4-8 (a) Raw data of curvature measurement, (b) quadratic fit of smoothed curvature data, and (c) curvature variation with x position along the scan length.

#### 4.4.2 Characterization using micro-mechanical structure



Figure 4-9 Reading from micro strain gauge indicating a tensile stress of 26-36 MPa

A MEMS structure is used to characterize the residual stress. Details of the structural design are discussed in section 3.2.4. A poly-C film grown on a silicon substrate with a  $CH_4/H_2$  ratio of 0.5% was used in this study. After poly-C patterning, the structure was released by isotropic etching of silicon using a Xactix XeF<sub>2</sub> tool. The released structure is shown in the optical image in Figure 4-9. Due to the existence of out-of-plane stress gradients, the tip-position of the indicator beam cannot be determined under optical microscopy because the microscope's depth of field is not sufficient to focus on the tip and the fingers (which are used to read the tip movement) at the same time. As a result, the tip-position was inspected by SEM. The reading shows a tensile stress of 26-36 MPa. This result is comparable to the stresses calculated by curvature characterization. A more accurate reading would require a more sensitive structure, which could be obtained by increasing the ratio of  $L_{ib}$  to  $L_{sb}$ . The extremely small residual stress of the poly-C films in this study guarantees the success of building multilayer SMM structures.

Micro-fabricated cantilever beam arrays were also used to inspect the stress gradient. Poly-C used in this study includes undoped poly-C grown with or without  $O_2$  during growth, and also highly doped poly-C. The SEM images of the released cantilever beams are shown in Figure 4-10. Undoped poly-C films grown without  $O_2$  on top of SiO<sub>2</sub> generally have an almost zero stress gradient as shown in Figure 4-10 (a). Beams made of undoped poly-C films grown with  $O_2$  on top of SiO<sub>2</sub> bend upward as shown in Figure 4-10 (b) with  $\sigma_1$  being positive in equation (4.3). Higher  $O_2$  concentrations result in more significant bending. As a result, low  $O_2$  concentration, such as 1000 ppm, is usually used for released insulating poly-C structures as long as the electrical resistivity of the insulating poly-C remains high enough for applications. Cantilever beams made of highly doped poly-C films grown on top of SiO<sub>2</sub> always stick to the substrate after release with  $\sigma_1$  being positive in equation (4.3). The elucidation of the origins of stress gradients requires further study. It should be noted that the downward bending in Figure 4-9 may be attributed to the poly-C growth on Si instead of on SiO<sub>2</sub>.



Figure 4-10 Stress gradients characterization using cantilever beam arrays. Beams are made of (a) undoped poly-C grown without  $O_2$ , (b) undoped poly-C grown with 2000 ppm  $O_2$  and (c) highly doped poly-C with 1 ppm TMB.

# 4.5 Multilayer Single-Material MEMS Fabrication Technologies

#### 4.5.1 Achievement of conformal poly-C coating

In order to build thin film structures, the film must be continuous everywhere. However, problems exist with the step coverage of poly-C film grown by MPCVD due to low seeding density. Step coverage for different spin speeds and various seeding concentrations in DPLW was studied.

A 6-µm plasma-enhanced-chemical-vapor-deposition (PECVD) SiO<sub>2</sub> was patterned to create a large step height as shown in Figure 4-11 (a). Typical step heights encountered in surface micromachining are usually less. It has been found that low seeding density will result in discontinuous film along the line of intersection, even after an extremely long growth time. Figure 4-11 (b) shows a sample that was grown for 15 hours with a growth rate of  $\sim 0.15 \,\mu m$  per hour; holes still remain in the film, especially along the line of intersection. Even though these holes can be eventually sealed in another 10-20 hours, the film is too thick for use in many MEMS applications. Samples with different spin rates and various powder concentrations in DPLW were studied. The continuity of samples was inspected by SEM after 15 hours of poly-C growth. In this study, 15 hours of growth time is used because it will result in a thickness of 2.5-3 µm which is suitable for a structural material with both excellent flexibility and mechanical strength. In addition, thicker film also brings difficulty to the dry etching. It was found that ultrahigh seeding density has to be used in order to generate continuous film within a proper growth time. This can be achieved by either using DPLW with high concentration of diamond powders or spinning DPLW at low spin rate. 5% DPLW spun at 2000 rpm can achieve ultra-high seeding density of  $10^{11}$  cm<sup>-2</sup> without obvious powder aggregation. This condition will also result in continuous film with the presence of step as shown in Figure 4-11 (c) with film thickness of 2.5-3.0  $\mu$ m. The larger grain size along the line of intersection compared to other areas results from the relatively lower seeding density.



Figure 4-11 (a) PECVD SiO<sub>2</sub> was patterned to form a high step as shown by the optical image on the right side. (b) Low seeding density results in a discontinuous film along the line of intersection as shown by SEM images on the right side. (c) 5% DPLW spun at 2000 rpm results in a continuous film with the absence of powder aggregation. (d) Powder aggregation when powder concentration in DPLW is too high or spin rate is too low. (e) Film still has pinholes after 15 hours growth caused by the relatively low seeding density when 5% DPLW is spun at 3000 rpm.

If the powder concentration is higher than 5% or the spin rate is lower than 2000 rpm, significant powder aggregation will occur. For example, 8.43% DPLW spun at 2000 rpm and 0.5% DPLW spun at 500 rpm can result in a continuous film in the presence of a step in a rather short growth time, but the film also shows significant powder aggregation as shown in Figure 4-11 (d). On the other hand, if the powder concentration is 5% with spin rate higher than 2000 rpm, it may result in pinholes in poly-C film after 15 hours of growth. For example, Figure 4-11 (e) shows the film grown for 15 hours with pinholes when 5% DPLW is spun at 3000 rpm. As a result, 5% DPLW spun at 2000 rpm is an optimized seeding condition which can generate a continuous 2.5 or 3-µm-thick poly-C film over the step with a near absence of powder aggregation on the sample.

#### 4.5.2 Optimization of dry etching of poly-C

Recipes of RIE of poly-C films of different thicknesses are required to obtain smooth etched surfaces and a satisfactory etch-rate. For the first time, RIE-related damage to underlying substrate (specific to RIE of poly-C) was eliminated to optimize the technology for single- and multi-material MEMS made from poly-C.

As discussed in chapter 2,  $O_2$  is generally used to etch diamond through the generation of CO and CO<sub>2</sub> byproducts. However, diamond columns may be formed unintentionally in  $O_2$  plasma. Furthermore, these columns can even be formed without a pre-coated metal layer. Since the etchrate of a poly-C layer is determined by the etching of diamond columns, the existence of these columns prevents the effective removal of poly-C. It was suggested that a small amount of CF<sub>4</sub> in  $O_2$  can increase the etch rate by prohibiting the formation of diamond column structures [53]. However, fluorine (F) in plasma etching causes damage to Si substrates and other underlying layers such as SiO<sub>2</sub> or Si<sub>3</sub>N<sub>4</sub> during the patterning of poly-C [55]. Such damage is inevitable, because poly-C growth starts with diamond seeds and, thus, the initial layer (this thickness depends on seeding density) of poly-C has holes between the growing seeds. The thickness of this porous layer is 200-300 nm when ultra-high nucleation density of  $10^{11}$  cm<sup>-2</sup> was used in our study. When the poly-C etching process reaches this thickness, the etching of the underlying substrate begins. This becomes a serious problem in poly-C MEMS fabrication, especially with poly-C films grown with low nucleation density. The roughness of as-grown poly-C is another factor to be considered during poly-C patterning. If the etch rate of poly-C is assumed to be uniform all over the sample, the roughness of poly-C would be "transferred" to the Si substrate or other underlying layers (such as SiO<sub>2</sub> or Si<sub>3</sub>N<sub>4</sub>) and can even be amplified due to the likely higher etch rate of the underlying layer as compared to that of poly-C in the same etching environment.

RIE was carried out using a RIE etching system, Plasmatherm 790. Typical film thickness is 2-4  $\mu$ m in this study. A 1.5- $\mu$ m-thick Al layer, patterned through lift-off, was used as a masking layer during RIE of Poly-C. Al is used as a mask because highest mask selectivity was measured as compared to Ti, Au and SiO<sub>2</sub>. The measured selectivity of Al and poly-C is above 100 in O<sub>2</sub> plasma at a pressure of 50 mTorr and RF power of 300 W. The selectivity reduces to approximately 11 when a small amount of CF<sub>4</sub> is present in plasma etching. SEM was used to observe surface morphologies. Energy dispersive spectroscopy (EDS) was used to characterize the etched surface. Etching depth was estimated from Dektak stylus profilometer measurements and surface roughness was measured by an Atomic force microscope (AFM).



Figure 4-12 Perspective view of remaining diamond columns after 2- $\mu$ m-thick poly-C film on sample A was etched in O<sub>2</sub> plasma; Inset is the close-up view of diamond columns. (b) Perspective view of remaining poly-C after 3.5- $\mu$ m-thick poly-C film was etched in O<sub>2</sub> plasma; Inset is the perspective view of the remaining porous poly-C layer which was broken intentionally on sample B'. (c) Holes were created in Si by CF<sub>4</sub> etching through the pores of remaining porous poly-C on sample B''. Inset is the close-up view of holes underneath the porous poly-C layer. (d) EDS analysis of the etched surface on sample A and B indicating the existence of Al.

Etched surfaces, as shown in Figure 4-12 (a-d) were used to demonstrate the problems associated with the typically-used etching scheme from the literature, i.e. poly-C etching in O<sub>2</sub> plasma with/without CF<sub>4</sub> gas. Al masks were used in this study. Sample A with a poly-C thickness of 2 µm and sample B with a poly-C thickness of 3.5 µm were prepared for this study. After the removal of poly-C in O<sub>2</sub> plasma on sample A, scattered diamond columns were observed on the etched surface as shown by the SEM image in Figure 4-12 (a). On the other hand, a thin poly-C layer was left after the poly-C on sample B was etched in O2 plasma as shown in Figure 4-12 (b). Then, Sample B was cut into two pieces, B' and B''. The remaining poly-C layer on sample B' was broken intentionally to find the thickness of it. A thickness of 1  $\mu$ m was determind as shown in the inset of Figure 4-12 (b). Before breaking the remaining poly-C layer on sample B", CF<sub>4</sub> gas was added to O<sub>2</sub> plasma to continue etching for a time. Then, the remaining layer was broken intentionally and holes in Si were observed as shown in Figure 4-12 (c). It is believed that CF<sub>4</sub> passed through the pores of the remaining poly-C layer and etched the underlying Si. Actually, both diamond columns on sample A and the remaining poly-C layer on sample B were etched very slowly in O2 plasma. It is believed that the generation of diamond columns and thin poly-C layers is caused by nano-masks, and these nano-masks impede the fast removal of poly-C underneath. EDS analysis on sample A and B provides the evidence for the existence of nano-masks as shown in Figure 4-12 (d). It indicates the presence of Al on the etched surface, revealing that Al particles are redeposited on the etched poly-C surface, probably in the form of Al<sub>2</sub>O<sub>3</sub> serving as nano-masks. It is also speculated that the density of nano-masks increases with time. Therefore, separate nano-masks begin connecting with each other after long etching periods and result in a thin porous layer as shown in Figure 4-12 (b). It should be noted that such a nano-masking layer also exists even when the sample does not have an Al masking layer. The etching result is almost the same as those shown in Figure 4-12 (a) and (b). The origin of the Al nano-masking layer is probably from Al emanating from the chamber itself—it has already been pointed out that materials sputtered from the chamber itself can act as nano-masks in [68].  $CF_4$  can be added to  $O_2$  plasma to avoid the nano-masking. However, the underlying Si surface can be greatly damaged due to the presence of fluorine (F) as indicated in Figure 4-12 (c). The same problem occurs to the SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub>.



Figure 4-13 Etch rate of poly-C and Al at various pressures in (a) O<sub>2</sub> plasma and (b) CF<sub>4</sub> plasma

Condition	Gases	Flow Rate	RF Power	Pressure	Etch Rate	Slope
		(sccm)	(W)	(mTorr)	(nm/min)	Angle
Step 1	CF <sub>4</sub>	5	300	50	~30	55°
Step 2	O <sub>2</sub>	50	300	80	~12	~75 <sup>°</sup>
Step 3	H <sub>2</sub>	20	300	50	<5	>80°

Table 4-5 Three-step etching conditions of poly-C



Figure 4-14 Schematic diagram of 3-step etching of poly-C

Due to the above problems with the typical poly-C etching recipes, a new three-step RIE of poly-C films is used in this work in order to obtain smooth etched surfaces. Etching parameters are given in Table 4-5. Pressures were selected to achieve the highest etch rate based on the measurement results in Figure 4-13. Slope angles as inspected by SEM are also given. The schematic diagram of 3-step etching is shown in Figure 4-14. In addition to the etched surface, study of the side wall is also important for certain applications, such as AFM cantilever tip production. As a result, the influence of side wall slope angle is also considered in Figure 4-14. Slope angle is defined as shown in Figure 4-14 (a). It should be noted that slope angles of the side wall in O<sub>2</sub> and H<sub>2</sub> plasmas is assumed to be 90°. CF<sub>4</sub> plasma is used in the first step to etch the poly-C film without the formation of diamond columns until a thin continuous poly-C layer remains as shown in Figure 4-14 (a). The slope angle is  $55^{\circ}$ . Since pure O<sub>2</sub> plasma effectively does not attack the commonly used underlying layers (such as Si, SiO2 and Si3N4) as indicated in Figure 4-12 (a) and (b), it is used next to continue etching the poly-C film accompanied by the formation of diamond columns as shown in Figure 4-14 (b). It should be noted that the area between the columns was etched faster than the columns themselves in O<sub>2</sub> plasma as indicated by the increasing height of diamond columns on the side wall in Figure 4-14 (c). Therefore, the etch rate of the poly-C layer in O<sub>2</sub> plasma is limited by the etch rate of the columns. Due to the slow etch rate of these diamond columns, they are difficult to remove entirely in O<sub>2</sub> plasma, but the diamond columns on the etched surface are much shorter than those on the side wall. Therefore, they can be effectively removed in H<sub>2</sub> plasma as shown in Figure 4-14 (d). An ultrasmooth etched surface is achieved using this 3-step RIE process as shown in Figure 4-15 (a). The

root mean square roughness ( $R_{RMS}$ ) is less than 2 nm as measured by AFM. The use of H<sub>2</sub> as the termination step can also allow the etching time in CF<sub>4</sub> plasma to be less strict. If the etching time of CF<sub>4</sub> plasma was not controlled very well, a relatively thick poly-C layer would remain for O<sub>2</sub> plasma etching. It is highly possible that a porous layer as in Figure 4-12 (b) would be formed instead of the scattered short diamond columns as in Figure 4-12 (a). Such a relatively thick porous layer will take a very long time to be remove in O<sub>2</sub> plasma due to the low etch rate.

Although there is a fast removal rate of poly-C using the 3-step etching scheme, the residual diamond columns on the side wall at the foot still remain even when diamond columns on other areas are already removed in H<sub>2</sub> plasma as shown in Figure 4-15 (b). This is mainly due to the equivalent etch rate of diamond columns and the areas between columns on the side wall in H<sub>2</sub> plasma. In order to avoid the formation of diamond columns on the side wall, more anisotropic etching in all plasmas are required. Currently, a smooth side wall is achieved by using only  $O_2$  plasma and H<sub>2</sub> plasma as shown in Figure 4-15 (c). 10 mTorr is used for  $O_2$  plasma etching because it generates almost 90° slope angle. However, the removal of poly-C is not as fast as the 3-step etching scheme in Figure 4-14, especially when a porous poly-C layer remains after  $O_2$  plasma etching. As a result, further study of anisotropic etching in CF<sub>4</sub> plasma is required to achieve both a smooth etched surface and side wall.



Figure 4-15 (a) The SEM image of smooth etched surface using multi-step recipes in Table I; (b) a dot of 5  $\mu$ m diameter was designed to study the etching of side wall; (c) smooth side wall is achieved when poly-C was etched in O<sub>2</sub> plasma and H<sub>2</sub> plasma at 10 mTorr.

#### 4.5.3 Surface micromachining of poly-C structures and devices

In order to produce multilayer poly-C structures, a general surface micromachining process for poly-C film must be developed. The process for fabricating released structures with one poly-C layer is shown in Figure 4-16. The process is developed based on traditional Si-based surface micromachining technology, and includes the following basic steps: (i) photolithography, metal evaporation and lift-off processes are performed to pattern Al masking layers on top of poly-C; (ii) The pattern of Al is transferred to poly-C by dry etching; (iii) Al is removed by Al Etchant Type A, and a hydrofluoric acid (HF) wet etch is used to remove the sacrificial SiO<sub>2</sub> layer, leaving the poly-C film suspended above the substrate.

The smallest feature size of photolithographic patterning is limited by the surface roughness ranging from 30-100 nm for the 2- to 6-µm-thick poly-C layers that are commonly used in SMM. This is because radiation reflects off of a rough surface and travels back through the photoresist interfering with the incoming waves during exposure leading to unwanted exposure in the

masked areas. The resolution is also limited by stress-induced wafer-warping. The wafer usually shows concave shape with the center of wafer lower than the edge after poly-C is grown on top of the substrate. Therefore, the mask cannot have hard contact with the center of the wafer during exposure. This also leads to the unwanted exposure in the masked areas. The resolution of the photolithographic patterning also propagates to poly-C patterning performed afterward. As a result, photolithographic parameters must be optimized to achieve high-resolution patterning. These parameters primarily include the type of photoresisit, photoresist spin rate, soft bake temperature and time, rehydration time, exposure time and development time (refer to the APPENDIX E for detailed parameters). By using an optimized photolithography recipe and lift-off process, a narrow Al layer can be patterned on a rough poly-C surface successfully as shown in Figure 4-17. The released cantilever beams are shown in Figure 4-18 based on the surface micromachining steps in Figure 4-16. A repeated sequence of procedure in Figure 4-16 can be used to build multilayer SMM structures as shown in Figure 4-19.



Figure 4-16 Process of fabricating released structures with one poly-C layer



Figure 4-17 Left: Photoresist 9260 patterned on top of rough poly-C surface using optimized photolithography parameters; Right: an Al ribbon was patterned successfully via lift-off step.



Figure 4-18 Released cantilevers using the surface micromachining techniques.



Figure 4-19 SEM images of various SMM structures made of poly-C.



(a)



(b)

Figure 4-20 Inspection of (a) lateral gap and (b) vertical gap using fabricated poly-C structures.

The primary purpose of fabricating testing structures such as those in Figure 4-18 and Figure 4-19 is to inspect the efficacy of surface micromachining techniques. Beams having a width of 3  $\mu$ m can be fabricated with photolithography patterning as shown in Figure 4-18. The smallest lateral gap that can be achieved by photolithography is also examined as shown Figure 4-20 (a), which shows a 3  $\mu$ m gap. Smaller features of about 2  $\mu$ m can be achieved if the stress-induced wafer-warping is less and the contact between the mask plate and wafer is more sound.

The vertical gap between the suspended poly-C layer and the underlying below it was inspected and is detailed in Figure 4-20 (b). Flat and suspended beams having a uniform gap above the underlying layer are ascribed to the relatively small tensile stress of the film. Also, there appears to be bumps formed on the backside of the suspended beam. This is probably due to etching of SiO<sub>2</sub> through the gaps between diamond seeds at the early stage of poly-C growth. The size of these bumps can be suppressed by using higher quality SiO<sub>2</sub> as well as high seeding density. Higher quality SiO<sub>2</sub> can be achieved by annealing at high temperatures in N<sub>2</sub>.

In relation to how small features can be fabricated, recently, cantilever arrays having a minimum width of 400 nm were fabricated as shown in Figure 4-21 with the help of e-beam writing. This result contradicts the comment in [171] which mentions that such small features cannot be patterned in microcrystalline diamond (MCD) due to the limitation imposed by the  $\sim$ 1 µm grain size. This shows that the device dimensions can be even smaller than the grain size. Therefore it is possible to build a single-crystal sensor within one grain of poly-C.



Figure 4-21 poly-C beams with widths varying from 200 nm  $- 2 \mu m$  inspected by (a) SEM and (b) optical microscopy.

# 4.6 Single-Material MEMS Examples

Based on the technologies demonstrated in this chapter, three single-material MEMS (SMM) devices were proposed and fabricated successfully. They are SMM capacitive RFMEMS, SMM integrated field emission devices (IFEDs) and SMM piezoresistive sensors. They have been proven to be mechanically and electrically functional after preliminary testing, but further testing is still needed for future study. They are not only all-diamond MEMS but also SMM, because an undoped insulating poly-C layer and a highly conducting poly-C layer are used in all three devices. In addition a semiconducting poly-C is also used in the SMM piezoresistive sensor. This is the first time that the SMM concept is used for RFMEMS, IFEDs and piezoresistive sensors.

The advantage of using SMM has been discussed in chapter 1. Taking these three devices as SMM examples, the efficacy of using the previously demonstrated technologies to build functional SMM devices are demonstrated.



4.6.1 SMM capacitive RFMEMS

Figure 4-22 SEM images of fabricated capacitive SMM RFMEMS and the fabrication process flow.

Poly-C is a promising material for RFMEMS resonators, because a resonator made of poly-C will have a much higher resonant frequency than a silicon resonator having equivalent dimensions due to its higher acoustic wave velocity. Poly-C cantilever beams have shown the

highest quality factor of cantilevers made of any polycrystalline material [172] at a resonant frequency of 536 KHz. Furthermore, poly-C is more advantageous for high temperature applications as compared to silicon.

A SMM capacitive RFMEMS was fabricated as shown in Figure 4-22. As it can be seen, poly-C surface micromachining technology is similar to poly-Si technology in concept. A released bridge resonator with spacing around 1.0 µm was fabricated. The bottom poly-C layer is used as the I/O electrodes and interconnects. It consists of an undoped poly-C layer and a highly doped poly-C layer which are grown in sequence in an MPCVD chamber on an n-type Si wafer as shown in Figure 4-22 (a). N-doped Si wafer is used instead of p-doped to avoid the outdiffusion of boron from *p*-type Si when undoped poly-C groupwth is under way. The undoped poly-C layer serves as an insulation layer. High resistivities over  $10^9 \ \Omega$  cm are achieved by using O<sub>2</sub> in the growth environment. Poly-C growth for another 4 hours creates a high resistance between the highly doped poly-C and the Si substrate. Then 1 ppm TMB is used to grow the p+(highly doped) poly-C having a resistivity on the order of  $10^{-3} \Omega \cdot cm$ . After poly-C growth the poly-C is patterned by RIE. All these techniques have been illustrated in Chapter 4. Then a 1- $\mu$ m-thick sacrificial PECVD SiO<sub>2</sub> layer is deposited and annealed in N<sub>2</sub> at 750 °C. The SiO<sub>2</sub> layer is patterned by RIE to create the anchor for the eventual resonators as shown in Figure 4-22 (b). The SiO<sub>2</sub> layer supports the structural poly-C layer during deposition and patterning, and defines the electrode-to-resonator gap spacing. A 2.3 µm-thick highly doped poly-C layer is then grown and patterned by RIE, as shown in Figure 4-22 (c). In the final step of the process, the wafer is dipped into a solution of hydrofluoric acid (HF) which etches away the sacrificial SiO<sub>2</sub> layer. Since poly-C is chemically inert, even chemically-aggressive HF causes no damage to the poly-C surface. A supercritical  $CO_2$  drying step is not necessary for poly-C beams. Free-standing poly-C resonators are achieved after careful cleaning, as shown in Figure 4-22 (d).

#### 4.6.2 SMM integrated field emission devices

The fabrication process shown in Figure 4-22 can also be applied to build IFEDs as shown in Figure 4-23 (a). The built-in top anode is used to permit (a) lower operating voltage, (b) high accuracy of anode to emitter spacing, and (c) well defined emitter area. This IFED has a well-defined small distance between the tips and the anode with very sharp tips of 200 nm in diameter, created in  $O_2$  plasma by an inductively plasma etching tool (ICP) as shown in Figure 4-23 (b).

The field emission process was confirmed using a needle anode instead of the integrated top poly-C anode. The measurement was taken at the pressure of  $\sim 10^{-6}$  torr, as shown in Figure 4-24. A high voltage supply (Keithley, model 248) applies a voltage between the tip and the sample, and the resulting emission current is measured with a digital multimeter (Keithley, model 2010) as the voltage across a 3.3 k $\Omega$  resistor. The F-N curve shows a non-linearity in the high anode voltage region, which might be due to partial thermionic emission. The field at the onset of emission is in the range of 0.5 - 0.75 MVcm<sup>-1</sup>.



Figure 4-23 (a) The concept diagram and SEM picture of fabricated IFED; (b) diamond needles created in  $O_2$  plasma by ICP.



Figure 4-24 Field emission measurement result with vacuum as insulator.

## 4.6.3 SMM pieozoresistive sensors



Figure 4-25 SEM images of SMM piezoresistive sensors.

This is the first reported application of piezoresistive sensors based on the SMM concept. The SEM images of SMM cantilever-based piezoresistive sensors made of poly-C are shown in Figure 4-25. The fabrication process is shown in Figure 4-26. Undoped poly-C with a resistivity above  $10^9 \Omega \cdot cm$  is used as both an insulating and structural material. Lightly-doped poly-C with a resistivity of 22  $\Omega$  cm was chosen for the piezoresistor with consideration of both the gauge factor (*GF*) and signal to noise ratio (SNR). Highly doped poly-C with a resistivity of  $5 \times 10^{-3}$  $\Omega$  cm is used as interconnects and also the interlayer between metal and poly-C resistor for the purpose of reducing the contact resistance. The SMM device is released by wet etching of silicon in KOH etchant. Displacement sensitivity S was characterized by pressing down the cantilever beam at its tip with a micro-manipulator which was explained in section 3.3. The resistance change was measured for every 1  $\mu$ m tip deflection and is plotted in Figure 4-27. S = 0.47m $\Omega/\Omega/\mu$ m for the long cantilever ( $l = 400 \ \mu$ m) and  $S = 0.66 \ m\Omega/\Omega/\mu$ m for the short cantilever (l= 250  $\mu$ m). Due to the small contact area in this design and a certain level of misalignment, the contact resistance is about 3.7 M $\Omega$  which is comparable with the 5.2 M $\Omega$  of the piezoresistor. Considering the contribution of contact resistance, the actual GF is 20-28. Another piezoresistive sensing design has been developed for RFMEMS applications with larger contact area to reduce the affect from the contact resistance. This will be discussed in Chapter 5.



Figure 4-26 Cross-sections of the fabrication sequence.



Figure 4-27 Relative change of resistance vs. cantilever tip deflection

# **Chapter 5**

# Single-Material MEMS Piezoresistive Resonators for RFMEMS

### 5.1 Introduction

Usually the MEMS resonators in RFMEMS use capacitive transduction to excite and detect the mechanical resonance. However these resonators typically exhibit high impedance at resonance. The high impedance can be even further increased when the resonator is downscaled in order to increase its resonance frequency. Additionally, approaches aimed at lowering the impedance always increase manufacturing complexity. The transduction scheme of using piezoresistive detection and piezoelectric actuation can be implemented using simple processing and allows for resonators with low effective impedance by (a) adjusting the piezoresistor dimensions and (b) increasing the mobility of holes by using intra-grain piezoresistors (which also leads to a piezoresistive gauge factor of over 4000 [97]). Furthermore, this type of transduction is insensitive to geometric scaling and allows for the realization of miniature high frequency MEMS resonators and oscillators without performance reduction. It should also be noted this scheme may also increase the quality factor of the resonator because there is no damping due to the electrostatic force as in the case of capacitive transduction. In this chapter, using piezoresistive detection and piezoelectric actuation, the fabrication and testing of polycrystalline diamond based single-material MEMS (SMM) resonators is reported for the first time.
#### 5.2 Sensor Design



Figure 5-1 Schematic diagram of piezoresistive resonator.

Figure 5-1 shows the fabricated device with two piezoresistors connected in a half-bridge configuration. The two piezoresistors, connected in series due to the presence of metal layer on the front portion of the cantilever, are located near the anchor point. Since the resonant frequency of resonator is proportional to  $\sqrt{k}$ , a high spring constant is desired for potential RFMEMS applications. Considering the fabrication difficulties and the total size of the device, the reasonable values used in this paper are:  $w_{PZR}=20 \ \mu\text{m}$ ,  $l_{PZR}=20 \ \mu\text{m}$ ,  $w=70 \ \mu\text{m}$ , l=430 and 630  $\mu\text{m}$ . In addition, values of  $t = 3 \ \mu\text{m}$  and  $t_{PZR} = 0.6 \ \mu\text{m}$  are also used by taking into account the sensitivity and also the poly-C etching and growth time. The values of device spring constant k,

and the average strain in piezoresistors  $\varepsilon_{PZR}$  are computed and listed in Table 5-1, by assuming *E* is 1000 GPa. The calculation details are given in APPENDIX H.

Finite element analysis (FEA) software (Coventorware) was also used to derive the spring constant of the cantilever and the average strain of the piezoresistive layer when *l* is 630 µm. The stress distribution at F=1 µN is shown in Figure 5-2 (a). Then force *F*, with an increase of 1-µN per step, is applied at the tip of cantilever. Tip deflection versus force is plotted in Figure 5-2 (b). The spring constant *k* is calculated as k=dF/dz where d*F* is the change in force applied (1 µN) and d*z* is the deflection in the cantilever measured at its tip. The results are given in Table II with analytical results also provided for comparison. The average strain in piezoresistors versus force is plotted in Figure 5-2 (c). The simulation results are fairly consistent with analytical results as shown in Table 5-1. The values of *k* and  $\varepsilon_{PZR}$  are not significantly affected by the undercut.

	Analytical	Sim. no undercut	Sim. 50 µm undercut
k N/m	3.054	3.045	3.028
ε <sub>PZR</sub> μstrain	18.262	15.297	15.196

Table 5-1 Analytical, simulation results of k and  $\varepsilon_{PZR}$ 



Figure 5-2 (a) stress contour of piezoresistive resonator, (b) tip deflection and (c) average strain in piezoresistors vs. applied force at the tip.

### 5.3 Sensor Fabrication



Figure 5-3 Fabrication process flow of SMM piezoresistive resonator.

Figure 5-3 shows the main steps of the 3-mask fabrication process. 3-µm-thick undoped poly-C with resistivity >  $10^9 \ \Omega$ ·cm is used as the structural material for the resonator. A 0.6-µm-thick boron-doped poly-C film with a resistivity of 9  $\Omega$ ·cm is used as a piezoresistive layer. 50-nmthick highly doped poly-C with a resistivity of  $5 \times 10^{-3} \ \Omega$ ·cm is used as an interlayer between the piezoresistive layer and Ti/Au (50/400 nm) double layer to reduce the contact resistance. 50 nm Ti, 500 nm Au and 700 nm Al layers were evaporated in sequence and patterned by a lift-off technique using the first mask. Al is used as a masking layer to etch highly doped and piezoresistive poly-C layers by RIE. The etching condition is 20 sccm CF<sub>4</sub> at a pressure of 80 mTorr and a RF power of 300 W. The etching time is monitored by measuring the electrical conductivity of remaining thin film. Then another 1000 nm Al is evaporated and patterned using the second mask. Undoped poly-C is then etched in O<sub>2</sub> plasma in an ICP tool using coil power of 1000 W, platen power of 100 W, pressure of 10 mTorr thus leaving a very smooth etched surface and vertical sidewall. After removing the Al, the third mask is used to wet etch the Ti and Au. Then Ti/Au is used as a mask layer to etch the highly doped poly-C in RIE leaving the piezoresistive poly-C layer exposed. Since the highly doped poly-C is very thin, only a very short etching time is needed. As a result, the Au layer is negligibly damaged which ensures the successful wire bonding later on. Then the cantilever is released by isotropic Si etching in XeF<sub>2</sub> gas. The SEM image of a fabricated piezoresistive resonator is shown in Figure 5-4 (a). Two boron-doped rectangular piezoresistive sensing regions (labeled as PZR) are connected in series by Ti/Au metal layers and form a piezoresistive sensor as shown in Figure 5-4 (b). This sensor is located at the end of resonator. The undercut boundary can also be seen clearly. The highly doped poly-C interlay can be seen clearly in between the Ti/Au and piezoresistive layer to reduce the contact resistance as shown in Figure 5-4 (c).



Figure 5-4 SEM images of fabricated piezoresistive resonator

#### 5.4 Sensor Characterization

5.4.1 Electrical conductivity and contact resistivity characterization

A micro-fabricated Van Der Pauw structure is also used to measure the electrical resistivity of doped poly-C as shown in Figure 5-5. The explanation of this structure is provided in section 3.2.1. Equation (3.2) is used to calculate the resistivity. The resistivity as a function of temperature is also measured by using the temperature control system explained in APPENDIX B. The result is shown in Figure 5-6. It shows a resistivity of 9  $\Omega$ ·cm at room temperature and

decreases to ~1  $\Omega$ ·cm at 200 °C, indicating an activation energy of 0.145 eV and a doping concentration of  $3.8 \cdot 10^{19}$  cm<sup>-3</sup>. Based on this measurement result, the resistance of a piezoresistive sensor can be calculated based on its actual dimensions inspected under SEM. The calculated resistance is 976±156 k $\Omega$  at room temperature. The error is caused by the rough estimation of the length and the thickness of the piezoresistive sensor. The actual measured resistance of the piezoresistive sensor is 1088±26 k $\Omega$  based on two-terminal measurement. The measured result is fairly consistent with the calculated result. The measured resistance decreases to ~120±4 k $\Omega$  at 200 °C.



Figure 5-5 SEM images of fabricated Van Der Pauw structure used to measure the electrical resistivity of doped poly-C.



Figure 5-6 Electrical resistivity of piezoresistive layer versus temperature.



Figure 5-7 (a) SEM image of transmission line model used to characterize the contact resistance (b) The total resistance between two neighboring metal pads as a function of spacing between pads is plotted for four different contact lengths.

The contact resistivity is characterized using a transmission line model which is explained in section 3.2.2. The fabricated structure is shown in Figure 5-7 (a). The total resistance between

two neighboring metal pads as a function of spacing between pads is plotted in Figure 5-7 (b) for four different contact lengths. Equations (3.4) and (3.5) are used to calculate the contact resistivity and transfer length. Contact resistivity  $\rho_c$  is =  $6.8 \times 10^{-3} \Omega \cdot cm^2$  and transfer length  $L_T$ is about 3.3 µm. A contact resistance  $R_c$  of approximately 10 k $\Omega$  is calculated for a single contact. Since there are four contacts for the pieziresistive sensor, the total contact resistance is ~40 k $\Omega$ . Therefore  $R_c$  is approximately 3.7% of the total resistance for the piezoresistive sensor in an actual device. The contact resistance in this design is significantly reduced as compared to the piezoresistive sensor design discussed in section 4.6.3.

#### 5.4.2 Displacement sensitivity and gauge factor characterization



Figure 5-8  $\Delta R/R$  vs. (a) cantilever tip deflection and (b) microstrain in piezoresistive sensor

Displacement sensitivity S was characterized by pressing down the cantilever beam at its tip with a micro-manipulator. The resistance change was measured per 5 µm tip deflection and is plotted in Figure 5-8 (a).  $S = 0.1591 \text{ m}\Omega/\Omega/\mu\text{m}$  for the long cantilever (l = 630 µm) and  $S = 0.3674 \text{ m}\Omega/\Omega/\mu\text{m}$  for the short cantilever (l = 430 µm). *GF* can be derived as shown in Figure 5-8 (b) showing a *GF* of 9.2±0.5 when resistivity is about 9  $\Omega$ ·cm. *GF* is small due to the high doping concentration which is not necessarily disadvantageous in terms of signal-to-noise ratio.

### 5.5 Measurement of Frequency Response

The concept diagram of the measurement setup for achieving the frequency response of the RFMEMS resonators using piezoresistive detection and piezoelectric actuation is shown in Figure 5-9. Labview is used to control the function generator and provide an AC voltage at a desired frequency to the driver circuit. The driver circuit is used to drive the piezoelectric actuator to vibrate the resonator. The details of driver circuit have been given in section 3.4.2. The mounting of the resonator sample on the piezoelectric actuator is also discussed in section 3.4.1. The piezoresistive sensor located at the anchor area of the resonator is used to detect the vibration amplitude of the resonator. The response of the piezoresistive sensor is converted to a small AC voltage by a Wheastone bridge structure. Then the AC voltage is amplified and filtered by the detection circuit. The details of the detection circuit have been demonstrated in section 3.4.3. The output of the detection circuit is read by a digital multimeter and sent to Labview. Labview is programmed to sweep the frequency in a desired frequency range. The details of Labview programming are discussed in APPENDIX G. It is worth mentioning that the output data sent to Labview at every driving frequency were repeated ten times and averaged in Labview. This results in that the thermal noise power is reduced by 10 times. Flicker noise cannot be reduced by averaging the data. However, it is proportional to the reciprocal of frequency and mostly blocked by the high-pass filter on the board. The actual images of circuits are shown in Figure 5-10. The detection circuit with a wire-bonded sample is shown at the bottom left in Figure 5-10.



Figure 5-9 Concept diagram of measurement setup for piezoresistive detection and piezoelectric actuation.

After frequency response data were collected by Labview, Lorentz curving fitting method was used to generate the most accurate fit for the resonant curve of micromechanical resonators. The equation for a Lorentz curve is given by [173]:

$$y = \frac{y_{\text{max}}}{\sqrt{1 + 4Q^2 \left(\frac{f}{f_0} - 1\right)^2}}$$
(5.1)

where  $f_0$  is peak frequency at resonance and Q is the quality factor.  $f_0$  and Q can be extracted from the fit.



Figure 5-10 Piezoresistive detection and piezoelectric actuation boards with wire-bonded sample shown at the bottom left.

The measured frequency response for a resonator with length  $l = 430 \ \mu\text{m}$ , when the power supply to Wheastone bridge is 24 V, is plotted in Figure 5-12 (a) showing a resonant frequency  $f_0$  of 49.2 kHz and a quality factor Q of 197 in air. When the power supply of Wheastone bridge is set to be zero, the output does not change with frequency as shown in Figure 5-12 (b). The comparison of results in Figure 5-12 verifies that the peak observed in Figure 5-12 (a) is the resonant peak of the resonator. The non-zero output in Figure 5-12 (b) is due to the induced current in the circuit caused by the piezoelectric actuator.



Figure 5-11 The measured frequency response in air for resonator with length  $l = 430 \mu m$  when power supplied to Wheastone bridge is (a) 24 V and (b) 0 V.

A more accurate estimation of Q was achieved by taking measurements with a smaller frequency increment per step. The measured frequency response for resonator with length l =630  $\mu$ m is plotted in Figure 5-12 (a) showing  $f_0$  of 25.1 kHz and Q of 72 in air. The measured frequency response for resonator with length  $l = 430 \,\mu\text{m}$  is plotted in Figure 5-12 (c) showing  $f_0$ of 49.7 kHz and Q of 142 in air. The variation of Q for the resonators with same dimensions may be due to the measurement inaccuracy in Figure 5-12 (a). The harmornic analysis feature in Coventorware was used to simulate the frequency response of the resonator. A harmonic displacement with vibration amplitude of 100 nm is applied to the anchor of the resonator in simulation to simulate the vibration of the piezoelectric actuator. The displacement of the resonator tip is plotted as a function of the frequency to achieve the frequency response. Simulation results are shown in Figure 5-12 (b) and (d). Q is also preset to be 100 (damping ratio is 0.005), which is suitable for resonators with designed dimensions in this work [174]. As seen from Figure 5-12, measurement results match the simulation results very well. In simulation, Young's modulus of diamond is set to be 1000 GPa. The matching of the results also indicates that the actual Young's modulus of the poly-C film used in this work is about 1000 GPa, which agrees well with the previous published results in our group [175].



112

20

18

22 24 Frequency (kHz)

(b)

0.0000000

28

26



Figure 5-12 (a) experimental and (b) simulation results of frequency response in air for resonator with beam length  $l = 630 \ \mu m$ ; (c) experimental and (d) simulation results with beam length  $l = 430 \ \mu m$ .

### **Chapter 6**

## **Single-Material MEMS Thin Film Packaging**

### 6.1 Introduction

Poly-C has emerged as a novel material for MEMS packaging. The unique chemical properties of poly-C offer protection against corrosive environments; the mechanical strength offers resistance to package deformation in thin film packaging applications. Poly-C's electrical properties make it a good candidate for use as both conducting and insulating materials. The high thermal conductivity also makes it a good heat sink.

This work reports two designs of a SMM thin film packaging process, each including an encapsulated SMM device. The first design uses boron-doped poly-C as electrical feedthroughs which can be embedded into the undoped, electrically insulating poly-C package. Access ports were opened along the package edge to release the thin film package and the device. Then, additional poly-C growth was used to seal the access ports. The second design is based on the concept of using porous diamond to release the structures from the top of the package, thereby significantly reducing the release and sealing time of the package, without significantly affecting the device. A preliminary test regarding the package has good fluidic hermeticity in an acidic environment. This is the first time that porous diamond created by RIE has been used in all-diamond thin film packaging including poly-C electrical feedthroughs, and an encapsulated SMM device has been reported. This is also the most complicated poly-C structures fabricated successfully.

#### 6.2 **Process Design and Fabrication**

The concept diagram and SEM images of SMM thin film packaging using poly-C is shown in Figure 6-1. It includes poly-C pads, poly-C feedthroughs, poly-C SMM device and poly-C package on an insulating undoped poly-C layer. The fabrication process is given by Figure 6-2. The fabrication process started with a thick, undoped insulating poly-C layer grown on a Si substrate. 0.5-µm thick feedthroughs and contact pads were deposited and patterned using highly boron-doped poly-C (Figure 6-2 (a)). Then SMM devices made of boron-doped poly-C were fabricated using two masks (Figure 6-2 (b, c)). The sacrificial PECVD SiO<sub>2</sub> for poly-C device is  $\sim$ 1 µm. The fluidic access ports for the removal of sacrificial layer are also created after these two steps. Next, a second PECVD SiO<sub>2</sub> layer was deposited and patterned to create the anchor region for the packaging cap (Figure 6-2 (d)). A 4-µm-thick undoped poly-C layer was then grown and patterned to form the thin film packaging cap (Figure 6-2 (e)). The nearly zero stress in poly-C film and the good adhesion between poly-C and SiO<sub>2</sub> guarantee the success of fabricating such multilayer structures. The sample was released in concentrated HF and sealed in a MPCVD system for an additional poly-C growth (Figure 6-2 (f) and (g)) with typical sealing pressures of 40 Torr. Another mask is used to remove the undoped poly-C layer in the area where the highly-doped-poly-C contacts pads are (Figure 6-2 (h)), which has not been accomplished yet. Since the released structures consist of poly-C only, the thermal mismatch stress is minimized. Furthermore, the feedthrough is also made of poly-C leading to perfect sealing around it.



Figure 6-1 SMM package concept diagram and the SEM picture of the structures

The thickness of the second  $SiO_2$  sacrificial layer needs to be selected carefully to make sure the package does not touch the underlying device due to the deflection of the package under the pressure difference after the package is sealed. The deflection of the poly-C package can be estimated using the pressure-deflection relationship given as:

$$p = \frac{3.41\sigma th}{a^2} + \frac{2.45Eth^3}{a^4}$$
(6.1)

where p is the pressure difference applied on the packaging; t, 2a and h are the thickness, width and deflection of the packaging respectively; E is the Young's Modulus of the packaging, which is assumed to be 1000 GPa. Neglecting the residual stress (i.e.  $\sigma = 0$ ) and assuming the pressure inside the packaging is 0, it is estimated that a 4-µm-thick poly-C packaging that is 650 µm on a side would deflect around 4.8  $\mu$ m. Therefore 6- $\mu$ m-thick PECVD SiO<sub>2</sub> was used for the second sacrificial layer between the SMM device and package.





Figure 6-2 General fabrication process flow of SMM thin film package

The sealing time for the current design is reduced by a factor of 12 by reducing the height of the fluidic access ports compared to the time in the previous work [158] [159]. The package sealing is also enhanced because sealing occurs between two poly-C layers instead of between silicon and a poly-C layer used in [158] [159].



#### 6.3 Release and Sealing Using Porous Diamond

Figure 6-3 (a) Creation of porous diamond by RIE of diamond (b) Release the structure by using HF to penetrate the porous diamond

The application of porous diamond techniques to the SMM thin film package in the second design can further reduce the release time as well as the sealing time due to the small pore size. As a result, the encapsulated SMM device is negligibly affected during the release and sealing process. It is known that poly-C growth rate is usually very low under low pressure conditions.

However, the application of porous diamond can make sealing possible to use ECR diamond growth at 10 mTorr [176].

The fabrication steps with the application of porous diamond are shown in Figure 6-3. Since most of the steps are the same as those in Figure 6-2, Figure 6-3 only shows the fabrication steps of porous diamond creation after the patterning of poly-C thin film packaging and the release of the structure through porous diamond. Porous diamond is created in the region of the release holes. It was created with  $O_2$  etching in RIE as illustrated in 4.5.1.



Figure 6-4 Poly-C package having porous diamond as release holes before being soaked in HF



Figure 6-5 Poly-C package having porous diamond as release holes after being soaked in HF for 10 minutes (top) and 30 minutes (bottom).

Figure 6-4 shows a poly-C package with release holes before HF etching while Figure 6-5 shows the SEM images after soaking in HF for 10 and 30 minutes. Some  $SiO_2$  remained after 10 minutes in HF indicating that HF can penetrate through the porous diamond to etch the underlying  $SiO_2$ . In contrast, the continuous poly-C layer can protect the etching of underlying  $SiO_2$ . The results also indicate the etch rate of  $SiO_2$  through the porous diamond is lower than the normal  $SiO_2$  etch rate in HF. The time for the package sealing is reduced approximately by at

least a factor of 10 using porous diamond. As seen in Figure 6-6, the holes in porous diamond are closed by additional poly-C growth.



Figure 6-6 Porous diamond grows into continuous poly-C film in MPCVD

### 6.4 Evaluation of Poly-C Hermeticity

An experiment was designed to test the hermeticity of a poly-C package in an acidic environment, as shown in Figure 6-7. The poly-C cap is fabricated, leaving the 6- $\mu$ m SiO<sub>2</sub> inside the package. The fabricated chip was then soaked in a concentrated hydrofluoric acid (HF) solution for up to two days at room temperature. Samples were then broken intentionally to see if the HF had penetrated the poly-C layer and etched the SiO<sub>2</sub>. Test chips with different thickness of poly-C package were prepared by breaking the chips at various steps of the testing, showing that a poly-C thin film with a thickness of 2.6  $\mu$ m (corresponds to 17 hours growth time) cannot prevent HF from penetrating it, as shown in Figure 6-7 (a). Thus, the SiO<sub>2</sub> sealed inside the poly-C package was etched away. However, the poly-C layer with a thickness of 2.9  $\mu$ m (corresponds to 19 hours growth time) shows good fluidic hermeticity in an acidic environment, as shown by Figure 6-7 (b). It also indicates that the 4-  $\mu$ m-thick poly-C package that is generally used in this study can provide excellent hemedicity.



Figure 6-7 Sample chips with poly-C layer of (a) 2.62 µm (b) 2.92 µm after 2 days soak in HF.

# **Chapter 7** System Integration



Figure 7-1 Design of system integration



Figure 7-2 Frequency response in air for a resonator with beam length of 430  $\mu$ m measured using the system integration setup in Figure 7-1.

MEMS system integration is becoming very complex because of the increased number of components and requirements of system miniaturization. A schematic diagram for wireless MEMS system integration is proposed in Figure 7-1. In this work, system integration is built to achieve the frequency response of piezoresistive RFMEMS resonators. A Texas instruments MSP430G series microcontroller is used to control the piezoelectric actuator and process the piezoresistive sensing signal. For the actuation, a timer module of the microcontroller is programmed to generate 50% duty cycle PWM (pulse-width modulation) signals on one of its outputs. The PWM signal is then amplified by analog interface (discussed in section 3.4) to drive the piezoelectric actuator. The piezoresistive sensing signal is amplified, filtered and rectified by analog interface, and then read by the 10-bit Analog-to-Digital converter module integrated in the microcontroller. Digital data is saved in its internal flash memory. In order to save more data, a RAM module can be added to the system to increase the memory size. Instead of using a wireless interface for communication with computers, C code in IAR Workbench, a developer environment for the microcontroller, is used to save the digital data in the microcontroller's flash memory as a text file. Then a Python script is written to read the file and convert the data in the text file into analog voltages. With the help of the Python script, a table of voltages versus frequency can be created to achieve the frequency response of the system. The C code and Python code are given in APPENDIX I. The frequency response in air of a resonator with beam length of 430  $\mu$ m is plotted in Figure 7-2. In order to achieve the resonant frequency  $f_0$  and quality factor Q, Lorentz curving fitting method discussed in section 5.5 was used to generate the most accurate fit for the resonant curve of micromechanical resonators. It shows  $f_0$  of 48.4 kHz with Q of 122. This result is fairly consistent with the result reported in Chapter 5, indicating that the system integration works as well as the previous measurement setup but with a more compact setup.

### **Chapter 8**

# **CONCLUSIONS AND FUTURE RESEARCH**

#### 8.1 Summary of Contributions

Fundamental research on SMM technology

Three crucial issues are addressed in this work for fabrication of SMM structures and devices. They are reported and discussed for the first time. First, highly insulating poly-C thin film (<1  $\mu$ m) with electrical resistivity higher than 10<sup>10</sup>  $\Omega$ ·cm is achieved in a reactor which is also used to dope poly-C. Second, conformal coating is achieved, which ensures poly-C film is continuous everywhere. Third, the dry etching of poly-C is optimized to achieve smooth etched surfaces as well as smooth and vertical side walls after the removal of poly-C. MEMS structures used to characterize poly-C electrical resistivity, contact resistivity and residual stress are also discussed. They can be integrated with designer's own chip design very conveniently without taking too much space. This is very helpful for future SMM designers.

#### Enabling technology development for SMM structures and devices

Surface micromachining technology was developed for multilayer SMM structures and devices. For the first time, SMM Piezoresistive RFMEMS resonators using highly doped poly-C as interconnects and electrodes, semiconducting poly-C as piezoresistive layer, and undoped poly-C as insulating layer are fabricated and tested. Two very important characteristics of micromechanical resonators, resonant frequency and quality factor Q, are extracted from the plot of measured frequency response. The determination of these two parameters can be used for the

mechanical filters in RFMEMS or the chemical sensor in BioMEMS in the future. Thin film packaging with SMM devices encapsulated is also reported for the first time and presented as the most complicated SMM structure. It shows excellent resistance to acidic corrosion environment.

#### 8.2 Future Research

- Develop the technology to smooth poly-C thin film. Film with low surface roughness can ease the fabrication process. The performance of poly-C nano-devices can be improved with low surface roughness. For example, the reduction of surface roughness can potentially increase the quality factor of poly-C nano-resonators in high vacuum. Smooth poly-C film can be achieved either by polishing the as-grown film or by using UNCD film for SMM instead of MCD or NCD.
- Explore highly sensitive SMM piezoresistive resonator using intra-grain piezoresistive sensors. The gauge factor of doped poly-C is 4000 when the sensor is within a single grain [97]. The intra-grain sensor can be patterned using e-beam lithography. This sensor can be applied to chemical and gas analysis.
- Explore the application of SMM in biosensors. The advantages of diamond in the field of biosensors are enormous: chemical stability, biocompatibility, easy surface modification, largest electrochemical potential window, etc.

# **APPENDICES**

# **APPENDIX** A



Figure A-1 Simplified Schematic of valence bands split-off model (a) no stress, (b) under tensile stress, and (c) under compressive stress.

The piezoresistive property of semiconductor such as doped diamond under uniaxial stress can be explained by the valence-bands split-off model, as shown in Figure A-1. The strain will change the concentration of heavy and light holes, so the strain can change the conductivity of the *p*-type semiconductor. The *GF* for single crystalline doped diamond was derived [177]

$$GF = -\frac{1}{\varepsilon} \frac{A + E_e C}{kT + A + E_e C} \approx \frac{1}{\varepsilon} \frac{A + E_e C}{kT}$$

$$C = \left( m_l^{1/2} - m_h^{1/2} \right) / \left( m_l^{1/2} + m_h^{1/2} \right) \text{ tensile strain}$$

$$C = \left( m_h^{1/2} - m_l^{1/2} \right) / \left( m_l^{1/2} + m_h^{1/2} \right) \text{ compressive strain}$$
(A.1)

where A and  $E_e$  are labeled in Figure A-1.  $m_h$  and  $m_l$  are the effective mass of heavy- and light band hole. The suffix 'h' and 'l' represent heavy and light holes, respectively. C represents the relative difference between the effective mass of heavy and light holes, in other words, they also represent the relative difference between the effective mobility of heavy and light holes. For diamond, C=-0.89 under tensile strain and C=0.89 under compressive strain. k (J/K) is Boltzmann constant, T (K) is the absolute temperature. The great difference of the effective mass between the heavy and light holes in diamond is one of the main factors causing the excellent piezoresistive effect of diamond [177] as indicated by equation (A.1). Since GF is positive under tensile, it indicates the resistivity increase under tensile stress. Likewise, the resistivity decreases under compressive stress due to the negative value of GF.

In general, the polycrystalline materials consist of crystalline grains interconnected by grain boundaries. Grains are considered as single crystals with a diamond lattice. The grain boundaries (GBs) are composed of disordered atoms and contain a large number of defects and dangling bonds which act as trapping states and/or segregation sites [178]. Trapping of holes creates a potential barrier at the boundary and a depletion region into the grains. The film can be modeled as grains of length  $L_g$  with boundary thickness  $\delta$  and depletion width  $w_g$  [179]. The fractional change in resistivity per unit strain of the polycrystalline materials can be represented as [180]:
$$\frac{\Delta\rho}{\rho}\frac{1}{\varepsilon} = \frac{L_g - (2w_d + \delta)}{L_g}\frac{\Delta\rho_g}{\rho_g}\frac{1}{\varepsilon} + \frac{(2w_d + \delta)}{L_g}\frac{\Delta\rho_b}{\rho_b}\frac{1}{\varepsilon}$$
(A.2)

where  $\rho_g$  and  $\rho_b$  are the resistivity of grain and grain boundary. Normally, it is believed that the piezoresistive effect of the GBs is much lower than the grain crystal. The random orientations of the GBs also lower the overall piezoresistive effect of grain. This is consistent with the reported low *GF* of polycrystalline materials than that of their single crystal counterparts. The effect of GBs on the gauge factor was studied in [98] and presented as below assuming there is no *GF* for GBs:

$$GF \approx \left(\frac{1}{1+\alpha}\right) GF_g$$
 (A.3)

where is  $\alpha$  the ratio of GBs resistance to grains resistance along the conduction path when film has no strain and  $GF_g$  is the gauge factor of single-crystalline material. This explains why polycrystalline film with high resistivity results in larger *GF*.

A more explicit equation of GF for polycrystalline material is given in [177]

$$GF = (M - F)GF_g + F(D - C)\frac{1}{kT}\frac{dE_e}{d\varepsilon}$$
(A.4)

*D* is also constant relating to the difference of the effective mass of holes like *C*. *D* equals to -1 and 1 under tensile and compressive stress for diamond film, respectively. *M* and *F* are two constants related to the grain-boundary scattering. When the stain is very small (~  $10^{-6}$ ), *M* is equal to 1 and 0 < F < 1. The last term on the right side of equation (A.4) is negative. Therefore, *GF* is smaller than *GF<sub>g</sub>*, indicating the *GF* of polycrystalline material is smaller than that of single crystalline material. Equation (A.4) also means if there is excellent piezoresistive effect in the crystalline materials, the more significant piezoresistive effect will exist in the corresponding polycrystalline material. As a result, poly-C is a good candidate for the application of piezoresistive sensor. The reported *GF*s of poly-C in the past are given in Table A-1. They range from 5-1000 at room temperature when poly-C resistivity is in the range of  $0.01-300 \Omega \cdot cm$ .

GF	ρ (Ω·cm)	Boron Concentration (cm <sup>-3</sup> )	Substrate	Deposition	Doping	Ref.
6		1.2x10 <sup>16</sup>	Diamond/SiO <sub>2</sub>	HFCVD		[87]
5.4	4		undoped poly- C	MPCVD	BCl <sub>3</sub>	[181]
116		2.5x10 <sup>18</sup>	Si	DC plasma CVD	B <sub>2</sub> O <sub>3</sub> powder	[91]
6-25	5-30		SiO <sub>2</sub>	HFCVD	boron powder	[182]
8	0.2					
10	2.2		Si	MPCVD	Boron compound	[106]
67	225		51		solid wafer	[100]
100	300					
1000	300		undoped poly- C	MPCVD	B <sub>2</sub> H <sub>6</sub>	[93] [94]

Table A-1 Gauge factors of poly-C reported by different groups

Table A-1 (cont'd)

7-9	0.1						
70-75	100			MPCVD		50.53	
0.1	1000		S1		B(CH <sub>3</sub> ) <sub>3</sub> in ethanol	[95]	
30-35	10 <sup>6</sup>		-				
690			Si	MPCVD	B(CH <sub>3</sub> ) <sub>3</sub>	[96]	
283*	0.27		undoped	MPCVD	boron powder	[97]	
4000**	0.1		poly-C		r r		
6.7/8.9*		~10 <sup>20</sup>	undoped	MPCVD		[98]	
25**			poly-C			[, ]]	
33	3.15		SiO2	HFCVD	H <sub>3</sub> BO <sub>3</sub> in methanol	[100]	
50	2.65				and acetone	[100]	
40	28		SiO <sub>2</sub>	MPCVD	B(CH <sub>3</sub> ) <sub>3</sub>	[180]	
8±0.5	0.01		SiO <sub>2</sub>	MPCVD	B(CH <sub>3</sub> ) <sub>3</sub>	[102]	

\*intergrain piezoresistor

\*intragrain piezoresistor

## **APPENDIX B**



(b)

Figure B-1 (a) Schematic diagram of temperature control system and (b) sample mounting on the heater.

A temperature control system was built to measure the resistance change of doped poly-C resistor as a function of temperature as shown in Figure B-1 (a). It can control the temperature from room temperature to 200 °C with a precision of  $\pm 1$  °C. A rectangular Kapton insulated flexible heater (rate temperature range -200 - 200 °C) with a power of 10 Watts per inch<sup>2</sup> was used. Two DC power supplies (Keithley E3612) with maximum output power of 120 W in total are used to power the electric heater. By reading the heater temperature via a K-type thermal couple, the temperature controller (CN7800) controls the mechanical relay to open and close. When the heater temperature exceeds the setting value, the mechanical relay will be open. It results in an open circuit and the temperature of the heater will decrease. A solid-state DC relay is used to isolate the mechanical relay from the heating loop (the left side of the schematic diagram) because the maximum current that can flow through the mechanical relay is much lower than the current flowing though the electric heater.

The sample mounting method is shown in Figure B-1 (b). The electrical heater was put on the wafer chuck of probe station with a quartz plate in between. Quartz plate is used for thermal isolation due to its low thermal conductivity (1.3 W/( $m \cdot k$ )). Electric heater warps due to the thermal expansion when temperature increases. As a result, a thin metal sheet was placed in between the sample and the electric heater. Two clips are used to fix them. Two probes were used to pick up the resistance which is read by a digital multimeter (Keithley 2010).

### **APPENDIX C**



Figure C-1 Schematic diagram of seeding process (a) Si has hydrophobic surface after removing the native oxide by diluted hydrofluoric acid (HF). Separate water drops can be observed after taking the wafer out from deionized (DI) water. (b) Super-hydrophilic surface is achieved after O<sub>2</sub> plasma treatment. A thin water layer forms on the surface after taking the wafer out from DI water. (c) Several drops of DPLW are put on silicon wafer immediately after O<sub>2</sub> plasma treatment. (d) Wafer is spun. Uniform and high seeding density is achieved across the wafer.

It is difficult to create highly insulating poly-C due to the reduction of layer thickness--a low nucleation density or small variation in seeding density could create a non-covered area in poly-

C thin film, such as pin-holes, leading to current leakage through the non-covered area. Therefore, in addition to the use of  $O_2$  during growth, another study is focused on achieving pin-hole free diamond film having a layer thickness less than 1  $\mu$ m.



Figure C-2 For the case of using 0.5% DPLW, (a) seeding density of  $10^9$  cm<sup>-2</sup> was achieved and (b) 8 hours are needed to form continuous poly-C thin film. For the case of using 5% DPLW, (c) seeding density above  $10^{11}$  cm<sup>-2</sup> was achieved and (d) less than 20 minutes are needed to form continuous film.

Undoped poly-C layers are always grown on top of a Si substrate, and are used as the bottom (insulating) layer for SMM devices in this research work. Si is chosen as the base layer because the adhesion force between poly-C and Si is generally stronger than that between poly-C and SiO<sub>2</sub> or poly-C and Si<sub>3</sub>N<sub>4</sub> due to the formation of SiC at the interface between poly-C and Si. Therefore, the study on nucleation density is only focused on undoped poly-C growth on Si substrates.

The usual seeding procedure is shown in Figure C-1. An  $O_2$  plasma treatment in a plasma asher at a power of 250 W and a pressure of 500 mTorr is used to convert the hydrophobic nature of the Si surface to super-hydrophilic before DPLW is spun. Such a treatment can allow a more uniform seeding density compared to the wet chemical treatment using ammonium hydroxide (NH<sub>4</sub>OH) and peroxide (H<sub>2</sub>O<sub>2</sub>) [183] reported previously by our group. Furthermore, O<sub>2</sub> plasma treatment is more convenient and controllable, and it generally creates a cleaner surface by removing organic residue.

Seeding density depends on the diamond powder concentration more than the spin rate. Seeding density was studied for diamond powder concentrations varying from 0.5%-5% when the spin rate is 2000 rpm. The seeding density was calculated using a scanning electron microscope (SEM, Hitachi S-4700II). The seeding density, defined by the number of seeding particles per cm<sup>2</sup>, was calculated by a graphical counting method. Figure C-2 (a) shows the seeding distribution on silicon with 0.5% DPLW (diamond powder concentration is 0.5%) with a seeding density of  $10^9$  cm<sup>-2</sup>. 8 hours growth time is needed to form a continuous thin film in this case as indicated by Figure C-2 (b). Figure C-2 (c) shows an ultrahigh seeding density of approximately  $10^{11}$  cm<sup>-2</sup>, which was achieved using 5% DPLW. This result is two orders of magnitude higher than that produced with 0.5% DPLW. Only 20 minutes growth time is needed to form a continuous thin film in this case as indicated by Figure C-2 (d).

Other advantages of ultrahigh seeding density are (i) it ensures continuous poly-C step coverage and (ii) it protects the underlying layer from being damaged by H<sub>2</sub> plasma in a MPCVD system prior to the formation of pin-hole free poly-C film.

#### **APPENDIX D**

A convenient way that is also used to characterize the electrical resistivity of undoped poly-C films grown with or without  $O_2$  in the growth environment is shown in Figure D-1. Undoped poly-C is grown on highly conductive n-type Si chip having areas of 1 cm x 1 cm. A Cr/Au metal layer was deposited on top of undoped poly-C as a top electrode. Al was deposited on the bottom of the Si substrate forming an ohmic contact. The chip was then glued to a metal plate using silver conductive epoxy. An *I-V* plot can be achieved using the voltage reading in power supply and the current reading in ampere meter.



Figure D-1 Measurement setup to characterize the electrical resistivity of undoped poly-C films.

# **APPENDIX E**

Fabrication process flow of one released poly-C layer with  $\mathrm{SiO}_2$  as sacrificial layer

I.	Prepare Substrates
1)	Material: 1.5"x1.5" square n-type Si wafers coated with SiO <sub>2</sub> on top; vendor: WRS materials
2)	Clean: Acetone for 20 seconds, IPA for 20 seconds and DI water for 1 minute
3)	Blow dry

#### II. Seeding

	~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~
1)	Diluted hydrofluoric acid (HF:DI 1:100) etch for 5 seconds
	Comment: to remove the native oxide
2)	Blow dry
3)	O <sub>2</sub> plasma etching for 3 minutes:
	Plasma asher: 250 W and 250 mTorr
	Comment: oxidize Si surface
4)	Spin 5% DPLW at 2000 rpm for 40 seconds.
	Comment:
	a) If there is a high step height, spinning at 500 rpm for 5 seconds is necessary to form continuous poly-C film everywhere.
	b) Ultrasonic bath must be used before spinning
	c) Spinning must be done immediately after O <sub>2</sub> plasma etching: otherwise the hydrophilic

c) Spinning must be done immediately after O<sub>2</sub> plasma etching; otherwise the hydrophilic property of oxidized Si surface would change.

#### III. Poly-C growth

1)	Pump down MPCVD system for 30 minutes
2)	Grow poly-C at ~750 °C
	Power 2.3 kW, pressure 40 Torr.
	Insulating poly-C: $O_2$ : $CH_4$ : $H_2 = 0.5$ : 2 : 200; Highly conducting poly-C: TMB 1 ppm
	Semiconducting poly-C: TMB 0.01-0.1 ppm.
3)	Vent system and load sample after system has been cooled down for 30 minutes

IV. Photolithography
1) Clean: Acetone for 20 seconds, IPA for 20 seconds and DI water for 1 minute
2) Blow dry
3) $O_2$ plasma etching for 1 minute
Plasma asher: 100 W and 250 mTorr
Comment: to remove the surface conducting layer of poly-C

4)	Prebake on hotplate to	) dehvdrate the sam	ple for 5 minutes at	:115 °	C
т <i>)</i>	I TOURC ON NOTPLACE IC	J delly diate the same	ipic for 5 minutes at	115	

5) Spin HMDS at 4000 rpm for 15 seconds

6) Spin AZ9260 at 4000 rpm for 30 seconds

- 7) Soft bake on hotplate for 2.5 minutes at 115 °C Comment: kinwipe must be used underneath the wafer
- 8) Rehydration for 15 minutes Comment: to develop rate will be lower than normal if rehydration time is not sufficient.
  9) Exposure Tool: MA/BA6; hard contact, for 35 seconds
  10) Develop

Developer AZ400 : DI = 1 : 4 for 1.5-2 minutes depending on the rehydration time.

11)Blow dry

V.	Metal Lift-off					
1)	Descum in O <sub>2</sub> plasma					
	Plasma asher: 120W 250 mT for 100 seconds					
	Comment: to remove the photoresist AZ9260 residuals.					
2)	Al deposition					
	Tool: Enerjet Evaporator					
	Comment: Al thickness depends on the thickness of poly-C.					
	The measured selectivity of Al and poly-C is above 100 in O <sub>2</sub> plasma and 11 in CF <sub>4</sub> plasma					
	in Plasmatherm RIE tool.					
	Selectivity of Al and poly-C is about 10 in O <sub>2</sub> plasma in LAM9400 ICP tool.					
3)	Lift-off					
	a) Leave in acetone facing down for more than 3 hours					
	b) Put some acetone in acetone lift-off beaker. Put the wafer facing up.					
	Provide ultrasonic for 5 minutes; Drain the acetone.					
	c) Put some IPA in acetone lift-off beaker.					
	Provide ultrasonic for 5 minutes; Drain the IPA.					
	d) Squirt DI using the shower. Drain the DI water; repeat three times.					

- e) Rinse 2 minutes in DI water
- f) Blow dry

VL	Dry etching of poly-C
1)	Tool: Plasmatherm 790 or LAM9400
	Comment: for poly-C film with thickness less than 2 $\mu$ m, O <sub>2</sub> plasma can be used in both tools to achieve smooth etched surface after poly-C removal. For poly-C film with thickness more than 2 $\mu$ m, three-step etching must be used in
	Plasmatherm 790. However, O <sub>2</sub> plasma can still be used in LAM9400.
	For efficient poly-C etching, three-step etching scheme (a sequential etching in CF <sub>4</sub> , O <sub>2</sub> and

	H <sub>2</sub> plasma) should always be used if the requirement for aspect ratio is not critical
2)	Al Etching
	In Al etching type A.
	Comment: Due to the Al <sub>2</sub> O <sub>3</sub> on top, the etch rate of Al is not a constant. Putting the wafer in
	the etchant more than 10 minutes usually can entirely remove the Al.
3)	Rinse in DI for more than 10 minutes
	Comment: Rinsing time must be longer than 10 minutes to ensure no residual remained.
4)	Blow dry

#### VII. Release

1) Immerse the wafer in BHF until all devices are released.

Comment: vertical etch rate of thermal  $SiO_2$  is about 10 minutes per  $\mu$ m; vertical etch rate of PECVD  $SiO_2$  is about 3 minutes per  $\mu$ m. Lateral etch rate is 5-6 times faster than the vertical etch rate. Therefore, the release time can be estimated.

2) Drain BHF and Put DI water; Leave wafer in DI water for 5 minutes; Repeat 3 times

3) Drain DI water and put methanol; leave wafer in methanol for 10 minutes;

4) Put wafer in basket and put basket on hotplate at 115 °C. Wait till methanol is totally evaporated.

Comment: methanol is used because its surface tension force is much less than DI water.

## **APPENDIX F**



Figure F-1 Labview program used to measure the change of the resistance of piezoresistive sensor as function of cantilever tip deflection.

The interface is shown in Figure F-1. It can fulfill the following functions:

- 1. Compute the mean value and the standard deviation of a set number of read points from digital multimeter, and then plot these points on the left graph.
- 2. After clicking on "write to file" button, the mean value of resistance will be saved to the file whose save path is defined by users. The point will be plotted on the right graph.
- Every time after the tip moves by a certain of distance, "write to file" should be clicked. After a number of clicks, the resistance change versus tip deflection is plotted. All data has been saved.
- 4. Data can be cleared and the software is ready for next data collection

### **APPENDIX G**



Figure G-1 Labview program used to measure frequency response

The interface of the program is shown in Figure G-1. The block diagram of the programming is shown in Figure G-2.



Figure G-2 The block diagram of the programming

The program can set the low and high frequency range, voltage amplitude, and the number of frequency steps. It can show the current frequency and voltage reading from the digital multimeter. The read data from digital multimeter at every frequency point can be averaged in order to reduce the thermal noise.

#### **APPENDIX H**

The top view and cross-sectional view of a piezoresistive resonator are shown in Figure H-1 with two short segments connected to the anchor. The cantilever has a spring constant *k*. f(x,y), which is a geometric factor, represents the relative change of the spring constant due to the two short segments as shown in equation (H.1), where  $k_0$  is the spring constant of a resonator with length *l*, width *w* and thickness  $t + t_{PZR}$  without the connecting segaments. *E* is the Young's modulus of poly-C.  $x = l_{PZR}/l$  (0<x<1) and  $y = 2w_{PZR}/w$  (0<y<1).

$$k/k_0 = f(x, y) \tag{H.1}$$

$$k_0 = Ew(t + t_{PZR})^3 / (4l^3)$$
(H.2)

$$f(x,y) = 1/\left[1 + \left(x^3 - 3x^2 + 3x\right)\left(1/y - 1\right)\right]$$
(H.3)

The displacement sensitivity S is defined as the normalized change in the resistance with the tip deflection of the cantilever  $\delta$  equal to 1 µm, which is given in equation (H.4) and (H.5).

$$S = \frac{\Delta R/R}{\delta} = GF \cdot \varepsilon_{PZR} = GF \cdot \left[\frac{3t}{4l^2} \cdot g(x, y)\right]$$
(H.4)

$$g(x,y) = \left[ \left( 2 - x \right) / y \right] \cdot f(x,y) \tag{H.5}$$

 $\varepsilon_{PZR}$  is the average strain in piezoresistors at  $\delta = 1 \ \mu m$ . It should be noted that highly doped poly-C and Ti/Au metal layers are not considered in the above and below analytical results.



Figure H-1 Top and cross-sectional view of piezoresistive resonator

Since the resonant frequency of resonator is proportional to  $\sqrt{k}$ , a high spring constant is desired for potential RFMEMS applications. f and g have to be maximized to achieve a high displacement sensitivity and spring constant. The plots of f(x,y) and g(x,y) as a function of x and y are shown in Figure H-2 (a) and (b), respectively. It should be noted that in Figure H-2 (b), the minimum y is set to be 0.1, because g(x,y) becomes infinite when y approaches zero. Considering the fabrication difficulties and the total size of the device, the reasonable values used in this paper are:  $w_{PZR}$ =20 µm,  $l_{PZR}$ =20 µm, w=70 µm, l= 430 and 630 µm. In addition, values of t = 3 µm and  $t_{PZR}$  = 0.6 µm are also used by taking into account of the sensitivity and also the poly-C etching and growth time. The values of x, y, f(x,y), g(x,y), k, and  $\varepsilon_{PZR}$  are computed and listed in Table H-1 by assuming E is 1000 GPa.



Figure H-2 (a) f(x,y) and (b) g(x,y) as a function of x and y

Table H-1 Values of x, y, f(x,y), g(x,y), k and  $\varepsilon_{PZR}$ .

<i>l</i> μm	x	у	f	g	<i>k</i> N/m	$\varepsilon_{PZR}$ µstrain
430	0.046	0.57	0.91	10.3	9.337	37.823
630	0.031	0.57	0.94	3.3	3.054	18.262

### **APPENDIX I**

#### //C Code

#include "msp430g2231.h"

#include <stdio.h>

#include <math.h>

void WRT2MEM(unsigned int value, int addr); void PWM(int freq); //PWM Signal Generator Function.

int main( void )

{

WDTCTL = WDTPW + WDTHOLD;

BCSCTL1 = CALBC1\_16MHZ;

DCOCTL = CALDCO\_16MHZ;

WDTCTL = WDTPW + WDTHOLD;

P1DIR |= 0x04;

P1SEL |= 0x04;

PWM(50);

\_BIS\_SR(CPUOFF);

\_\_delay\_cycles(9000000);

 $ADC10CTL0 = SREF_0 + ADC10SHT_0 + ADC10ON + ADC10IE;$   $ADC10CTL1 = INCH_7;$   $ADC10AE0 \models 0x80;$ int addr = 0xF900;

```
for(int i=1; i<=500; i++){
```

```
ADC10CTL0 |= ENC + ADC10SC;
```

```
__bis_SR_register(CPUOFF + GIE);
```

```
unsigned int temp = ADC10MEM;
```

WRT2MEM(temp, addr);

addr +=2;

```
__delay_cycles(8);
```

```
}
```

return 0;

```
}
```

void WRT2MEM(unsigned int value, int addr){

FCTL2 = FWKEY + FSSEL0 + FN5 + FN4 + FN3 + FN2 + FN1;

```
unsigned int *MEM_PTR;
```

MEM\_PTR = (unsigned int \*)addr;

```
FCTL3 = FWKEY;
```

FCTL1 = FWKEY + BLKWRT + WRT;

\*MEM\_PTR = value; FCTL1 = FWKEY; FCTL3 = FWKEY + LOCK; }

```
void PWM(int freq){
```

```
float step_dif = (1.0/(10.0*1000.0)) - (1.0/(freq*1000.0));
```

```
float cycle = roundf(step_dif/0.00000004665);
```

unsigned long int icycle = (int)cycle;

unsigned long int count0 = 2144 - icycle;

```
unsigned long int count1 = count0/2;
```

```
CCR0 = count0;
```

```
CCTL1 = OUTMOD_7;
```

```
CCR1 = count1;
```

```
TACTL = TASSEL_2 + ID_0 + MC_1;
```

```
}
```

```
#pragma vector=ADC10_VECTOR
```

```
__interrupt void ADC10_ISR(void){
```

```
__bic_SR_register_on_exit(CPUOFF);
```

}

#### //Python code

from \_\_future\_\_ import division

```
def Convert2Volt(value,Vhi,Vlo):
```

```
result = float((int(value,16)/int(1023))*(Vhi-Vlo))
```

return (result)

```
fname = raw_input("File Name (*.txt) :")
```

Vhi = float(raw\_input("High Converter Voltage:"))

```
Vlo = float(raw_input("Low Converter Voltage:"))
```

```
text = open(fname,'r')
```

```
start_addr = text.readline()
```

datalst=[]

```
null='ff'
```

data = 0

```
pickvolt = 0
```

print "\nStart Address:",start\_addr

while True:

```
line = text.readline()
```

```
line = line.split(" ")
```

```
count = 8
mem = 0
if line[mem] == 'q':
  break
while \operatorname{count} > 0:
  count = count-1
  data += 1
  if line[mem+1] == 'ff':
     break
  if mem != 14:
     value = line[mem+1] + line[mem]
     #print value
     voltage = Convert2Volt(value,Vhi,Vlo)
     datalst.append(voltage)
     print 'Read Voltage %d : %.4f V'%(data,voltage)
     mem = mem + 2
  else:
     linelast = line[mem+1].split('\n')
     value = linelast[0]+line[mem]
     voltage = Convert2Volt(value,Vhi,Vlo)
     datalst.append(voltage)
```

```
print 'Read Voltage %d : %.4f V'%(data,voltage)
```

for i in datalst:

if i > pickvolt:

pickvolt =i

else:

pass

print '\nPeak voltage: %.4fV\n'%pickvolt

print '\*\*\*END'

# **BIBLIOGRAPHY**

#### **BIBLIOGRAPHY**

- [1] K.J. Gabriel, F. Behi, R. Mahadevan and M. Mehregany, "In situ friction and wear measurements in integrated polysilicon mechanisms," *Sensors and Actuators A: Physical*, vol. 21, pp. 184-188, 1990.
- [2] A.R. Krauss, O. Auciello, D.M. Gruen, A. Jayatissa, A.V. Sumant, J. Tucek, D.C. Mancini, N. Moldovan, A. Erdemir, D. Ersoy, M.N. Gardos, H.G. Busmann, E.M. Meyer and M.Q. Ding, "Ultrananocrystalline diamond thin films for MEMS and moving mechanical assembly devices," *Diamond and Related Materials*, vol. 10, pp. 1952-1961, 2001.
- [3] J.K. Luo, Y.Q. Fu, H.R. Le, J.A. Williams, S.M. Spearing and W.I. Milne, "Diamond and diamond-like carbon MEMS," *Journal of micromechanics and microengineering*, vol. 17, pp. 147-163, 2007.
- [4] O. Auciello and A.V. Sumant, "Status review of the science and technology of ultrananocrystalline diamond (UNCD<sup>™</sup>) films and application to multifunctional devices," *Diamond & Related Materials*, vol. 19, pp.699-718, 2010.
- [5] W.M.V. Spengen, R. Puers, R. Mertens and I.D. Wolf, "A low frequency electrical test set-up for the reliability assessment of capacitive RF MEMS switches," *Journal of Micromechanics and Microengineering*, vol. 13, pp. 604-612, 2003.
- [6] W. M.V. Spengen, "MEMS reliability from a failure mechanisms perspective," *Microelectronics Reliability*, vol. 43, pp. 1049-1060, 2003.
- [7] S.M. Spearing and K.S. Chen, in "*Tribology Issues and Opportunities in MEMS*", edited by B. Bhushan, Kluwer Academic Publisher, The Netherlands, pp. 95, 1998.
- [8] Z. Rymuza, Z. Kusznierewicz, M. Misiak, K. Schrnidt-Szalowski, Z. Rzanek-Boroch and J. Sentek, "*Tribology Issues and Opportunities in MEMS*", edited by B. Bhushan, Kluwer Academic Publisher, The Netherlands, pp. 579, 1998.
- [9] O.A. Williams, M. Nesladek, M. Daenen, S. Michaelson, A. Hoffman, E. Osawa, K. Haenen and R.B. Jackman, "Growth, electronic properties and applications of nanodiamond," *Diamond and Related Materials*, vol. 17, pp. 1080-1088, 2008.
- [10] J.E. Butler and H. Windischmann, "Development in CVD-diamond synthesis during the past decades," *MRS Bulletin*, vol. 23, pp. 22-27, 1998.

- [11] M.A. Prelas G. Popovici and K.L. Biglow, Eds. (Marcel Dekker, NY, 1997)R.S. Sussmann (Ed.), *Handbook of Industrial Diamonds and Diamond Films*, Wiley, Chichester, UK, 2009.
- [12] J.E. Butler, and A.V. Sumant, "The CVD of nanodiamond materials," *Chemical Vapor Deposition*, vol.14-160, pp. 145, 2008.
- [13] J. Philip, P. Hess, T. Feygelson, J.E. Butler, S. Chattopadhyay, K.H. Chen and L.C. Chen, "Elastic, mechanical, and thermal properties of nanocrystalline diamond films," *Journal of Applied Physics*, vol. 93, pp. 2164-2171, 2003.
- [14] L. Sekaric, J.M. Parpia, H.G. Craighead, T. Feygelson, B.H. Houston and J.E. Butler, "Nanomechanical resonant structures in nanocyrstalline diamond," *Applied Physics Letters*, vol. 81, pp. 4455-4457, 2002.
- [15] E. Kohn, P. Gluche and M. Adamschik, "Diamond MEMs a new emerging technology," *Diamond and Related Materials*, vol. 8, pp. 934-940, 1999.
- [16] F.J.H. Guillen, K. Janischowsky, J. Kusterer, W. Ebert and E. Kohn, "Mechanical characterization and stress engineering of nanocrystalline," *Diamond and Related Materials*, vol. 14, pp. 411-415, 2005.
- [17] O.A. Williams, M. Daenen, J. D'Haen, K.Haenen, J. Maes, V.V. Moshchalkov, M. Nesládek and D.M. Gruen, "Comparison of the growth and properties of ultrananocrystalline diamond and nanocrystalline diamond," *Diamond & Related Materials*, vol. 15, pp. 654-658, 2006.
- [18] F.R. Marciano, L.F. Bonetti, D.A. Lima-Oliveira, C.B. Mello, M. Ueda, L.J. Corat and V.J. Trava-Airoldi, "Characterization of crystalline diamond incorporated diamond-like carbon films," *Diamond and Related Materials*, vol.19, pp. 1139-1143, 2010.
- [19] D.R. McKenzie, D. Muller and B.A. Pailthorpe, "Compressive-stress-induced formation of thin-film tetrahedral amorphous," *Physics Review Letters*, vol. 67, pp. 773-776, 1991.
- [20] M. Weiler, S. Sattel, K. Jung, H. Ehrhardt, V.S. Veerasamy and J. Robertson, "Highly tetrahedral, diamond-like amorphous hydrogenated carbon prepared from a plasma beam source," Applied Physics Letters, vol. 64, pp. 2797-2799, 1994.
- [21] S.S. Zuo, *Microwave Plasma-Assisted CVD Polycrystalline Diamond Films Deposition at Higher Pressure Conditions*, Ph.D. Dissertation, Michigan State University, 2009.
- [22] N.G. Ferreira, E. Abramof, E.J. Corat and V.J. Trava-Airoldi, "Residual stress and crystalline quality of heavily boron-doped diamond films analysed by micro-Raman spectroscopy and X-ray diffraction," *Carbon*, vol. 41, pp. 1301-1308, 2003.

- [23] P. Gupta, Synthesis, *Structure and Properties of Nanolayered DLC/DLC Films*, Ph.D. Dissertation, Louisiana State University, 2000.
- [24] Z.G. Hu, P. Prunici, P. Hess and K.H. Chen, "Optical properties of nanocrystalline diamond films from mid-infrared to ultraviolet using reflectometry and ellipsometry," *Journal of material science: material in electronics*, vol. 18, pp. 37-41, 2007.
- [25] E. Sillero, O.A. Williams, V. Lebedev, V. Cimalla, C.-C. Röhlig, C.E. Nebel and F. Calle, "Static and dynamic determination of the mechanical properties of nanocrystalline diamond micromachined structures," *Journal of micromechanics and Microengineering*, vol. 19, pp. 1-6, 2009.
- [26] S. Balachandran, J. Kusterer, D. Maier, M. Dipalo, T. Weller and E. Kohn, "High power nanocrystalline diamond RF MEMS- A combined look at mechanical and microwave properties," *International Microwaves, Communications, Antennas and Electronic Systems conference*, pp. 1-8, 2008.
- [27] H.D. Espinosa, B.C. Prorok, B. Peng, K.H. Kim, N. Moldovan, O. Auciello, J.A. Carlisle, D.M. Gruen and D.C. Mancini, "Mechanical properties of ultrananocrystalline diamond thin films relavant to MEMS/NEMS Devices," *Experimental mechanics*, vol. 43, pp. 256-268, 2003.
- [28] Halliday, Resnick, Walker, Fifth Edition of Fundamentals of Physics Extended, Wiley, New York, 1997.
- [29] H.O. Pierson, Handbook of Carbon, Graphite, *Diamond and Fullerenes Properties, Processing and Applications*, William Andrew Publishing/Noyes, 2004.
- [30] C. Beck and M. Ahmed, *Diamond Chemically Vapour Deposited Diamond*, Azom, 2004.
- [31] Y. Muto, T. Sugino and J. Shirafuji, "Electrical conduction in undoped diamond films prepared by chemical vapor deposition," *Applied Physics Letters*, vol. 59, pp. 843-845, 1991.
- [32] B. Huang, D.K. Reinhard, "Electric field dependent conductivity of polycrystalline diamond thin films," *Applied Physics Letters*, vol. 59, pp. 1494-1496, 1991.
- [33] M. Werner, O. Dorsch, A. Hinze, E. Obermeier, R.E. Haper, C. Johnston, P.R. Chalker and I.M. Buckley-Golder, "Space-charge-limited current flow and trap density in undoped diamond films," *Diamond and Related Materials*, vol. 13, pp. 825-828, 1993.
- [34] T. Sugino, Y. Muto, J. Shirafuji and K. Kobashi, "Electrical conduction mechanisms in polycrystalline chemically vapour-deposited diamond films," *Diamond and Related Materials*, vol. 2, pp. 797-802, 1993.

- [35] S. Jin and T.D. Moustakas, "Electrical conductivity studies of diamond films prepared by electron cyclotron resonance microwave plasma," *Applied Physics Letters*, pp. 2354-2356, 1993.
- [36] A.K. Kulkarni, A. Shrotriya, P. Cheng, H. Rodrigo, R. Bashyam and D.J. Keeble, "Electrical properties of diamond thin films grown by chemical vapor deposition technique," *Thin Solid Films*, vol. 253, pp. 141-145, 1994.
- [37] A.K. Kulkarni, K. Tey and H. Rodrigo, "Electrical characterization of CVD diamond thin films grown on silicon substrates," *Thin Solid Films*, vol. 270, pp. 189-193, 1995.
- [38] G. De Cesare, S. Salvatori, R. Vincenzoni, P. Ascarelli, E. Cappelli, F. Pinzari and F. Galluzzi, "On the electrical properties of polycrystalline diamond films on silicon," *Diamond and Related Materials*, vol. 4, pp. 628-631, 1995.
- [39] A.K. Sikder, A.P. Jacob, T. Sharda, D.S. Misra, M. Pandey, D. Kabiraj and D.K. Avasthi, "DC electrical conductivity of chemical vapour deposited diamond sheets: a correlation with hydrogen content and paramagnetic defects," *Thin Solid Films*, vol. 332, pp. 98-102, 1998.
- [40] L. Wang, Y. Xia, J. Ju and W. Zhang, "Electrical properties of chemical vapor deposition diamond films and electrical response to X-ray," *Diamond and Related Mateirals*, vol. 9, pp. 1617-1620, 2000.
- [41] B.J. Lee, B.T. Ahn, J.K. Lee and Y.J. Baik, "A study on the conduction path in undoped polycrystalline diamond films," *Diamond and Related Materials*, vol. 10, pp. 2174-2177, 2001.
- [42] Q. Su, J. Lu, L. Wang, J. Liu, J. Ruan, J. Cui, W. Shi and Y. Xia, "Electrical properties of [100]-oriented CVD diamond film," *Solid-State Electronics*, vol. 49, pp. 1044-1048, 2005.
- [43] E.J. Correa, Y. Wu, J. Wen, R. Chandrasekharan and M. Shannon, "Electrical conduction in undoped ultrananocrystalline diamond thin films and its dependence on chemical composition and crystalline structure," *Journal of Applied Physics*, vol. 102, 2007.
- [44] B.R. Stoner, J.T. Glass, L. Bergman, R.J. Nemanich, L.D. Zoltal and J.W. Vandersande, "Electrical conductivity and photoluminescence of diamond films grown by downstream microwave plasma CVD," *Journal of Electronic Materials*, vol. 21, pp. 629-634, 1992.
- [45] A.P. Malshe, B.S. Park, W.D. Brown and H.A. Naseem, "A review of techniques for polishing and planarizing chemically vapor-deposited (CVD) diamond films and substrates," *Diamond and Related Materials*, vol. 8, pp. 1198-1213, 1999.

- [46] H. Ohashi, E. Ishiguro, T. Sasano and K. Shobatake, "Synchrotron radiation excited etching of diamond," *Applied Physics Letters*, vol. 68, pp. 3713-3715, 1996.
- [47] A. Massod, D.M. Aslam, M.A. Tamor and T.J. Potter, "Techniques for patterning of CVD diamond films on non-diamond substrate," *Journal of the Electrochemical Society*, vol. 138, pp. L67-68, 1991.
- [48] T. Roppel, R. Ramesham and S.Y. Lee, "Thin film diamond microstructures," *Thin Solid Films*, vol. 212, pp. 56-62, 1992.
- [49] J.L. Davidson, C. Ellis and R. Ramesham, "Selective deposition of diamond films," *New Diamond*, vol. 6, pp. 29-32, 1990.
- [50] T. Inoue, H. Tachibana, K. Kumagai, K. Miyata, K. Nishimura, K. Kobashi and A. Nakaue, "Selected-area deposition of diamond films," *Journal of Applied Physics*, vol. 67, pp. 7329-7336, 1990.
- [51] K. Hirabayashi, Y. Taniguchi, O. Takamatsu, T. Ikeda, K. Ikoma and N.I. Kurihara, "Selective deposition of diamond crystals by chemical vapor deposition using a tungstenfilament method," *Applied Physics Letters*, vol. 53, pp. 1815-1817, 1988.
- [52] N.N. Efremow, M.W. Geis, and D.C. Flanders, "Ion-beam-assisted etching of diamond," *Journal of Vaccum and Science Technology*, B3 (1), 1985, pp. 416-418.
- [53] P.W. Leech, G.K. Reeves, A.S. Holland, and F. Shanks, "Ion beam etching of CVD diamond film in Ar, Ar/O<sub>2</sub> and Ar/CF<sub>4</sub> gas mixtures," *Diamond & Related Materials*, vol. 11 2002, pp. 833-836.
- [54] G.S. Sandhu, and W.K. Chu, "Reactive ion etching of diamond," *Applied Physics Letter*, vol. 55, no. 5, 1989, pp.437-438.
- [55] O. Dorsch, M. Werner and E. Obermeier, "Dry etching of undoped and boron doped polycrystalline diamond films," *Diamond & Related Materials*, vol. 4, 1995, pp.456-459.
- [56] C. Vivensang, L. Ferlazzo-Manin, and M.F. Ravet, "Surface smoothing of diamond membranes by reactive ion etching process," *Diamond & Related Materials*, vol. 5 1996, pp. 840-844.
- [57] Gopi M.R. Sirineni, H.A. Naseem, A.P. Malshe, and W.D. Brown, "Reactive ion etching of diamond as a means of enhancing chemically-assisted mechanical polishing efficiency," *Diamond and Related Materials*, vol. 6, 1997, pp. 952-958.

- [58] H. Shiomi, "Reactive ion etching of diamond in O<sub>2</sub> and CF<sub>4</sub> Plasma, and fabrication of porous diamond for field emitter cathodes," *Japanese Journal of Applied Physics*, vol. 36, 1997, pp. 7745-7748.
- [59] E.-S. Baik, and Y.-J. Baik, "Aligned diamond nanowhiskers," *Journal of Materials Research*, vol. 15, no. 14, 2000, pp. 923-926
- [60] E.-S. Baik, Y.-J. Baik, S.W. Lee, and D. Jeon, "Fabrication of diamond nano-whiskers," *Thin Solid Films*, no. 377-378, 2000, pp. 295-298.
- [61] E.-S. Baik, Y.-J. Baik, and D. Jeon, "Control of diamond micro-tip geometry for field emitter," *Thin Solid Films*, no. 377-378, 2000, pp. 299-302.
- [62] T. Shibata, Y. Kitamoto, K. Unno, and E. Makino, "Micromachining of diamond film for MEMS applications," *Journal of Microelectromechanical Systems*, vol. 9, no. 1, 2000, pp. 47-51.
- [63] R. Otterbach, U. Hilleringmann, and K. Goser, "Reactive ion etching of CVD-diamond for sensor devices with Al feature size of 100 nm," *Industrial Electronics Society Conference*, vol.3, 2000, pp. 1873-1877.
- [64] Y. Nishibayashi, Y. Ando, H. Saito, T. Imai, T. Hirao, and K. Oura, "Anisotropic etching of a fine column on a single crystal diamond," *Diamond and Related Materials*, vol. 10, 2001, pp. 1732-1735.
- [65] P.W. Leech, G.K. Reeves, and A. Holland "Reactive ion etching of diamond in CF<sub>4</sub>, O<sub>2</sub>,
   O<sub>2</sub> and Ar-based mixtures," *Journal of Materials Science*, vol. 36, no. 14, 2001, pp. 3453-3459.
- [66] Y. Fu, H. Du, and J. Miao, "Patterning of diamond microstructures on Si substrate by bulk and surface micromachining," *Journal of Materials Processing Technology*, 132, 2003, pp. 73-81.
- [67] X. Wang, G.D. Hong, J. Zhang, B.L. Lin, H.Q. Gong, and W.Y. Wang, "Precise patterning of diamond films for MEMS application," *Journal of Materials Processing Technology*, vol. 127, 2002, pp. 230-233.
- [68] Y. Ando, Y. Nishibayashi, K. Kobashi, T. Hirao, and K. Oura, "Smooth and high-rate reactive ion etching of diamond," *Diamond and Related Materials*, vol. 11, 2002, pp. 824-827.
- [69] Y. Ando, Y. Nishibayashi, A. Sawabe, "'Nano-rods' of single crystalline diamond," *Diamond and Related Materials*, vol. 13, 2004, pp. 633-637.

- [70] C.Y. Li, and A. Hatta, "Effect of metal coating on the formation of diamond whiskers in O<sub>2</sub> RF plasma," *Diamond & Related Materials*, vol. 15, 2006, pp. 357-360.
- [71] G. Ding, H. Mao, Y. Cai, Y. Zhang, X. Yao, and X.L. Zhao, "Micromachining of CVD diamond by RIE for MEMS applications," *Diamond & Related Materials*, vol. 14, 2005, pp. 1543-1548.
- [72] C.Y. Li, and A. Hatta, "Preparation of diamond whiskers using Ar, O<sub>2</sub> plasma etching," *Diamond & Related Materials*, vol. 14, 2005, pp. 1780-1783.
- [73] C.Y. Li, and A. Hatta, "Nanowhiskers formation by radio frequency Ar/O<sub>2</sub> plasma etching of Al coated diamond films," *Thin Sold Films*, vol. 515, 2007, pp. 4172-4176.
- [74] M.D. Stoikou, P. John, and J.I. Wilson, "Unusual morphology of CVD diamond surfaces after RIE," *Diamond and Related Materials*, vol. 17, 2008, pp. 1164-1168.
- [75] S. Grot, G. Gildenblat, and a. Badzian, "Diamond thin-film recessed gate field-effect transistors fabricated by electron cyclotron resonance plasma etching," *IEEE Electron Device Letters*, vol. 13, 1992, pp. 462-464.
- [76] S.J. Pearton, A. Katz, F. Ren, and J.R. Lothian, "ECR plasma etching of chemically vapour deposited diamond thin films," *Electronic Letters*, vol.28, no. 9. 1992, pp. 822-824.
- [77] S.A. Grot, R.A. Ditizio, G.Sh. Gildenblat, A.R. Badzian, and S.J. Fonash, "Oxygen based electron cyclotron resonance etching of semiconducting homoepitaxial diamond films," *Applied Physics Letters*, vol. 61 no. 19, 1992, pp. 2326-2328.
- [78] S. Kiyahara, Y. Yagi, and K.i Mori, "Plasma etching of CVD diamond films using an ECR-type oxygen source," *Nanotechnology*, vol. 10, 1999, pp. 385-388.
- [79] M. Bernard, A. Deneuville, T. lagarde, E. Treboux, J. Pelletier, P. Muret, N. Casanova, and E. Gheeraert, "Etching of p- and n-type doped nanocrystalline diamond using an ECR oxygen plasma source," *Diamond & Related Materials*, vol. 11, 2002, pp. 828-832.
- [80] M. Bernard, A. Deneuville, L. Ortega, K. Ayadi and P. Muret, "Electron cyclotron resonance oxygen plasma etching of diamond," *Diamond and Related Materials*, vol. 13, 2004, pp. 287-291.
- [81] D.T. Tran, T.A. Grotjohn, D.K. Reinhard, and J. Asmussen, "Microwave plasma-assisted etching of diamond," *Diamond and Related Materials*, vol. 17, 2008, pp. 717-721.
- [82] D.S. Hwang, T. Saito, and N. Fujimori, "New etching process for device fabrication using diamond," *Diamond & Related Materials*, vol. 13, 2004, pp. 2207-2210.

- [83] J. Enlund, J. Isberg, M. Karlsson, F. Nikolajeff, J. Olsson, and D. Twitchen, "Anisotropic dry etching of boron doped single crystal CVD diamond," *Carbon*, vol. 43, 2005, pp. 1839-1842.
- [84] T. Yamada, H. Yoshikawa, H. Uetsuka, S. Kumaragurubaran, N. Tokuda, and S. Shikata, "Cycle of two-step etching process using ICP for diamond MEMS applications," *Diamond and Related Materials*, vol. 16, 2007, pp. 996-999.
- [85] H. Uetsuka, T. Yamada, and S. Shikata, "ICP etching of polycrystalline diamonds: Fabrication of diamond nano-tips for AFM cantilevers," *Diamond and Related Materials*, vol. 17, 2008, pp. 728-731.
- [86] B. Chapman, *Glow Discharge Processes*, John Wiley and Sons, Inc., New York, 38 (1980).
- [87] D.M. Aslam, I. Taher and A. Masood, "Piezoresistivity in vapor-deposited diamond films," *Applied Physics Letters*, vol. 60, pp. 2923-2925, 1992.
- [88] I. Taher, *CVD Diamond Piezoresistive Microsensors*, Ph.D. Dissertation, Michigan State University, 1994.
- [89] D.R. Wur and J.L. Davidson, "Piezoresistivity of polycrystalline diamond films," *Materials Research Society Symposium Proceeding*, vol. 283, pp. 879, 1993.
- [90] O. Dorsch, K. Holzner, M. Werner, E. Obermeier, R.E. Harper, C.Johnston, P.R. Chalker and I.M. Buckley-Golder, "Piezoresistive effect in boron-doped diamond thin films," *Diamond and Related Materials*, vol. 2, pp. 1096-1099, 1993.
- [91] W. Wanlu and L. Kejun, "Piezoresistive effect of diamond films produced by dc plasma CVD," *Chinese Physics Letters*, vol. 11, pp. 589-592, 1994.
- [92] M. Deguchi, N. Hase, M. Kitabatake, H. Kotera, S. Shima and H. Sakakima, "Piezoresistive properties of *p*-type CVD diamond films," *Diamond Films Technology*, vol.6, pp. 77, 1996.
- [93] M. Deguchi, M. Kitabatake and T. Hirao, "Piezoresistive properties of chemical vapor deposited *p*-type diamond strain gauges fabricated on diaphragm structure," *Diamond and Related Materials*, vol. 5, pp. 728-731, 1996.
- [94] M. Deguchi, N. Hase, M. Kitabatake, H. Kotera, S. Shima and M. Kitagawa, "Piezoresistive property of CVD diamond films," *Diamond and Related Materials*, vol. 6, pp. 367-373, 1997.
- [95] Y. Boiko, P. Gonon, S. Prawer and D.N. Jamieson, "Piezoresistivity of boron doped CVD diamond films," *Materials Science and Engineering B*, vol. 46, pp. 112-114, 1997.

- [96] W.L. Wang, X. Jiang, K. Taube and C.-P. Klages, "Piezoresistivity of polycrystalline *p*-type diamond films of various doping levels at different temperatures," *Journal of Applied Physics*, vol. 82, pp. 729-732, 1997.
- [97] S. Sahli and D.M. Aslam, "Ultra-high sensitivity intra-grain poly-diamond piezoresistors," *Sensors and Actuators A*, vol. 71, pp. 193-197, 1998.
- [98] L. Fang, W.L. Wang, P.D. Ding, K.J. Liao and J. Wang, "Study on the piezoresistive effect of crystalline and polycrystalline diamond under uniaxial strains," *Journal of Applied Physics*, vol.86, pp. 5185-5193, 1999.
- [99] M. Adamschik, R. Müller, P. Gluche, A. Flöter, W. Limmer, R. Sauer and E. Kohm, "Analysis of piezoresistive properties of CVD-diamond films on silicon," *Diamond and Related Materials*, vol. 10, pp. 1670-1675, 2001.
- [100] A. Yamamoto and T. Tsutsumoto, "Piezoresistive effect of CVD polycrystalline diamond films," *Diamond and Related Materials*, vol. 13, pp. 863-866, 2004.
- [101] A. Yamamoto, N. Norio and T. Takahiro, "Evaluation of diamond gauge factor up to 500 °C," *Diamond & Related Materials*, vol. 16, pp. 1670-1675, 2007.
- [102] P. Kulha, A. Kromka, O. Babchenko, M. Vanecek, M. Husak, O.A. Williams and K. Haenen, "Nanocrystalline diamond piezoresistive sensor," *Vacuum*, vol. 84, pp. 53-56, 2010.
- [103] M. Deguchi, M. Kitabatake and T. Hirao, "Piezoresistive properties of chemical vapor deposited *p*-type diamond strain gauges fabricated on diaphragm structure," *Diamond and Related Materials*, vol. 5, pp. 728-731, 1996.
- [104] I. Taher, D.M. Aslam, M.A. Tamor, T.J. Potter and R.C. Elder, "Piezoresistive microsensors using *p*-type CVD diamond films," *Sensors and Actuators A: Physical*, vol. 45, pp. 35-43, 1994.
- [105] M. Werner, O. Dorsch and E. Obermeier, "High-temperature pressure sensor using *p*-type diamond piezoresistors," *Diamond and Related Materials*, vol. 4, pp. 873-876, 1995.
- [106] D.R. Wur, J.L. Davidson, W.P. Kang and D.L. Kinser, "Polycrystalline diamond pressure sensor," *Journal of Microelectromechanical Systems*, vol. 4, pp. 34-41, 1995.
- [107] S. Sahli and D.M. Aslam, "Pressure microsensors using *p*-type diamond films," *The 8th International Conference on Solid-State Sensors and Actuators*, Sweden, pp. 592-595, 1995.

- [108] J.L. Davidson, D.R. Wur, W.P. Kang, D.L. Kinser and D.V. Kerns, "Polycrystalline diamond pressure microsensor," *Diamond and Related Materials*, vol. 5, pp. 86-92, 1996.
- [109] A. Yamamoto, N. Nawachi, T. Tsutsumoto and A. Terayama, "Pressure sensor using ptype polycrystalline diamond piezoresistors," *Diamond & Related Materials*, vol. 14, pp. 657-660, 2005.
- [110] J. Lu, Z. Cao, D.M. Aslam, N. Sepúlveda and J.P. Sullivan, "Diamond micro and nano resonators using laser, capacitive or piezoresistive detection," *the 3rd IEEE International Conference on Nano/Micro Engineered and Molecular Systems*, Sanya, pp. 873-876, 2008.
- [111] Z. Cao and D.M. Aslam, "Piezoresistive sensor technology for RFMEMS using *p*-type polycrystalline diamond," *IEEE Nanotechnology Materials and Devices Conference*, Traverse City, pp. 190-195, 2009.
- [112] A. Tibrewala, E. Peiner, R. Bandorf, S. Biehl and H. Lüthje, "Piezoresistive gauge factor of hydrogenated amorphous carbon films," *Journal of Micromechanics and Microengineering*, vol. 16, pp. 75-81, 2006.
- [113] E. Peiner, A. Tibrewala, R. Bandorf, S. Biehl, H. Lüthje and L. Doering, "Micro force sensor with piezoresisive amorphous carbon strain gauge," *Sensors and Actuators A*, vol. 130-131, pp. 75-82, 2006.
- [114] A. Tibrewala, E. Peiner, R. Bandorf, S. Biehl and H. Lüthje, "Longitudinal and transversal piezoresistive effect in hydrogenated amorphous carbon films," *Thin Solid Films*, vol. 515, pp. 8028-8033, 2007.
- [115] A. Tibrewala, E. Peiner, R. Bandorf, S. Beihl and H. Lüthje, "The piezoresistive effect in diamond-like carbon films," *Journal of Micromechanics and Microengineering*, vol. 17, pp. 77-82, 2007.
- [116] Š. Meškinis, R. Gudaitis, V. Kopustinskas and S. Tamulevičius, "Electrical and piezoresistive properties of ion beam deposited DLC films," *Applied Surface Science*, vol. 254, pp. 5252-5256, 2008.
- [117] C.T.-C. Nguyen, "Integrated micromechanical radio front-ends," *International Symposium on VLSI Technology, Systems and Application*, pp. 3-4, 2008.
- [118] K. Wang, A.-C. Wong and C.T.-C. Nguyen, "VHF free-free beam high-Q micromechanical resonators," *Journal of Microelectromechanical systems*, vol. 9, pp. 347-360, 2000.

- [119] J. Wang, Z. Ren and C.T.-C. Nguyen, "1.156-GHz self-aligned vibrating micromechanical disk resonator," *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control*, vol. 51, pp. 1607-1628, 2004.
- [120] Y. Xie, S. Li, Y. Lin, Z. Ren, Clark T.-C. Nguyen, "1.52-GHz micromechanical extensional wine-glass mode ring resonators," *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control*, vol. 55, pp. 890-907, 2008.
- [121] S. Roy, R.G. Deanna, C.A. Zorman and M. Mehregany, "Fabrication and characterization of polycrystalline SiC resonators," *IEEE Transactions on Electron Devices*, vol. 49, pp. 2323-2332, 2002.
- [122] D.J. Young, İ.E. Pehlivanoğlu and C.A. Zorman, "Silicon carbide MEMS-resonatorbased oscillator," *Journal of Micromachanics and Microengineering*, vol. 19, pp. 1-10, 2009.
- [123] M.D. Whitfield, B. Audic, C.M. Flannery, L.P. Kehoe, G.M. Crean and R.B. Jackman, "Acoustic wave propagation in free standing CVD diamond: Influence of film quality and temperature," *Diamond and Related Materials*, vol. 8, pp. 732-737, 1999.
- [124] R.Hull, *Properties of Crystalline Silicon*. Portland: IEE Publishing, 1999.
- [125] G.L. Harris, *Properties of Silicon Carbide*. Portland: IEE Publishing, 1995.
- [126] M. Liao, C. Li, S. Hishita and Y. Koide, "Batch production of single-crystal diamond bridges and cantilevers for microelectromechanical systems," *Journal of Micromechanics* and Microengineering, vol. 20, 2010.
- [127] J. Wang, J.E. Butler, D.S.Y. Hsu and C.T.-C. Nguyen, "CVD polycrystalline diamond high-Q micromechanical resonators," 15th International IEEE Microelectromechanical Systems Conference, Las Vegas, pp. 657-660, 2002.
- [128] N. Sepúlveda, D.M. Aslam and J.P. Sullivan, "Polycrystalline diamond MEMS resonator technology for sensor applications," *Diamond and Related Materials*, vol. 15, pp. 398-403, 2005.
- [129] Sepúlveda, J. Lu, D.M. Aslam, J.P. Sullivan, "High-performance polycrystalline diamond micro- and nanoresonators," *Journal of Microelectromechanical Systems*, vol. 17, pp. 473-482, 2008.
- [130] J. Wang, J.E. Butler, D.S.Y. Hsu and C.T.-C. Nguyen, "High-Q micromechanical resonators in CH<sub>4</sub>-reactant-optimized high acoustic velocity CVD polydiamond," *Solid-State Sensor, Actuator, and Microsystems Workshop*, Hilton Head, South Carolina, pp. 61-62, 2002.
- [131] J. Wang, J.E. Butler, T. Feygelson and C.T.-C. Nguyen, "1.51-GHz nanocrystalline diamond micromechanical disk resonator with material-mismatched isolating support," *17th International IEEE Microelectromechanical Systems Conference*, Masstricht, The Netherland, pp. 641-644, 2004.
- [132] M. Imboden, P. Mohanty, A. Gaidarzhy, J. Rankin and B.W. Sheldon, "Scaling of dissipation in megahertz-range micromechanical diamond oscillators," *Applied Physics Letters*, vol. 90, pp. 1-4, 2007.
- [133] A.B. Hutchinson, P.A. Truitt, K.C. Schwab, L. Sekaric, J.M. Parpia, H.G. Craighead and J.E. Butler, "Dissipation in nanocrystalline-diamond nanomechanical resonators," *Applied Physics Letters*, vol. 84, pp. 972-974, 2004.
- [134] J.W. Baldwin, M.K. Zalalutdinov, T. Feygelson, B.B. Pate, J.E. Bulter and B.H. Houston, "Nanocrystalline diamond resonator array for RF signal processing," *Diamond & Related materials*, vol. 15, pp. 2061-2067, 2006.
- [135] A. Gaidarzhy, M. Imboden, P. Mohanty, J. Rankin and B.W. Sheldon, "High quality factor gigahertz frequencies in nanomechanical diamond resonators," *Applied Physics Letters*, vol. 91, 2007.
- [136] V.P. Adiga, A.V. Sumant, S. Suresh, C. Gudeman, J.A. Carlisle, O. Auciello and R.W. Carpick, "Mechanical stiffness and dissipation in ultrananocrystalline diamond resonators," *Physical Review B*, vol. 9, 2009.
- [137] D.H.C. Chua, W.I. Milne, D. Sheeja, B.K. Tay and D. Schneider, "Fabrication of diamond-like amorphous carbon cantilever resonators," *Journal of Vacuum Science and Technology B*, vol. 22, pp. 2680-2684, 2004.
- [138] D.A. Czaplewski, J.P. Sullvan, T.A. Friedmann and J.R. Wendt, "Mechanical dissipation at elevated temperatures in tetrahedral amorphous carbon oscillators," *Diamond and Related Materials*, vol. 15, pp. 309-312, 2006.
- [139] M. Imboden, P. Mohanty, A. Gaidarzhy, J. Rankin and B.W. Sheldon, "Scaling of dissipation in megahertz-range micromechanical diamond oscillators," *Applied Physics Letters*, vol. 90, pp. 1-4, 2007.
- [140] J. Wang, J.E. Butler, D.S.Y. Hsu and C.T.-C. Nguyen, "CVD polycrystalline diamond high-Q micromechanical resonators," *15th International IEEE Microelectromechanical Systems Conference*, Las Vegas, pp. 657-660, 2002.
- [141] J. Wang, J.E. Butler, T. Feygelson and C.T.-C. Nguyen, "1.51-GHz nanocrystalline diamond micromechanical disk resonator with material-mismatched isolating support," *17th International IEEE Microelectromechanical Systems Conference*, Masstricht, The Netherland, pp. 641-644, 2004.

- [142] S. Ertl, M. Adamschik, P. Schmid, P. Gluche, A. Flöter and E. Kohn, "Surface micromachined diamond microswitch," *Diamond and Related Materials*, vol. 9, pp. 970-974, 2000.
- [143] M. Adamschik, J.Kusterer, P. Schmid, K.B. Schad, D. Grobe, A. Flöter and E. Kohn, "Diamond microwave micro relay," *Diamond and Related Materials*, vol. 11, pp. 672-676, 2002.
- [144] Y. Gurbuz, O. Esame, I. Tekin, W.P. Kang and J.L. Davidson, "Diamond semiconductor technology for RF device applications," *Solid-State Electronics*, vol. 49, pp. 1055-1070, 2005.
- [145] R. Ramesham, T. Roppel and C. Ellis, "Fabrication of microchannels in synthetic polycrystalline diamond thin films for heat sinking applications," *Journal of Electrochemical Society*, vol. 138, pp. 1706-1709, 1991.
- [146] S. Guillaudeu, X. Zhu and D.M. Aslam, "Fabrication of 2-μm wide poly-crystalline diamond channels using silicon molds for micro-fluidic applications," *Diamond and Related Materials*, vol. 12, pp. 65-69, 2003.
- [147] D.H. Szarowski, M.D. Andersen, S. Retterer, A.J. Spence, M. Isaacson, H.G. Craighead, J.N. Turner and W. Shain, "Brain responses to micromachined silicon devices," *Brain Research*, vol. 983, pp. 23-35, 2003.
- [148] L. Tang, C. Tsai, W.W. Gerberich, L. Kruckebeu and D.R. Kania, "Biocompatibility of chemical-vapour-deposited diamond," *Biomaterials*, vol. 16, pp. 483-488, 1995.
- [149] Y. Tang, D. M. Aslam, J. Wang and K. D. Wise, "Poly-Crystalline Diamond Piezoresistive Position Sensors for Cochlear Implant Probe," *Proceedings of Solid-State Sensors, Actuators and Microsystems*, pp. 542-546, 2005.
- [150] G.M. Swain and R. Ramesham, "The electrochemical activity of borondoped polycrystalline diamond thin film electrode," *Analytical Chemistry*, vol. 65, pp. 345-351, 1993.
- [151] H.-Y. Chan, M. Varney, D.M. Aslam and K.D. Wise, "Fabrication and characterization of all-diamond microprobes for electrochemical analysis," *IEEE Internatinal Conference* on Nano/Micro Engineered and Molecular Systems, pp. 532-535, 2008.
- [152] H.-Y. Chan, D.M. Aslam, S.H. Wang, G.M. Swain and K.D. Wise, "Fabrication and testing of a novel all-diamond neural probe for chemical detection and electrical sensing applications," *IEEE International. Conference on Micro Electro Mechanical Systems*, pp. 244-247, 2008.

- [153] H.-Y. Chan, D.M. Aslam, J. Wiler and B. Casey, "A novel diamond microprobe for neuro-chemical and –electrical recording in neural prosthesis," *Journal of Microelectromechanical Systems*, vol. 18, pp. 511-521, 2009.
- [154] A. Bennett, J. Wang, Y. Show and G. M. Swain, "Effect of sp2-bonded nondiamond carbon impurity on the response of boron-doped polycrystalline diamond thin-film electrodes", *Journal of Electrochemical Society* vol. 151, pp. 306-313, 2004.
- [155] C.E. Nebel, H. Kato, B. Rezek, D. Shin, D. Takeuchi, H. Watanabe and T. Yamamoto, "Electrochemical properties of undoped hydrogen terminated CVD diamond", *Diamond and Related Materials*, vol. 15, pp. 264-268, 2006.
- [156] G.M. Swain and R. Ramesham, "The electrochemical activity of boron-doped polycrystalline diamond thin film electrode", *Analytical Chemistry*, vol. 65, pp. 345-351,1993.
- [157] H.-Y. Chan, *Polycrystalline CVD Diamond Probes For Use in In Vivo and In Vitro Neural Studies*, Ph.D. Dissertation, Michigan State University, 2008.
- [158] X. Zhu and D.M. Aslam, "CVD diamond thin film technology for MEMS packaging," *Diamond & Related Materials*, vol. 15, pp. 254-258, 2006.
- [159] X. Zhu, D.M. Aslam and J.P. Sullivan, "The application of polycrystalline diamond in a thin film packaging process for MEMS resonators," *Diamond & Related Materials*, vol. 15, pp. 2068-2072, 2006.
- [160] X. Zhu, D.M. Aslam, Y. Tang, B.H. Stark and K. Najafi, "The fabrication of all-diamond packaging panels with built-in interconnects for wireless integrated microsystems," *Journal of Microelectromechanical Systems*, vol. 13, pp. 396-405, 2004.
- [161] H.H. Berger, "Models for contacts to planar devices," *Solid-State Electronics*, vol. 15, pp. 145-148, 1972.
- [162] L. Lin, A.P. Pisano and R.T. Howe, "A micro strain gauge with mechanical amplifier," *Journal of Microelectromechanical Systems*, vol. 6, pp. 313-321, 1997.
- [163] J. wang and F. Lü, "Effect of pressure on the nucleation of diamond with addition of oxygen in the microwave plasma chemical vapor deposition system," *Chinese Physics Letters*, vol. 13, pp. 473-476, 1996.
- [164] T.H. Borst and O. Weis, "Electrical characterization of homoepitaxial diamond films doped with B, P, Li and Na during crystal growth," *Diamond and Related Materials*, vol. 4, pp. 948-953, 1995.

- [165] T. Tachibana and J. Glass, *Electrical contacts to diamond*, in *Diamond: electronic properties and applications*, L.S. Pan and D.R. Kania, Editors. 1995, Kluwer Academic Publishers: Norwell.
- [166] S. Sahli and D.M. Aslam, "Nonuniform conduction in B-doped chemical vapor deposited diamond studied by intra- and intergrain measurements," *Applied Physics Letters*, vol. 70, pp. 2192-2194, 1997.
- [167] J.G. Kim and J. Yu, "Comparative study of residual stresses measurement methods on CVD diamond films," *Scripta Materialia*, vol. 39, pp. 807-814, 1998.
- [168] W. Fang and J.A. Wickert, "Determining mean and gradient residual stresses in thin films using micromachined cantilevers," *Journal of micromechanics and Microengineering*, vol. 6, pp. 301-309, 1996.
- [169] F.J. Hernández Guillén, K. Janischowsky, J. Kusterer, W. Ebert and E. Kohn, "Mechanical characterization and stress engineering of nanocyrstalline dimaond films for MEMS applications," *Diamond & Related Materials*, vol. 14, pp. 411-415, 2005.
- [170] J. Zhang, J.W. Zimmer, R.T. Howe and R. Maboudian, "Characterization of boron-doped micro- and nanocrystalline diamond films deposited by wafer-scale hot filament chemical vapor deposition for MEMS applications," *Diamond & Related Materials*, vol. 17, pp. 23-28, 2008.
- [171] O. Auciello, J. Birrell, J.A. Carlisle, J.E. Gerbi, X. Xiao, B. Peng and H.D. Espinosa, "Materials science and fabrication processes for a new MEMS technology based on ultracrystalline diamond thin films," *Journal of Physics: Condensed Matter*, vol. 16, pp. 539-552, 2004.
- [172] N. Sepúlveda, J. Lu, D.M. Aslam, J.P. Sullivan, "High-performance polycrystalline diamond micro- and nanoresonators," *Journal of Microelectromechanical Systems*, vol. 17, pp. 473-482, 2008.
- [173] P. J. Petersan and S. M. Anlage, "Measurement of resonant frequency and quality factor of microwave resonators: Comparison of methods," *Journal of Applied Physics*, vol. 84, pp. 3392-3402, 1998.
- [174] F. Lochon, I. Dufour and D. Rebière, "A microcantilever chemical sensor optimization by taking into account losses," Sensors and Actuators B, vol. 118, pp. 292-296, 2006.
- [175] N. Sepúlveda, *Polycrystalline Diamond RF MEMS Resonator Technology And Characterization*, Ph.D. Dissertation, Michigan State University, 2005.
- [176] C.R. Eddy Jr., D.L. Youchison, B.D. Startwell and K.S. Grabowski, "Deposition of diamond onto aluminum by electron cyclotron resonance microwave plasma-assisted CVD," *Journal of Materials Research*, vol. 7, pp.3255-3259, 1992.

- [177] L. Fang, W.L. Wang, P.D. Ding, K.J. Liao and J. Wang, "Study on the piezoresistive effect of crystalline and polycrystalline diamond under uniaxial strains," *Journal of Applied Physics*, vol. 86, pp. 5186-5193, 1999.
- [178] M.M. Mandurah, K.C. Saraswat, C.R. Helms and T.I. Kamins, "Dopant segregation in polycrystalline silicon," *Japanese Applied Physics*, vol. 51, pp. 5755-5763, 1980.
- [179] P.J. French and A.G.R. Evans, "Piezoresistance in polysilicon," *Electronics Letter*, vol. 20, pp. 999-1000, 1984.
- [180] Y. Tang, D.M. Aslam, J. Wang and K.D. Wise, "Study of polycrystalline diamond piezoresistive position sensors for application in cochlear implant probe," *Diamond & Related Materials*, vol. 15, pp. 199-202, 2006.
- [181] M. Deguchi, M. Kitabatake and T. Hirao, "Piezoresistive properties of chemical vapor deposited *p*-type diamond strain gauges fabricated on diaphragm structure," *Diamond and Related Materials*, vol. 5, pp. 728-731, 1996.
- [182] I. Taher, D.M. Aslam, M.A. Tamor, T.J. Potter and R.C. Elder, "Piezoresistive microsensors using *p*-type CVD diamond films," *Sensors and Actuators A: Physical*, vol. 45, pp. 35-43, 1994.
- [183] K. Hermansson, U. Lindberg, B. Hök and G. Palmskog, "Wetting properties of silicon surfaces," *IEEE Int. Conf. on Solid-state Sensors and Actuators*, pp. 193-196, 1991.