





This is to certify that the dissertation entitled

POLY-CRYSTALLINE DIAMOND (POLY-C) TECHNOLOGY AND PIEZORESISTIVE SENSOR APPLICATION FOR COCHLEAR PROSTHESIS

presented by

YUXING TANG

has been accepted towards fulfillment of the requirements for the

Ph. D.

Electrical Engineering

In. Main

degree in

Major Professor's Signature

5-3-06

Date

MSU is an Affirmative Action/Equal Opportunity Institution

PLACE IN RETURN BOX to remove this checkout from your record. TO AVOID FINES return on or before date due. MAY BE RECALLED with earlier due date if requested.

1

DATE DUE	DATE DUE	DATE DUE
	· · · · · · · · · · · · · · · · · · ·	2/05 p:/CIRC/DateDue.indd-p.1

POLYCRYSTALLINE DIAMOND (POLY-C) TECHNOLOGY AND PIEZORESISTIVE SENSOR APPLICATION FOR COCHLEAR PROSTHESIS

By

Yuxing Tang

A DISSERTATION

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

DOCTOR OF PHILOSOPHY

Department of Electrical Engineering

2006

ABSTRACT

POLYCRYSTALLINE DIAMOND (POLY-C) TECHNOLOGY AND PIEZORESISTIVE SENSOR APPLICATION FOR COCHLEAR PROSTHESIS

By

Yuxing Tang

Polycrystalline diamond (poly-C), with high piezoresistivity and unique mechanical, chemical and thermal properties, becomes a promising material for piezoresistive sensor application in particular in harsh environments and high temperature. However, due to the technology limitation and integration difficulty, the application of poly-C is far from being a standard technology like silicon process, which limited the mass production and commercialization of diamond based sensors.

The research of this dissertation developed several optimum poly-C technologies for better integration with microsystems and analyzed the dependence of poly-C piezoresistivity on film properties. Then, the application of poly-C piezoresistive sensor was successfully demonstrated by integrating it into the silicon thin film cochlear implant probe as the position sensor for its high piezoresistive gauge factor (GF).

A new diamond seeding method, spin-coating of diamond powder loaded water (DPLW), was developed with uniform, nondestructive and repeatable high seeding density $(2x10^{10}/cm^2)$ on 4 inch oxidized wafer. Uniform poly-C growth, with less than 20% thickness variation, was realized on 4 inch size wafer using MPCVD method with 3 kW microwave power. Low-resistance contact between poly-C and titanium was realized by adding a thin highly doped poly-C interlayer.

The dependence of poly-C piezoresistive GF on film resistivity and grain size were studied in detail using cantilever beam method. Optimized poly-C process, with resistivity in the range of 20 to 80 Ω^* cm and average grain size of 0.8 µm, was chosen for piezoresistive sensor application. This process yielded high GFs in the range of 30-70.

Cochlear probe is a device that can be implanted into a person's cochlea and deliver sound signal to the auditory nerves of deaf people. The position sensors on the probe are critical to determine the probe insertion position accurately. Poly-C position sensor was successfully integrated into two generations of cochlear probes and demonstrated its high piezoresistive GF of 44 for the first time. Design, fabrication and characterization of the poly-C sensors were accomplished with several improvements, such as high microwave power, contact interlayer, short resistor and large grain size. This work demonstrated a successful integration and promising application of poly-C technology with the silicon based microsystems.

To

my parents and my wife

ACKNOWLEDGMENTS

I would like to thank my whole family for their patience, understanding and support during this study. I owe my greatest thanks to my parents for their hard work and sacrifice so I can be where I am today. Special thanks go to my dear wife Niya Gu for her understanding, support and sacrifice during the long time of study. Thanks also go to my brothers, Huaxing and Jianxing, and all my other relatives for their encouragement.

I would like to thank my advisor, Dr. Dean Aslam, for his encouragement, guidance and financial support throughout this research. I also thank my committee members, Dr. Reinhard, Dr. Hogan, Dr. Naguib and especially Dr Kensall Wise for the advice and support. Thanks go to the members of our research group for their help and discussion in particular, Xiangwei, Nelson and Yang. Thanks also go to the faculties, stuffs and friends of the WIMS center at University of Michigan who gave me great help for the probe fabrication.

TABLE OF CONTENTS

LIST OF TABLESix
LIST OF FIGURESx
1 Research Motivation and Goals1
1.1 Introduction1
1.2 Objective of this Work
1.3 Overview of this Thesis6
Chapter 2 Background7
2.1 Introduction
2.2 Diamond Properties and Technologies7
2.2.1 Diamond Properties7
2.2.2 Diamond Seeding9
2.2.3 Poly-C Growth11
2.2.4 Poly-C Doping14
2.2.5 Poly-C Metallization17
2.2.6 Poly-C Patterning18
2.3 Theory of Piezoresistivity
2.3.1 Definition of Gauge Factor
2.3.2 Single Crystalline Materials
2.3.3 Polycrystalline Materials
2.4 Diamond Piezoresistivity
2.5 Cochlear Implant Probe

2.6 Summary	
Chapter 3 Poly-C Film Technologies	35
3.1 Introduction	35
3.2 Diamond Seeding	35
3.2.1 DPLW Spin-Coating	36
3.2.2 Seeding Density and Uniformity	
3.3 Film Growth Using MPCVD	42
3.4 Doping and Surface Conductivity	49
3.5 Metallization	54
3.6 Patterning	57
3.7 Summary	60
Chapter 4 Testing of Poly-C Piezoresistivity	61
4.1 Introduction	61
4.2 The Cantilever Beam Method	61
4.3 Sample Preparation	64
4.4 Piezoresistive Results	68
4.5 GF Dependence on the Film Properties	70
4.6 Summary	71
Chapter 5 Poly-C Sensor for 2 nd Generation Cochlear Probe	72
5.1 Introduction	72
5.2 Design and Fabrication of the Probe	72
5.2.1 Technology for Poly-C Process Integration	72
5.2.2 Design of the 2 nd Generation Probe	75

5.2.3 Fabrication of the 2 nd Generation Probe	'6
5.3 Testing of the 2 nd Generation Probe	31
5.4 Summary8	34
Chapter 6 Poly-C Sensor for 3 rd Generation Cochlear Probe	16
6.1 Introduction	\$6
6.2 Design of the 3 rd Generation Probe	36
6.3 Fabrication of the Probe9)0
6.4 Testing of the Probe9)5
6.4.1 Low-resistance poly-C contact9	95
6.4.2 Testing of the Piezoresistive GF10)0
6.5 Summary10)2
Chapter 7 Summary and Future Research10	13
7.1 Summary of Contributions)3
7.1.1 Results reported for the first time10)3
7.1.2 Other significant contributions10)4
7.2 Future Research)5
APPENDIX	6
BIBLIOGRAPHY11	9

LIST OF TABLES

2.1	Comparison of diamond properties with other semiconductors	8
2.2	Comparison of different seeding methods on seeding density, substrate material and surface morphology	10
2.3	Comparison of different CVD methods for poly-C growth	13
2.4	Gauge factors for various materials in the longitudinal direction	20
2.5	Piezoresistive property of poly-C reported by different groups	28
3.1	Typical MPCVD poly-C growth parameters	43
4.1	Comparison of DPLW and DPR seeding methods	63
5.1	Growth conditions and properties poly-C films for the 2nd generation probe	75
6.1	Comparison of the 2 nd and 3 rd generation cochlear probe	85
6.2	Masks used for the 3 rd generation cochlear probe with poly-C sensors	87
6.3	Detail growth parameters of poly-C film	89

LIST OF FIGURES

1.1	Overview of the research tasks and contributions in this thesis	5
2.1	Atomic C-H-O diamond growth phase diagram indicating the diamond growth domain	12
2.2	a) Conductivity data of boron doped poly-C at different temperature;b) Activation of boron doping with doping concentration and temperature	16
2.3	Contact resistivities between poly-C and different metals from literatures	17
2.4	Simplified geometric drawing for the definition of gauge factor	20
2.5	Simplified sketch of silicon valence band diagram under (a) zero stress and (b) uniaxial tension	22
2.6	Schematic of the normal and shear stress in three directions	24
2.7	Illustration of a typical cochlear prosthesis system	30
2.8	Design sketch of a silicon cochlear probe with strain gauges	31
3.1	SEM of Diamond seeding on oxide silicon wafer with different DPLW spin speed after 5 minutes deposition	35
3.2	Relation between diamond seeding densities on oxide silicon wafer and different DPLW spin speeds	36
3.3	SEM results of DPLW spin seeding with high speed multiple spin. a.) spin at 3000rpm twice; b.) spin at 3000rpm three times; c.) spin at 4000rpm twice; d.) spin at 4000rpm three times	37
3.4	DPLW spin seeding density distribution at different places on 4 inch wafer with different spin options	38
3.5	Optical picture and schematic of the MPCVD diamond growth system	41
3.6	Characteristics of the poly-C film seeded by DPLW spin method. a) Front surface of the film; b) 60 degree view of the film cross section; c) the AFM image of the surface; d) Raman spectrum of the DPLW seeded poly-C film	42
3.7	Dependence of the poly-C growth rate on the substrate temperature for growth with CH_4/H_2 ratio of 1.5/100	43

3.8	AFM images of the poly-C film grown at 700 °C on a 4 inch wafer	45
3.9	Radial distributions of normalized poly-C film thickness at different deposition conditions and the Raman spectra of different samples	46
3.10	Dependence of poly-C film conductivities on the doping levels	48
3.11	As-grown and annealed conductivities of poly-C films (1µm) deposited at 700 °C with different doping levels	51
3.12	I-V curve of contact between poly-C film and Ti/Au film after anneal. Inset: the schematic of Kelvin Bridge for contact measurement	53
3.13	Resistivity dependence of the annealed contact resistivity between poly-C film and Ti/Au film	54
3.14	SEM images of poly-C structures prepared by the ECR plasma etch: a) the free standing poly-C finger structure and, b) single grain piezoresistor on large grain poly-C plate	57
4.1	Piezoresistive testing stage using cantilever beam method	60
4.2	Schematic diagram of the cantilever beam method used for piezoresistive measurement	61
4.3	SEM pictures of a) DPLW seeding; b) poly-C film (with thickness of 2 μ m) seeded by DPLW; c) DPR seeding; d) poly-C film (with thickness of 2 μ m) seeded by DPR.	64
4.4	Layout of the two masks (for poly-C and contact metal) designed for testing the piezoresistors.	65
4.5	a) SEM of the 400/20 μ m piezoresistor with 4-contact; b) SEM of the Kelvin Bridge with gold wire bonding	65
4.6	Relation between resistance change and strain for poly-C film prepared by the DPR seeding	66
4.7	Relations between the GF and the boron doped resistivity for both DPLW and DPR seeded poly-C films	67
5.1	Process flow for integrating poly-C sensor into Si-based microsystems	71

5.2	Cross-sectional profile of the cochlear implant probe with poly-C sensors	72
5.3	Thickness distribution of poly-C film on the 4 inch wafers	76
5.4	Released 2 nd generation cochlear implant probe with poly-C position sensors	78
5.5	Testchip on the 2 nd generation probe wafer for process characterization	80
5.6	Sketch of the testing structure for probe position sensing and the measured gauge factor result	81
6.1	Cross-sectional view the 3 rd generation cochlear probe with poly-C piezoresistive position sensors	86
6.2	Process flow of the 3 rd generation cochlear probe with poly-C piezoresistive position sensor	88
6.3	Change of the sheet resistance of poly-C film vs. the dry etch time	90
6.4	Overview of the fabricated 3rd generation cochlear implant probe with poly-C position sensors	92
6.5	Detail growth parameters of poly-C film	93
6.6	Schematic of the Kelvin Bridge used for testing contact resistivity	94
6.7	SEM pictures of the Kelvin Bridge and close view of the poly-C surface	96
6.8	I-V curve of the contacts with/without highly doped interlayer	97
6.9	SEM pictures of the lightly doped poly-C piezoresistor with highly doped contact areas	98
6.10	GF of poly-C sensor on the cochlear probe achieved by measuring the resistance dependence on strain	99

Chapter 1

Research Motivation and Goals

1.1 Introduction

The US market for sensor products (sensors, transducers and associated housings) is projected to increase 15% per year from \$5.9 billion in 2000 to \$13.4 billion in 2006, and the world market for sensors is expected to reach US \$ 50-51 billion by 2008 [1]. The North American market for silicon based piezoresistive pressure sensors stood at about \$284.6 million in 2001 [2]. Since the pressure sensors cannot be sealed in most situations, the sensor material without passivation will be exposed to the environment. The operation of silicon pressure sensors in chemically harsh, high temperature environments is limited by leakage current and corrosion. Thus, the ultrahigh piezoresistivity and chemical inertness of polycrystalline diamond (poly-C) thin film make it a promising piezoresistive material better than silicon especially in harsh environment and high temperature applications [3, 4].

The presence of unique sp³ C-C bonds in the diamond lattice leads to its unique mechanical, chemical, optical and thermal properties not matched by any other known material. Consequently, diamond becomes a unique material for a number of applications including micro-electro-mechanical systems (MEMS) and wireless microsystem, especially at high temperatures and in harsh environments. However, the difficulty to

fabricate the sp³ C-C bonds, which delayed the production of chemical vapor deposition (CVD) of diamond, is now causing a delay in the development of a reliable and economical diamond micro-fabrication technology that is compatible with conventional microsystems/MEMS technologies.

A detail study and optimization of the poly-C film fabrication technologies, including seeding, growing, doping and patterning, is needed for an optimum integration with the silicon based microsystems.

The piezoresistive gauge factor (GF) of poly-C and related piezoresistive sensors have been reported by several groups [3, 5-8] but with large variations in the piezoresistive GF; typically in the ranges of 8 – 100 [5-7], 500 – 3200 [3] and 4000 [8] for poly-C inter-grain, single crystal diamond and poly-C intra-grain, respectively. Normally, the high GF values in the prior studies were achieved from films with resistivities over 100 Ω^* cm, which are impractical for sensor application due to the high film resistances and high noise level. More research on the piezoresistivity of poly-C film (in the doped region with resistivity from 10 to 100 Ω^* cm) is needed to optimize the film fabrication parameters for practical sensor applications.

In this work, poly-C films with their ultra-high piezoresistive sensitivity and biocompatibility were chosen as the position sensors for the cochlear prosthesis project in the Engineering Research Center for Wireless Integrated MicroSystems (WIMS ERC) funded by National Science Foundation (NSF). Cochlear prostheses have been used as an enabling technology to help deaf people in hearing by electrically stimulating the auditory nerve cells with the implanted electrode. The position sensors are developed to determine the placement of the cochlear electrode array within the cochlea both during

insertion and post-operation. Incorporating poly-C sensors into the probe fabrication process and achieving high sensitivity will demonstrate a successful technology integration and sensor application for poly-C thin film.

1.2 Objective of this Work

The goal of this work is to study the poly-C thin film technology and its piezoresistivity for application as the position sensor in cochlear implant probe. It aims to develop an optimum poly-C technology for high piezoresistivity and good integration with silicon based microsystem. Then the application of poly-C piezoresistive sensor was demonstrated by integrating poly-C into the cochlear probe as the position sensor. In a summary, the goals of this dissertation research will focus on:

- Fabricating and characterizing poly-C thin films on 4 inch oxidized silicon wafer using IC-compatible processes. Developing and optimizing the crucial poly-C technologies including seeding, growing, doping and patterning for the system integration.
- 2) Studying the piezoresistivity of poly-C thin films prepared under different parameters including grain size and doping levels. Optimizing the poly-C fabrication condition to reach a compromise between high gauge factor and low resistance for sensor application.
- Integrating of the poly-C piezoresistive position sensor into the silicon based cochlear implant probe and achieve good integration and high sensitivity.

The major accomplishments and contributions to the scientific community reported in this thesis were summarized in Figure 1.1. Based on the research strategy of NSF, these contributions can be divided into three levels: fundamental research, enabling technology and system level.

	SENSOR APPLICATION FOR COCHL	EAR PROSTHESIS	
Poly-C Film Te	chnology	Application of Poly-C	Piezoresistive Sensor for Cochlear Prosthesis
Tasks	Expected Unique Contributions	Tasks	Expected Unique Contributions
1. Diamond seeding	Non-destructive and uniform high density seeding on 4 inch oxidized Si wafers	6. Piezoresistivity of poly-C	Demonstrate the dependence of the poly-C piczoresistivity on film properties
 Large area poly- C growth 	Efficient poly-C growth over large area with low microwave power	7. Poly-C integration for the 2 nd gen. cochlear probe	First time integration and application of poly-C piezoresistive sensor in biological microsystem
3. Poly-C doping & surface conductivity	Quantitatively calculate the surface conductivity using numeric fit	8. Test the 2 nd gen.	Measure the electrical and piezoresistive properties of poly-C sensor on the probe
4. Low contact resistance	New method of lowering the contact resistance between poly-C and Ti metal	9. Design the 3 rd gen. probe	Optimize the design of 3^{rd} gen. probe based on the results from the 2^{nd} gen. probe test
5. Technology integration	Develop a poly-C technology for sensor application with Optimized processes	10 Fabrication of the 3 rd gen. probe	Make 4 masks for poly-C sensors and run a 10- mask fabrication process for the 3 rd gen. probe with poly-C sensors
Tasks related	to: research	11 Test the 3 rd gen. probe	Electrical and piezoresistive test for good integration and high sensitivity
Enabling tech	nology		

Figure 1.1 Overview of the research tasks and contributions in this thesis

System level

1.3 Overview of this Thesis

This thesis presents the development and characterization of the technology of poly-C piezoresistive position sensor and its integration with the silicon based cochlear implant probe. Chapter 2 introduces the theory of piezoresistivity and the diamond piezoresistivity. It also summarizes the previous work on fabrication technology of polycrystalline diamond thin film. A brief introduction about the cochlear implant probe is also presented. Chapter 3 talks about the poly-C thin film technology used for the piezoresistive sensor devices. The details of poly-C film seeding, growing, doping and patterning are presented with several unique contributions by this thesis research. Chapter 4 describes the measurement techniques and the equipment set up used for piezoresistivity study. It discusses the measurement results of piezoresistive gauge factor and its dependence on both the doping levels and film grain size. Then, in chapter 5, the initial attempt of integrating diamond piezoresistive sensor into the 2nd generation cochlear implant probe is discussed. Chapter 6 shows the detail design, fabrication and testing of the optimum 3rd generation cochlear probe with poly-C sensors. Last chapter summarizes the work and results of this dissertation and presents suggestions for future related research.

Chapter 2

Background

2.1 Introduction

This chapter presents an introduction to the previous work on fabrication technology of poly-C thin film such as seeding, growing, doping and patterning. It also explains the piezoresistivity with mathematical derivation of the piezoresistive coefficients and gauge factor. An overview of the recent progress in diamond piezoresistive study and related sensors is given. A brief introduction to the concept of the cochlear implant probe and its recent progress are presented also.

2.2 Diamond Properties and Technologies

2.2.1 Diamond Properties

Diamond is comprised of covalently bonded carbon atoms in a diamond cubic crystal structure. In the diamond lattice, each carbon atom is bonded with its four neighbor atoms using covalent bonds with hybrid sp³ atomic orbits that give it hardness and strength. The properties of both natural diamond and poly-C are summarized and compared with other semiconductor materials in Table 2.1. Diamond has long been

known to be the strongest and the hardest of all materials. As we can see its Young's modulus is about five times higher than silicon, which makes it a promising material for the applications as mechanical transducers. The thermal conductivity of diamond at room temperature, about 20 W/cm^{*}K, is higher than that of any other materials, which enables a quick dissipation of heat as heat sink.

Properties	Si	GaAs	SiC	Natural diamond	Poly-C
Density (g/cm ³)	2.329	5.317	3.216	3.52	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	800-1180
Poisson's Ratio	0.22-0.24	0.31-0.32		0.1-0.21	0.07-0.148
Lattice constant (Å)	5.43	5.65	4.36	3.57	3.57
Band Gap (eV)	1.12	1.42	3.0	5.45	
Carrier mobility Electron (cm ² /V*s) Hole(cm ² /V*s)	1450 500	8500 400	400 50	1800-2000 1600-2100	23 10-1000
Dielectric Constant	11.7	10.9	10	5.7	6.5
Breakdown Voltage (x10 ⁶ V/cm)	0.37-0.5	0.6	2-3	4-20	0.1-1
Intrinsic resistivity (Ω*cm)	1x10 ³	1x10 ⁸		1x10 ¹⁶	10 ⁶ -10 ¹⁴
Thermal conduc- tivity (W/cm*K)	1.5	0.5	5	20	4-20
Thermal expansion coef. (x10 ⁻⁶ /°C)	2.6	5.9	4.7	1.1	2.6

Table 2.1 Comparison of diamond properties with other semiconductors [9].

Electrically, pure diamond is an excellent insulator. The band gap associated with the indirect transition between the valence and the conduction band is 5.45 eV. If massive production of large scale crystalline diamond becomes cheap enough, it will be a good candidate for high temperature (large band gap), high speed (high carrier mobility), and high power (high thermal conductivity) electronic device in the future.

Based on the special properties of poly-C as shown in Table 2.1, a lot of work has been reported on the poly-C applications such as temperature sensors and heaters [10, 11], piezoresistive sensors [3, 4], gas/chemical sensors [12, 13], radiation sensors [14, 15], field emission and optoelectronics devices [16-18], MEMS packaging [19], and RF MEMS resonators [20, 21].

As a new emerging technology in microsystem application, the properties of the poly-C films varies much upon different preparation parameters and different synthesizing system, and the applications of poly-C films in sensors are far from being a standard technology like silicon. Efforts are needed to optimize the poly-C film seeding, fabrication and patterning procedure to generate a practical, IC compatible process for sensor applications. This section presents an overview of the recent progress in CVD diamond film technology including the seeding, growing, doping and patterning.

2.2.2 Diamond Seeding

The CVD growth of poly-C films requires a pretreatment step to generate seeds (or nuclei) on the substrate before the growth begins. Surface abrasion [22-24], sonication of diamond powder loaded solution [25], bias enhanced nucleation (BEN) [26-28], spinning

of diamond-powder-loaded photoresist (DPR) [29] and spraying of diamond-loaded fluids [30, 31] are typically used for the pretreatment as shown in Table 2.2.

Table 2.2 Comparison of different seeding methods on seeding density, substrate material and surface morphology.

Seeding Method	Sonication /Abrasion	BEN	DPR	Spray/paint	DPLW spin
Seeding Density	Up to 10 ¹⁰	Up to 10 ¹¹	~10 ⁸	Up to 10 ¹¹	10 ⁸ ~10 ¹¹
Substrate Selectivity	Most dielectric & metal	Conductive Si or Metal,	Most dielectric & metal	Most dielectric & metal	hydrophilic Surface like (SiO ₂ & Si ₃ N ₄)
Surface Affection	scratch surface, not good for thin film	No damage	No damage	No damage	No damage
Uniformity & Controllability	not uniform	Uniform & repeatable on whole wafer	Uniform & repeatable on whole wafer	Not uniform	Uniform & repeatable on whole wafer

Most of the previous seeding methods focus on the seeding on bare Si wafer or a tungsten surface and very few have reported diamond seeding on a dielectric surface. For the electrical application in microsystem, it is common to insulate a conductive film using dielectric layers (SiO₂ or Si₃N₄) and make connection through contact holes or vias. So having the ability to achieve nondestructive, controllable, uniform and high density diamond seeding on dielectric layer is an important step for poly-C sensor applications.

The last column in Table 2.2 lists the new method we developed for seeding on dielectric substrate, which will be discussed in Chapter 3.

2.2.3 Poly-C Growth

The growth of diamond by high pressure techniques was well established since the 1950s when GE researchers succeeded in transforming graphite to single crystalline synthetic diamond [32]. Typically synthetic diamond is fabricated at temperatures around 4237 K and a pressure near 200 kbar. The high pressure high temperature (HPHT) conditions make it costly and only suitable for production in the form of small pieces, grit and powders.

Interest in diamond was renewed by successful CVD growth from hydrocarbon mixtures in a lower pressure and temperature region. The CVD technique is based on decomposition of carbon-containing precursor molecules (typically CH₄) diluted in H₂ gas. Addition of atomic hydrogen allowed for preferential etching of the sp² bonding (graphite) from the film and helped to yield high quality sp³ carbon structure (diamond). Oxygen, in the form of O₂, CO or CO₂, has been added sometimes to get various film properties. Bachmann and co-authors compiled numerous published CVD recipes and constructed the C/H/O phase diagram plotted in Figure 2.1 [33]. The diagram shows that all successful diamond growth results appear within a well defined region.



Figure 2.1 Atomic C-H-O diamond growth phase diagram indicating the diamond growth domain [33].

New developments in nanocrystalline diamond growth was recently reported by D. M. Gruen from Argonne national Lab [34]. Most reports of the growth of these films describe using a microwave plasma CVD reactor and gas mixture of 1%CH₄ in Ar, usually with addition of 1–5% H₂. This method can produce films with much smaller grain sizes (2-5 nm) and smoother surface than the regular hydrogen based growth.

Nowadays, poly-C can be grown using a variety of CVD methods including microwave plasma CVD (MPCVD)[35, 36], hot filament CVD [37, 38], radio frequency (RF) CVD and dc-arc jet CVD [39] as seen in Table 2.3. However, MPCVD is the most widely used technique for electronic and optical applications because of its high efficacy to produce high quality film, large substrate size, less contamination and good controllability.

Methods	HFCVD	MPCVD	DC-arc Jet CVD	Combustion Synthesis	RFCVD
Growth Rate (µm/hr)	0.1-10	0.1-10	30-150	4-40	<0.1
Substrate Temp. (°C)	300-1000	300-1200	800-1100	600-1400	700-1200
Growth Area (cm ²)	5-900	5-100	<2	<3	100
Advantages	Simple, large area	Quality, stability	High rate, good quality	Simple, high rate	High quality
Disadvantages	Contaminations, fragile filament	Rate	Contaminations, small area	Small area	Low rate, expensive

Table 2.3 Comparison of different CVD methods for poly-C growth.

The reaction chamber in a typical microwave plasma system is composed of a quartz tube which is placed inside the microwave chamber and a substrate holder which is positioned inside the tube. A plasma of a carbon carrier gas and hydrogen mixtures, was formed and diamond was deposited at a pressure of 10 -100 torr, a substrate temperature of 600 - 1000 °C, and methane concentrations of 0.2-5% in hydrogen.

Most of the MPCVD growths focus on the small size samples for lab test. Some articles in the literatures reported the deposition of diamond on large scale substrates (4 to 6 inch) and discussed the microwave power, growth and uniformity. V.G. Ralchenko reported a growth on 60mm substrate using a 5 kW microwave power with 10% thickness variation [40]. Y. Ando et al reported a growth on 6 inch substrate with 30% thickness variation using 15-60 kW microwave power [41]. Research on improving the power efficiency and film uniformity on 4 inch substrate will be interesting for reducing production cost.

2.2.4 Poly-C Doping

Accurate control of the doping level and resistivity is particularly important for poly-C thin film electrical applications in sensors. Different boron containing compounds typically used for p-type doping are pure boron powder [42], boron trioxide (B_2O_3) [43], diborane (B_2H_6) [44, 45] and trimethylboron (B(OCH₃)₃, TMB) [46]. Although boron ion implantation has been used for diamond doping [47, 48], its effectiveness has been limited by the implant damages and very low diffusion ability of boron in poly-C [49]. In situ doping with the trimethylboron has become a preferred source because the TMB gas, diluted in hydrogen, is safer to use and more controllable than other boron compounds

[46]. Figure 2.2.a shows the conductivity of poly-C film under different boron doping concentrations and different temperatures [50]. Figure 2.2.b shows the activation energy and hole concentrations of boron doped poly-C films under different doping levels and temperatures [51].





Figure 2.2 a)Conductivity data of boron doped poly-C at different temperature [50].

Continue Figure 2.2 b) Activation of boron doping with doping concentration and temperature [51].

It has been found that CVD diamond films grown in a hydrogen plasma have a thin hydrogenated surface layer, which becomes conductive after exposure to the atmosphere. Annealing in vacuum at 600 °C can remove this conductive layer due to the hydrogen desorption effect [52, 53]. A detail study on the surface conduction can give better view about how it affects the film resistivity especially for lightly doped films.

2.2.5 Poly-C Metallization

Low-resistivity ohmic contact between poly-C and metal thin films is essential for any diamond electronic device. The type of metal contact on poly-C (ohmic or rectifying) depends not only on the choice of metals but also on the doping concentration, annealing and other parameters.



Figure 2.3 Contact resistivities between poly-C and different metals from literatures [54-57].

Titanium and chromium (with co-deposited gold on top of them to prevent oxidation) are most widely used metals because they can form carbides with poly-C and yield good conductivity after anneal. Werner et al. had demonstrated the Al (with 1% Si) film also gave good contact with poly-C by forming silicon carbide [54]. For Ti/Au, Cr/Au and Al/Si films, a vacuum annealing at 400 °C to 600 °C for 20 minutes is enough to achieve stable ohmic contacts [58, 59]. Figure 2.3 shows the reported contact resistivity (also known as specific contact resistance) as a function of poly-C doping levels from literatures [54-57]. The contact resistivity is defined as the total contact resistance times the size of contact area.

For the application as piezoresistor, it requires the poly-C film to be doped lightly to get high piezoresistive sensitivity and contact resistance will become a problem in these situations. More studies are needed for reducing the contact resistance for lightly doped poly-C films.

2.2.6 Poly-C Patterning

For integrating poly-C devices into Si-based microsystems, patterning of poly-C films using techniques consistent with silicon processing is required. As the wet etching poly-C is impractical, the only patterning techniques are in-situ pattering and dry etching. The insitu patterning techniques rely on selective seeding which has been achieved using different methods including diamond power-loaded photoresist (DPR) employing standard lithography [29, 60], selective seeding using SiO₂ as a mask [61, 62] and direct spray writing [30]. However, in all 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. The dry etching of poly-C, which uses different active gas species such as oxygen, argon, hydrogen, CF_4 and SF_6 with metal or SiO₂ masks [63-65], seems to be excellent choice for poly-C patterning. Most researches have used conventional reactive ion etching (RIE) method where the gas species are excited by the RF power [65, 66]. Dry etching using electron cyclotron resonance (ECR) assisted microwave plasma at low substrate temperatures and pressures has led to very clean structures with small feature sizes and sharp edges [67, 68].

2.3 Theory of Piezoresistivity

2.3.1 Definition of Gauge Factor

The piezoresistive effect was first discovered in 1856 by Lord Kelvin using copper and iron wires. The piezoresistive effect in semiconductors was found to be much larger than metals by C. S. Smith in the mid 1950's with germanium and silicon [69].

Piezoresistivity of a material is the dependence of electrical resistivity on strain and normally is 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 ε . Typical gauge factors for various materials in the longitudinal direction are shown in Table 2.4.

Material	Descriptions	Gauge Factors (GF)
Platinum [70]	100%	6.1
Copper [70]	100%	2.6
Silver [70]	100%	2.9
Platinum/Tungsten [70]	92% Pt, 8 % W	4.0
Single Crystal Silicon [69], [71]	p-type	100 – 175
	n-type	-133
Poly-Silicon [72]		2 – 20
Germanium [69]	p-type	48 - 102
Silicon Carbide [73]	n-type	-26 to -994
Poly-C [4] [74] [75]	p-type	5-1000

Table 2.4 Gauge factors for various materials in the longitudinal direction.



Figure 2.4 Simplified geometric drawing for the definition of gauge factor.

For a material shown in Figure 2.4, the longitudinal resistance R of a uniform conductor with a length L, cross-sectional area A, and resistivity ρ is given by:

$$R = \rho \frac{L}{A}.$$
 (2.1)

Differentiating Eq. 2.1 and dividing by the total resistance R leads to the fractional change of resistance:

$$\frac{\Delta R}{R} = \frac{\Delta \rho}{\rho} + \frac{\Delta L}{L} - \frac{\Delta A}{A}.$$
 (2.2)

The longitudinal strain

$$\varepsilon_l = \frac{\sigma_l}{Y} = \frac{\Delta L}{L}, \qquad (2.3)$$
where σ_l is longitudinal stress and Y is the Young's Modules of the material. The gauge factor in the longitudinal (strain is parallel to current) direction can be written as:

$$GF_{l} = \frac{(dR/R)_{l}}{\varepsilon_{l}} = 1 + 2\upsilon + \frac{1}{\varepsilon_{l}} \left(\frac{\Delta\rho}{\rho}\right)_{l}, \qquad (2.4)$$

where v is the Poisson ratio. It can be seen from Eq. 2.4 that the strain sensitivity of any material is due to two factors, i.e., (i) the change in the dimensions of the conductor, and (ii) the change in resistivity.

For the transverse direction where the current flow is perpendicular to the strain direction, the gauge factor can be written as [76]:

$$\left(GF\right)_{t} = \left(\frac{\Delta R}{R}\right)_{t} \frac{1}{\varepsilon_{t}} = \left(\frac{\Delta \rho}{\rho}\right)_{l} \frac{1}{\varepsilon_{t}} - 1, \qquad (2.5)$$

For some semiconductor materials like silicon, silicon carbide or diamond, the GF is much higher due to the strain dependence of the resistivity caused by energy band deformation and the first two parts in Eq. 2.4 and the second part in Eq. 2.5 can be ignored.

2.3.2 Single Crystalline Materials

For the single crystalline semiconductor like silicon and diamond, the piezoresistive effect is anisotropic and the piezoresistivity need to be represented using the piezoresistive coefficients. Silicon will be chosen as the example to explain the principle of piezoresistivity in semiconductors [76].



Figure 2.5 Simplified sketch of silicon valence band diagram under (a) zero stress and (b) uniaxial tension.

The piezoresistive effect in silicon crystal was explained by the many-valley model developed by Herring [77]. As shown in Figure 2.5(a), there are two valence sub-bands, heavy- and light-hole bands, degenerated at k=0, and a split-off band 40 meV below these bands at zero stress. When stress is applied, the valence bands split and redistribution of holes take place. The application of uniaxial stresses to the crystal lifts the cubic symmetry and removes the degeneracy at k=0 of the valence band [9]. This causes the sub-bands to be shifted relative to each other and results in transfer of carriers between heavy holes and light holes. Under uniaxial tensile stress, the heavy-hole subband moves up and the light-hole subband moves down as shown in Figure 2.5(b). This leads to a result of more carriers with low mobility and less carriers with high mobility. Since the conductivity is proportional to the mobility, the resistance increases with the tensile stress [76].

For the 3-dimensional anisotropic crystal, the generalized relationship between electrical field E, resistivity ρ and current i can be written as:

$$\begin{bmatrix} E_{1} \\ E_{2} \\ E_{3} \end{bmatrix} = \begin{bmatrix} \rho_{1} & \rho_{6} & \rho_{5} \\ \rho_{6} & \rho_{2} & \rho_{4} \\ \rho_{5} & \rho_{4} & \rho_{3} \end{bmatrix} \cdot \begin{bmatrix} i_{1} \\ i_{2} \\ i_{3} \end{bmatrix}.$$
 (2.6)

The Resistivity coefficients are also a function of strain imposed on the material:

$$\begin{bmatrix} \rho_{1} \\ \rho_{2} \\ \rho_{3} \\ \rho_{4} \\ \rho_{5} \\ \rho_{6} \end{bmatrix} = \begin{bmatrix} \rho \\ \rho \\ \rho \\ 0 \\ 0 \\ 0 \end{bmatrix} + \begin{bmatrix} \Delta \rho_{1} \\ \Delta \rho_{2} \\ \Delta \rho_{3} \\ \Delta \rho_{4} \\ \Delta \rho_{5} \\ \Delta \rho_{6} \end{bmatrix}.$$
(2.7)



Figure 2.6 Schematic of the normal and shear stress in three directions.

Due to the symmetry of silicon crystal cubic structure, the resistivity change can be written using both normal and shear stress (shown in Figure 2.6) in all three directions:

$$\frac{1}{\rho} \begin{bmatrix} \Delta \rho_1 \\ \Delta \rho_2 \\ \Delta \rho_3 \\ \Delta \rho_4 \\ \Delta \rho_5 \\ \Delta \rho_6 \end{bmatrix} = \begin{bmatrix} \pi_{11} & \pi_{12} & \pi_{12} & 0 & 0 & 0 \\ \pi_{12} & \pi_{11} & \pi_{12} & 0 & 0 & 0 \\ \pi_{12} & \pi_{12} & \pi_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & \pi_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & \pi_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & \pi_{44} \end{bmatrix} \cdot \begin{bmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \tau_1 \\ \tau_2 \\ \tau_3 \end{bmatrix}.$$
(2.8)

The above analysis has so far assumed that the piezoresistive coefficients are

aligned to the primary axes of the crystal silicon. But in many instances the position of piezoresistors may not coincide with the coordinate system. Therefore, we need to develop a transformation between the old coordinate system (x, y, z) (i.e., the system that is aligned to the Si crystal structure) and the new system (x^*, y^*, z^*) (the system that is aligned with the longitudinal direction of the piezoresistor):

$$\begin{bmatrix} x * \\ y * \\ z * \end{bmatrix} = \begin{bmatrix} l_1 & m_1 & n_1 \\ l_2 & m_2 & n_2 \\ l_3 & m_3 & n_3 \end{bmatrix} \cdot \begin{bmatrix} x \\ y \\ z \end{bmatrix}, \quad (2.9)$$

where l_i , m_i and n_i (i=1,2,3) are the direction cosines of the transformation [78]. (l1, m1, n1) is the set of direction cosines between the longitudinal resistor direction and the crystal axis, and (l2, m2, n2) is the set of direction cosines between the transverse resistor direction and the crystal axis.

For the longitudinal direction, the gauge factor can be written as

$$GF_{l} = \left(\frac{\Delta\rho}{\rho}\right)_{l} \frac{1}{\varepsilon_{l}} = \pi_{l}Y . \quad (2.10)$$

The longitudinal piezoresistive coefficient π_l equals to

$$\pi_{l} = \pi_{11} - 2(\pi_{11} - \pi_{12} - \pi_{44})(l_{1}^{2}m_{1}^{2} + m_{1}^{2}n_{1}^{2} + n_{1}^{2}l_{1}^{2}). \quad (2.11)$$

For the transverse direction, the gauge factor can be written as

$$GF_t = \left(\frac{\Delta\rho}{\rho}\right) t \frac{1}{\varepsilon_t} = \pi_t Y . \quad (2.12)$$

where the transverse piezoresistive coefficient equals to

$$\pi_t = \pi_{12} + \left(\pi_{11} - \pi_{12} - \pi_{44}\right) \left(l_1^2 l_2^2 + m_1^2 m_2^2 + n_1^2 n_2^2\right).$$
(2.13)

2.3.3 Polycrystalline Materials

In general, the polycrystalline materials consist of crystalline grains interconnected by grain boundaries. Grains are considered as small single crystals with the same lattice and same energy band structure as single crystal silicon. The grain boundaries (GBs) are composed of disordered atoms and contains a large number of defects and dangling bonds which act as trapping states and/or segregation sites [79]. Trapping of holes creates a potential barrier at the boundary and a depletion region into the grains.

For polycrystalline semiconductor material, the resistivity of the film can be described by contribution from both the grain and grain boundary and is given by [80]

$$\rho = \frac{L - (2w + \delta)}{L} \rho_g + \frac{(2w + \delta)}{L} \rho_b, \quad (2.14)$$

where ρ , ρ_g and ρ_b are the resistivities of the film, grain and grain boundary, respectively, and δ is the grain boundary thickness. L and w are the lengths of the grain and depletion region, respectively.

From Eq. 2.14, the fractional change in resistivity per unit strain (gauge factor) can be written by:

$$GF = \frac{\Delta\rho}{\rho} \frac{1}{\varepsilon} = \frac{L - (2w + \delta)}{L} \frac{\Delta\rho_g}{\rho_g} \frac{1}{\varepsilon} + \frac{(2w + \delta)}{L} \frac{\Delta\rho_b}{\rho_b} \frac{1}{\varepsilon}$$
(2.15)

Normally it is believed that the piezoresistive effect of grain boundary is much lower than the grain crystal. The random orientations of the grains also lower overall piezoresistive effect of grain. This is consistent with the reported low GF of polysilicon compared to single crystalline silicon.

2.4 Diamond Piezoresistivity

Interest for diamond piezoresistive sensors grew with the first quantitative study of the piezoresistive gauge factor in homoepitaxial and polycrystalline diamond films [3]. Several groups have confirmed the observed large piezoresistive gauge factor for different diamond films.

For synthetic crystalline diamond, the GF, extracted from the work of Latsa et, al, was found to be higher than 10^3 [4]. The GF for homoepitaxial diamond, measured using a boron doped type 2a film epoxy-glued on a stainless cantilever beam, was reported to be at least 550, four times higher than the highest value of Si [4]. Sahli S. reported a high GF of 4000 from the intra-grain measurement on large size grain (75 µm) poly-C plate [8].

The piezoresistive property of poly-C was studied for different structures, doping levels and temperatures using cantilever beam, three-point bending fixture and differential pressure setups. Table 2.5 lists most data published on room temperature GF for CVD diamond along with the corresponding sample specifications.

The effect of GBs on the electrical properties of poly-C has been addressed by measuring and interpreting the effective electrical properties of CVD diamond. For the boron doped CVD diamond films, frequency conduction measurements for different doping levels showed activation energies in the range of 0.18 to 1.07 eV and identified

three conduction mechanisms namely conduction, hoping conduction and frequencydependent thermal emission of carriers from trap levels [9].

GF	ρ (Ω*cm)	substrate	Deposition	Doping	Measurement setup	
5	5		HFCVD H ₂ /CO/CH ₄ = 100/12/0.7	Boron powder		
10	20	SiO ₂			Glue film on oxidized Si cantilever beam [4]	
15	30					
50	225	Si				
100	300	Si	MPCVD H ₂ /CH ₄ =99/1	Boron Wafer	Film epoxied on AlN, stressed by 3-point bending [74]	
75	82	W				
9	20.8	AlN				
2.3	0.01				Contilouon hoom [91]	
5.4	2				Cantilever deam [81]	
1000	300	Undoped poly-C	MPCVD	B ₂ H ₆	Gas pressure system [75]	
10	3	Si		Boron Wafer		
70	101	Si	$\frac{1}{1} \frac{MPCVD}{H_0/CH_{1}=00/1}$		3-point bending [74]	
100	209	Si				
283*	0.27	Undoped poly-C	MPCVD	Boron powder		
4000**	0.1				Cantilever beam [8]	

 Table 2.5 Piezoresistive property of poly-C reported by different groups.

*/**: These value were inter(*) & intra(**)-grain measurement from very large grain

film (75µm) [8]

Based on the samples with listed resistivity, GF increases with the resistivity. The results also suggest that grain size, structure, substrate and processing may affect the Piezoresistivity, however, there is no systematic study of the GF-grain size relation yet.

The results listed in Table 2.5 demonstrate the feasibility and confirm the high sensitivity of boron doped poly-C piezoresistors. The current challenge for diamond piezoresistors is to deliver stable, reliable and reproducible sensor under the reasonable resistivity range and integrate them into the microsystems.

2.5 Cochlear Implant Probe

Cochlear prosthesis is a device that can be surgically implanted into a person's cochlea to stimulate it to cause hearing. As shown in Figure 2.7, it consists of a tiny receiver which is placed under the skin in the bony part behind the ear. The receiver connects to a bundle of metal wire electrodes (usually 22 now), which are implanted into the cochlea. A cochlear implant can be useful if the hearing loss is caused by problems with the cochlea (usually damaged hair cells) or where the loss is caused by problems with the middle ear that cannot be corrected. A person with a cochlear implant also wears a hearing aid like device that has a microphone, a processor and a transducer. The processor manipulates what the microphone hears and sends a signal to the transducer, which is usually worn just behind the ear. The transducer changes the signal from an electrical signal to an electromagnetic signal that can be received through the skin by the implanted receiver.

Over two million people in the United States suffer from the hearing impairment for which traditional hearing aids offer little or no help. Many of these individuals could benefit from the cochlear prosthesis. Worldwide, nearly 100,000 people have received cochlear implants with the implant devices primarily provided by three companies [82].



Figure 2.7 Illustration of a typical cochlear prosthesis system [82].

In spite of considerable recent progress, several problems continue to plague present implants. These include limited frequency range (due to the difficulties in achieving deep insertion), limited pitch discrimination (due to the small number of wires that can be accommodated in the cochlea (16-22)), and rather poor ability to distinguish individual conversations in a crowded room. While cochlear implant users can often communicate normally using the telephone, music is usually less than enjoyable. The surgical implant procedure today is still performed blindly without benefit of position feedback. Currently, no satisfactory non-invasive visualization technique exists for cochlear implants. Conventional X-rays, CT scans, and phase-contrast radiography provide limited solutions and are used primarily for pre-surgical or post-surgical evaluations [82]. While implants use rather sophisticated electronics and are powered from an external speech processor by inductive RF coupling, the wire electrode bundles are constructed largely by hand, are limited in performance, and are relatively expensive [83].



Figure 2.8 Design sketch of a silicon cochlear probe with strain gauges [84].

Thin-film silicon probe with electrode arrays [84] are being developed by researchers in the NSF Engineering Research Center for Wireless Integrated Microsystems (WIMS ERC), which was issued to University of Michigan, Michigan State University and Michigan Tech University under the direction of Dr. Kensall D. Wise. These probes are batch fabricated using bulk micromachining technology and aim to improve pitch perception by increasing the number of stimulating sites to as many as 128. In order to allow real-time visualization and accurate positioning of the arrays, a position-sensing system, which consists of a distributed series of piezoresistors along the insertion part of the cochlear probe, is being built into the arrays as shown in Figure 2.8. With the help of the position sensors, the sites should ideally be implanted deeply within the cochlea and should hug the interior wall of the scala tympani to minimize the distance to the receptors, lower the stimulation thresholds, reduce implant power, and increase frequency discrimination [83]. Due to the high piezoresistive sensitivity and biocompatibility, using a poly-C piezoresistor will help to improve the performance of the positioning system. Incorporating poly-C position sensors into the probe fabrication process and achieving high sensitivity will demonstrate a successful technology integration and sensor application for poly-C thin film.

2.6 Summary

This chapter reviews the poly-C thin film technology and the theory of piezoresistivity. The principle of poly-C piezoresistivity and related sensors are

summarized. Although poly-C piezoresistive sensors have been successfully fabricated, there still exists large scattering in the piezoresistive sensor response no real application in a complicated microsystem. The introduction to cochlear implant probe leads to a promising application of poly-C piezoresistive sensors in a silicon based cochlear implant probe.

-

Chapter 3

Poly-C Film Technologies

3.1 Introduction

One part of the work in this thesis focuses on developing the poly-C technologies that can lead to controllable high quality poly-C films for the sensor applications. This chapter reports the poly-C film technologies used for fabrication with emphasis on the new seeding method, the large area growth and the surface conductivity.

3.2 Diamond Seeding

For sensor applications, poly-C film needs to be grown on dielectric layer (SiO₂ or

Si₃N₄). Controllable and non-destructive seeding method with high seeding density is required on dielectric substrate for growing smooth, pinhole-free poly-C thin film. Different seeding methods have been reviewed in Chapter 2. In this work, we developed a new seeding method by spin-coating diamond power loaded water (DPLW) on the samples. The goals of studying this new seeding method are: i) Achieve controllable, repeatable, non-destructive and large scale (4 inch wafer) seeding on a dielectric (SiO₂ or Si₃N₄) surface using DPLW spin-coating.

ii) Study the relation between the seeding density and the spin speed.

iii) Study the relation between the seeding uniformity and the multiple-spin.

3.2.1 DPLW Spin-Coating

The diamond powder loaded water (DPLW) is hydrophilic on cleaned silicon dioxide surface and can spread on it and form a uniform thin film on the SiO₂ surface due to the following two reasons: a). The surface tension between SiO₂ and water (120mN/m) is much larger than the water surface tension (72mN/m) [85], and b). The SiO₂ surface partly terminated with H and F atoms (caused by the HF etching) is favorable to water due to the Coulomb attraction through high polarity of the Si-F bonding [86].

25 carat diamond powder, with an average size of 25nm, was mixed in 1000ml De-Ionized (DI) water to create the DPLW. Polyether Polyol is used as suspension agent. Ultrasonication was used to break the aggregated powders and improved the suspended powder density in water.

The 4" silicon wafer, with an oxide layer of 300nm on top of it, was cleaned with RCA procedure, which includes 10 minutes degrease clean (degrease solution includes H_2O_2 :NH₄OH:H₂O at a ratio of 1:1:5 at 50 °C) and 10 minutes demetal clean (demetal solution includes H_2O_2 :HCl:H₂O at a ratio of 2:1:8 at 50 °C). Then the wafer was dipped into the 5% HF water solution for 30 second, rinsed in DI water for 3 minutes and blown dry by nitrogen gas.

After the clean procedure, the sample wafer was put on spinner, and DPLW with 25nm size powder was dropped on the wafer to form a thin and uniform film. The wafer was spun at a specific speed for 30 second once the dropped DPLW covers the whole wafer. Different spin speeds were tested to study the relation between seeding densities and spin speeds. Multiple spin, which means repeating the DPLW spin procedure twice or 3 times on same wafer at relatively high spin speed, was also be studied to improve the seeding quality.



Figure 3.1 SEM of Diamond seeding on oxide silicon wafer with different DPLW spin speed after 5 minutes growth.

3.2.2 Seeding Density and Uniformity

The DPLW was spun on the oxidized silicon wafers with spin speeds in the range of 1000-4000 revolutions per minute (rpm) to optimize the seeding density. To get a continuous film with a thickness of about $0.6\mu m$ with very few pinholes, a seeding density of approximately 10^{10} nuclei/cm² is needed with these 25nm average size diamond seeds.

Figure 3.1 shows the SEM pictures of the seeding results with different DPLW spin speed. For these samples, poly-C was deposited for 5 minutes at 700 °C after the seeding. The seeding density was calculated from the SEM pictures using the statistical method as shown in the Figure 3.2.



Figure 3.2 Relation between diamond seeding densities on oxide silicon wafer and different DPLW spin speeds.



Figure 3.3 SEM results of DPLW spin seeding with high speed multiple spin. a.) spin at 3000rpm twice; b.) spin at 3000rpm three times; c.) spin at 4000rpm twice; d.) spin at 4000rpm three times.



Figure 3.4 DPLW spin seeding density distribution at different places on 4 inch wafer with different spin options.

The SEM pictures in Figure 3.1 show that, at low spin speeds, spin seeding gave high seeding densities but lots of the diamond powders aggregated together, which will reduce the effective seeding density and weaken the adhesion force between the diamond film and underneath silicon dioxide layer. At high spin speed, fewer particles aggregate together and the dispersion is more uniform, but the seeding density is a little lower than the expected value of $10^{10}/\text{cm}^2$. Seeding at 3000 rpm will be a better compromise between the low aggregation and high seeding density needed to grow high quality, continuous poly-C film at the thickness of 0.6 µm. Experiments proved that the seeding density is reciprocally related to the spin speed in a repeatable manner as shown in the Figure 3.2.

The seeding uniformity was studied by checking the seeding density at different areas on 4 inch oxidized wafers with different spin speeds. On each wafer, 5 spots were chosen to measure the seeding density by using SEM images. These five spots are equally distributed from wafer center to edge in a 1/2 inch separation. The results in Figure 3.4 showed that the seeding densities were uniform all over the 4 inch wafer even if the rotating speed is different at different radius. This conclusion ensures getting a uniform seeding on the whole 4 inch wafer and broadening the future applications of this method.

In summary, the high density $(>10^{10} \text{ nuclei/cm}^2)$ and uniform diamond seeding was achieved on 4 inch oxidized silicon wafer by the DPLW spin method. The DPLW spinning method for diamond seeding on silicon dioxide, reported for the first time to our knowledge, is a simple, economical, nondestructive and repeatable procedure compared to BEN and surface scratching (Table 2.2). It can also be applied to other hydrophilic dielectric thin film which is not fit for BEN and surface scratching. Combined with the diamond dry etching technology, it can provide an IC compatible diamond thin film deposition and pattern procedure, which will greatly extend the possible applications of poly-C film in the sensors and MEMS area. From the multiple spin results, we can expect that higher seeding density (like 10¹¹ nuclei/cm²) can also be available with this method by using smaller diamond powers, low spin speed and more spin times.

Another seeding method, the spin-coating of diamond loaded photoresist (DPR), was also used in this research to achieve a low density seeding around 10⁸ nuclei/cm². DPR seeding was developed at Michigan State University [29]. The diamond powder, with particle size of 100 nm, was mixed with photoresist with a ratio of 800 mg powder per 80 ml PR.

3.3 Film Growth Using MPCVD

For the results reported in this work, a bell jar type MPCVD chamber (WavematTM MPDR 313EHP) was used with 9 inch chamber diameter and 5 inch quartz bell jar diameter. A 2.45 GHz, 5 kW microwave power supply (SairemTM, GMP60KSM) and a large chamber size ensured the uniformity of the plasma and the poly-C deposition on 4 inch size. Figure 3.5 shows the optical picture and the schematic of the MPCVD system. The sample wafer was heated by the plasma and its temperature was monitored by a pyrometer. The typical deposition conditions are listed in the Table 3.1. Trimethylboron (TMB) diluted in hydrogen (TMB/H₂ = 0.1% in volume ratio) was introduced during the poly-C growth environment for the in-situ boron doping.



Figure 3.5 Optical picture and schematic of the MPCVD diamond growth system .



Figure 3.6 Characteristics of the poly-C film seeded by DPLW spin method. a) Front surface of the film; b) 60 degree view of the film cross section; c) the AFM image of the surface; d) Raman spectrum of the DPLW seeded poly-C film.

Samples	#1	#2	#3
H ₂ /CH ₄ Gas flow (sccm)	200/3	200/3	200/3
Microwave power (kW)	1.6	2.4	2.8
Gas pressure (torr)	22	35	43
Substrate temperature (°C)	520	700	780
Growth rate (µm/hr)	0.1	0.2	0.3

Table 3.1 Typical MPCVD poly-C growth parameters.



Figure 3.7 Dependence of the poly-C growth rate on the substrate temperature for growth with CH_4/H_2 ratio of 1.5/100.

Figure 3.6 shows the different characters of the poly-C film by SEM, AFM (atomic force microscope) and Raman spectrum (green laser with a wavelength of 532 nm was used as the light source). The poly-C film was grown using the #2 conditions from Table 3.1 with a thickness of 1.6 μ m. The average grain size is about 0.6 μ m, and average surface roughness is 42 nm from the AFM image.

The relation between the film growth rate and the substrate temperature is shown in Figure 3.7 for growth with CH_4/H_2 ratio of 1.5/100. Both the growth rate and substrate temperature are referred to the center area of substrate with a diameter about 2 inches.

For the diamond growth in MPCVD chamber, the radial decrease of plasma intensity causes the non-uniformity of film thickness on large area, which limits the possible applications of diamond thin films. Different deposition parameters were tested as shown in Table 3.1 to optimize the film uniformity on 4 inch wafer. Figure 3.8 shows the AFM images of the poly-C film deposited on a 4 inch wafers using 2.4 kW of microwave power and 35 torr pressure (the film thickness at the center of the wafer is 1.6 μ m).



Figure 3.8 AFM images of the poly-C film grown at 700 °C on a 4 inch wafer.



Figure 3.9 Radial distributions of normalized poly-C film thickness at different deposition conditions and the Raman spectra of different samples.

The thickness variation over the 4 inch area of the wafer is shown in Figure 3.9 for three samples with comparable thickness, but grown at different pressures and microwave powers. The thickness of each sample is measured using the SEM method and normalized to 1 for better comparison of the thickness variation along the radius. The inset of Figure 3.9 shows the Raman spectra of the three different samples, which demonstrated that the high growth temperature yielded better film quality.

At low deposition temperature of 520 °C (Table 3.1), the film thickness shows the largest variation which seems to be due to the focusing of plasma near the center of the wafer. The measured temperature of 520 °C is for the center area. As for some applications low temperature growth of poly-C may be required [87], the plasma uniformity can be enhanced by using electron cyclotron resonance (ECR) at low growth pressures. The sample deposited at 780 °C reveals a thickness variation of less than 25%, which is comparable to that reported for 6 inch [41] and 2.36 inch (60mm) [40] wafers using microwave power levels of 15-60 kW and 5 kW, respectively. However, the power consumption per unit area for samples used in this study (0.22 kW/inch²) is much lower than that for 6 inch wafer (0.53 kW/inch²) and for 2.36 inch wafer (1.14 kW/inch²).

3.4 Doping and Surface Conductivity

Accurate control of the doping level and resistivity is particularly important for poly-C thin film applications as the piezoresistive sensors. In this work, the boron doping of poly-C film was investigated as the function of TMB/CH₄ gas ratio, growth temperature and post-growth anneal. Typically, poly-C films with a thickness of 1 μ m were grown at 700 °C with growth conditions described earlier.

Figure 3.10 shows the poly-C film conductivities at different doping levels and different growth temperatures. The films were annealed at 600 °C after the growth to remove the affect by surface hydrogenation. As we can see, at the same TMB/CH₄ gas ratio, higher growth temperature can yield higher doping level and thus higher conductivity. These data can be used as experimental guidelines for doping control.



Figure 3.10 Dependence of poly-C film conductivities on the doping levels.

It has been found that CVD diamond films grown in hydrogen plasma have a thin hydrogenated surface layer, which becomes conductive after exposure to the atmosphere. Annealing at 600 °C or a short time dry etch can remove this conductive layer hydrogen desorption effect [52, 53]. In this work, the boron doping of poly-C film was investigated as a function of TMB/CH₄ gas ratio, deposition temperature and post-deposition anneal. Typically, poly-C films with a thickness of 1 μ m were grown at 700 °C with growth conditions described earlier. The poly-C films were annealed at 600 °C for 20 minutes at 1x10⁻⁵ torr to remove the surface conductive layer by de-hydrogenation.

The actual data measured using the 4-probe method yield the sheet resistance

$$R_{sh} = \xi \frac{V}{I}, \qquad (3.1)$$

where ξ is the geometric coefficient dependent only on the sample size and shape. If the thin film has only one conductive layer with known thickness *t*, the conductivity is given by

$$\sigma = \frac{1}{R_{sh} t}.$$
(3.2)

Since the conductivity of the annealed film is only contributed by the poly-C bulk layer, Eq. 3.2 becomes

$$\sigma_{anneal} = \sigma_{bulk} = \frac{1}{\rho_{bulk}} = \frac{1}{R_{sh anneal} t_{bulk}}.$$
 (3.3)

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

$$\frac{1}{R_{as-grown}} = \frac{1}{R_{surf}} + \frac{1}{R_{bulk}},$$
(3.4)

where R_{surf} and R_{bulk} are resistance of the surface layer and bulk layer, respectively. Since $R = R_{sh} \frac{L}{W}$ (L/W is the Length/Width ratio), Eq. 3.4 can be represented in sheet

resistance as

$$\frac{1}{R_{sh as-grown}} = \frac{1}{R_{sh surf}} + \frac{1}{R_{sh bulk}}.$$
(3.5)

Using Eq. 3.2, the relation between measured total sheet resistance and conductivity is

•••

$$\frac{1}{R_{sh as-grown}} = \sigma_{surf} t_{surf} + \sigma_{bulk} t_{bulk}.$$
(3.6)

Since the thickness of surface layer is much less than the bulk layer: $t_{surf} \ll t_{bulk}$, the conductivity for as-grown film $\sigma_{as-grown}$ can be defined as

$$\sigma_{as-grown} = \frac{1}{R_{sh\ as-grown}} \left(t_{bulk} + t_{surf} \right)$$

$$\approx \frac{1}{R_{sh\ as-grown}} \left(t_{bulk} + t_{surf} \right) = \sigma_{surf} \frac{t_{surf}}{t_{bulk}} + \sigma_{bulk} \quad . \tag{3.7}$$

Figure 3.11 shows the conductivities of as-grown and annealed poly-C films calculated from Eq. 3.3 and 3.7 using the measured sheet resistances. The numerical fits obtained using exponential functions with fractional power yield conductivities, $\sigma_{as-grown}$ and σ_{anneal} , for as-grown and annealed films, respectively

$$\sigma_{as-grown} = 0.033 + 4.677 \times 10^{-4} \times \exp(264.5 \times x^{0.56}), \qquad (3.8)$$
$$\sigma_{anneal} = 4.677 \times 10^{-4} \times \exp(264.5 \times x^{0.56}), \qquad (3.9)$$

where x is the TMB/CH₄ gas ratio. Eq. 3.8 and 3.9 can be used as an empirical guidance for doping estimation. Comparison of Eq. 3.7 and 3.8 reveals that surface layer contributes a constant conductivity of

$$\sigma_{surf} \frac{t_{surf}}{t_{bulk}} = 0.033 (\Omega \cdot cm)^{-1}. \qquad (3.10)$$

If the thickness of surface conductive layer is assumed to be 40nm [88], the corresponding conductivity can be calculated as

•••

$$\sigma_{surf} = 0.033 \times \frac{1\mu m}{40nm} = 0.825 (\Omega \cdot cm)^{-1}.$$
 (3.11)

Eq. 3.11 demonstrates that the surface conductivity is independent of the doping concentrations. The corresponding surface sheet resistance is 330 k Ω /sq.



Figure 3.11 As-grown and annealed conductivities of poly-C films (1µm) deposited at 700 °C with different doping levels.

Theoretically, the bulk conductivity can be written as

$$\sigma_2 = \sigma_{bulk} = qp\mu_p = q\alpha \exp\left(-\frac{E_A}{kT}\right)N_A\mu_p, \qquad (3.12)$$

where q is the charge of electron, p is the hole concentration, N_A is the concentration of acceptors, α is the activation coefficient and E_A is the activation energy. However, theoretical explanation of the relation between the bulk conductivity and TMB/CH₄ gas ratio is not presented here due to the complicate situation of impurity incorporation from gas phase into film, which is related to the growth temperature, pressure, gas flow rate and etc. Combining the theoretical Eq. 3.12 and experimental Eq. 3.9, a tentative expression for the acceptor concentration can be written as

$$N_{A} = \frac{4.677 \times 10^{-4}}{q \alpha \mu_{p}} \exp\left(\frac{E_{A}}{kT} + 264.5 \times x^{0.56}\right).$$
 (3.13)

Although E_A , α and μ_p are all depended on x, Eq. 3.13 can be used as an expression of the relation between the TMB/CH₄ ratio and the acceptor concentration by using those experimental data between $N_A \sim \alpha$ and $N_A \sim E_A$ [89].

3.5 Metallization

In this work, titanium was used for the poly-C metallization with co-deposited gold for protection and wire bonding. Figure 3.12 shows a typical I-V curve of the ohmic contact between poly-C and Ti/Au film at room temperature after annealing at 600 °C in vacuum for 20 min. This I-V curve was measured using a Kelvin Bridge structure as shown in the inset of Figure 3.12. A given current I flows from the poly-C layer to the Ti/Au metal layer by crossing the contact area. The generated voltage difference V is picked from the other two electrodes shown as VI and V2 with V=V1-V2.



Figure 3.12 I-V curve of contact between poly-C film and Ti/Au film after anneal. Inset: the schematic of Kelvin Bridge for contact measurement.

From the I-V curve of the Kelvin Bridge, the contact resistivity can be calculated by:

$$\rho_C = R_C * A = \frac{V}{I} * A,$$
(3.14)

where ρ_c is the contact resistivity, R_c is the contact resistance, and A is the size of contact area.



Figure 3.13 Resistivity dependence of the annealed contact resistivity between poly-C film and Ti/Au film.

The contact resistivity between poly-C (2 μ m) and Ti/Au (300/2000 Å) was studied by using poly-C samples with different doping levels. All samples are seeded with DPR method and grown at 750 °C. After the Kelvin Bridges are formed, the samples were annealed at 600 °C for 20 minutes in vacuum. Figure 3.13 shows the resistivity dependence of the annealed contact resistivity between poly-C film and Ti/Au film.

3.6 Patterning

The dry etching of poly-C film was carried out in an ECR MPCVD chamber and the plasma was generated by a 2.45 GHz, 1.5 KW (ASTeXTM) microwave power supply. The outside magnetic bar array will make the electrons resonate in cyclotron shape to increase the uniformity of the plasma. The inner diameter of the quartz dome is 13 inch and the etching is uniform compare to the diamond deposition system. Coupled by two capacitors and the matching network, the RF power will generate negative bias on the substrate of etching sample holder, which is critical to the etching rate. The diamond will not be etched without the bias. The RF power is about 50 - 100 W and the generated negative bias is in the range of -50 V to -150 V. The O₂, SF₆ and Ar [63], with a flow ratio of 28/2/2, were used as the reactive ion source for etching using a pressure in the range of 4 - 8 militorr. With a rotary pump and turbo-molecular pump, the achievable base vacuum is approximately 10^{-7} torr.
The positively ionized oxygen atoms are the main source for etching the diamond atoms (sp³ carbon atomic bonding) under the negative substrate bias. The small amount of SF₆ and Ar will increase the uniformity of the etching process. Since alumina is very stable and resistive compound, the oxygen plasma won't etch Al much. So Al (there will be a thin oxide layer on top of it in atmosphere) is the ideal mask for etching diamond, which only have an etching rate about 1/13 of the etching rate of diamond [67].

A typical etch rate for poly-C is 50 -120 nm/min with a variation of less than 7% over the 4 inch area of the wafer. Figure 3.14 shows SEM images of two poly-C structures prepared by the ECR plasma etching. The first SEM shows a finger structure with 1 μ m thickness and 2 μ m minimum feature size. The second SEM shows an intragrain p-type piezoresistor fabricated on an electrically-insulating 300- μ m-thick large-grain free-standing poly-C plate.

Sometimes the dry etching of poly-C can cause aluminum sputtering over some areas of the diamond surface, especially for a long time etching which will cause prominent heating of substrate. When this happens, the plasma can not attack the diamond under the sputtered aluminum. As a result, one might end up getting "spikes" around the patterned structure after the dry etching of diamond is completed in the rest of the sample. This is called the *grass effect* as we can see from the Figure 3.14b. We had to go through a more vigorous rinsing and clean process in order to eliminate those diamond spikes from the sample [90].



Figure 3.14 SEM images of poly-C structures prepared by the ECR plasma etch: a) the free standing poly-C finger structure and, b) single grain piezoresistor on large grain poly-C plate.

3.7 Summary

In this chapter, several newly developed technologies were reported for the sensor applications of poly-C film. The first time reported DPLW spin seeding method yielded high density $(2x10^{10}/cm^2)$, non-destructive and repeatable diamond seeding on 4 inch Si wafer with dielectric surface. The Poly-C growth on 4 inch wafer was achieved in our MPCVD system with less than 25% thickness variation using 2.8 kW microwave power and 43 torr pressure. The poly-C surface conductivity, which was caused by the surface hydrogenation, was quantitatively studied. The calculation shows that the surface conductivity is about $0.825/\Omega^*$ cm and is independent to the doping level of poly-C film.

Chapter 4

Testing of Poly-C Piezoresistivity

4.1 Introduction

As shown in literature review, high piezoresistive effect of poly-C shows a promising application as piezoresistive sensor. However, the reported piezoresistive gauge factor of the poly-C films was in the range of 5 to 1000 with large variations. Normally, those high GF values in the prior studies were achieved from films with resistivities over 100 Ω^* cm, which are impractical for sensor application due to the high film resistances leading to high noise levels.

In this chapter, a cantilever beam method was used to study the piezoresistivity of poly-C films. Poly-C thin films, with thicknesses about 2 μ m and with different resistivities, will be fabricated and tested to explore how the film properties affect the piezoresistive GF. Two seeding method, DPLW and DPR, will be used to study the relation between the GF and the film grain size.

4.2 The Cantilever Beam Method

For measuring the poly-C piezoresistive effect, it is necessary to apply longitudinal mechanical strain to the piezoresistors. In order to apply this strain and measure the

resistance change, a testing stage for cantilever beam method was build as shown in Figure 4.1. The silicon wafer, where the poly-C piezoresistor was grown, was diced into a cantilever beam with a dimension about 60x10x0.5 mm³. As shown in Figure 4.1, the cantilever beam was clamped between two metal plates and was fixed on the stage from one end using four screws. The mechanical strain was applied on the beam by bending the other end of the beam using a linear positioner. The bending of the beam was measured using a movement gauge with a minimum scale of 0.001 inch. The resistor was electrically connected to the testing instruments using wire bonding.



Figure 4.1 Piezoresistive testing stage using cantilever beam method.



Figure 4.2 Schematic diagram of the cantilever beam method used for piezoresistive measurement.

The schematic of the cantilever beam testing setup is shown in Figure 4.2 with all the parameters labeled in the figure. The piezoresistive sensor was fabricated on an oxidized silicon wafer for electrical insulation purpose. Since the thickness of the poly-C thin film resistor (normally less than 2 μ m) is much smaller as compared with the thickness of Si beam, *h* (normally 500-525 μ m), the strain of poly-C film, ε , is approximately equal to the strain on the surface of cantilever beam. Bending the free end of the beam through a distance of δ , the longitudinal strain of the poly-C film, ε , can be calculated using the parameters *h*, *l*, *a* and *b* as shown in the Figure 4.2 [76, 90],

$$\varepsilon = \frac{3h}{2l^3} \left[l - \frac{(a+b)}{2} \right] \delta \,. \tag{4.1}$$

The strain calculated using Eq. 4.1 was verified by attaching a commercial metal strain gauge on the beam and showed an error of less than 5%. The gauge factor of the poly-C can be expressed as

$$GF = \frac{R - R_0}{R_0} \cdot \frac{1}{\varepsilon} = \frac{2(R - R_0)l^3}{3R_0h\delta\left[l - \frac{(a+b)}{2}\right]},$$
(4.2)

where R is the resistance with strain ε and R_0 is the resistance at zero strain.

4.3 Sample Preparation

Poly-C films were prepared on the silicon cantilever beam using the technology mentioned in Chapter 3. The silicon beam has a thin layer of SiO_2 for electrical insulation. Two different spin seeding methods, DPLW [57] and DPR [91], were used to grow films with different morphologies result from different seeding densities. DPLW or DPR was spin coated on the samples with a typical spin speed of 3000 rpm. Table 4.1 compares the two seeding methods in preparation conditions, seeding density and grown film morphology.

Seeding Method	DPLW	DPR
Mean diamond powder size (nm)	25	50
Carrier fluid	Water	Photoresist
Density (carats/liter)	40	12
Seeding density (cm ⁻²)	10 ¹⁰	10 ⁸
Ave. grain size (μm) (film thickness is 2 μm)	0.3	0.8
Surface roughness (RMS) (nm)	30	46

Table 4.1	Comparison	of DPLW and	DPR	seeding	methods.
-----------	------------	-------------	-----	---------	----------

Figure 4.3 shows, for both the DPLW and DPR methods, the SEM pictures before and after the growth of poly-C films with thickness of 2 μ m. The affect of seeding on the piezoresistivity will be discussed late in section 4.4.

After the seeding procedure, the sample wafers were grown at temperature of 700 °C with CH_4/H_2 flow rate of 1/100 sccm. The film thickness is about 2 µm with a growth rate about 0.15 µm/hr. Trimethylboron was introduced during the growth for in-situ boron doping and achieved resistivities from 0.05 Ω *cm to 70 Ω *cm. Samples were dry etched and then coated with E-beam evaporated Ti/Au (300/2000 Å) film. After the metallization, the samples were annealed at 600 °C in vacuum (10⁻⁵ torr) for 30 minutes.

For studying the piezoresistivity, a mask set with two masks was designed for poly-C layer and contact metal layer as shown in Figure 4.4. Different resistor sizes were designed with length/width ratio from 500/20 μ m to 50/200 μ m. Two kind of metal contacts, 4-contact and 2-contact, were used for the electrical connection. The 4-contact method was used to avoid the affect of the sensor resistance by the contact resistance. Testing structures such as contact chains, Kelvin Bridges and resistivity study were

included in the design to characterize. Figure 4.5 shows one piezoresistor and one Kelvin Bridge fabricated using this mask set.



Figure 4.3 SEM pictures of a) DPLW seeding; b) poly-C film (with thickness of 2 μm) seeded by DPLW; c) DPR seeding; d) poly-C film (with thickness of 2 μm) seeded by DPR.



Figure 4.4 Layout of the two masks (for poly-C and contact metal) designed for

testing the piezoresistors.



Figure 4.5 a) SEM of the 400/20 µm piezoresistor with 4-contact; b) SEM of the Kelvin Bridge with gold wire bonding.

4.4 Piezoresistive Results

Figure 4.6 shows a typical measurement result of the piezoresistivity using the cantilever beam method. The sample used in Figure 4.6 was prepared using the parameters mentioned in section 4.3. The strain in this figure is tensional achieved by bending the beam down. The good linearity of the results proved that the measurement of strain is consistent and the GF is a constant value along the measurable strain range.



Figure 4.6 Relation between resistance change and strain for poly-C film prepared by the DPR seeding.

The 4-contact piezoresistor in Figure 4.4 and 4.5 are used to avoid the affect of contact resistance to the measurement. By applying a current using the two outer electrodes and

picking up the voltage difference between the two inner electrodes, the measured resistance will only be contributed by the poly-C piezoresistor itself. For piezoresistors with high doping level or with large length/width ratio, the affect by contact resistance will be ignorable and the 4-contact structures showed similar results as the 2-contact structures. For lightly doped sample or with very short length/width ratio, 4-contact was chosen for the test.



Figure 4.7 Relations between the GF and the boron doped resistivity for both DPLW and DPR seeded poly-C films.

To study the GF dependence on the film properties, a set of samples were prepared with different doping resistivity varies from 0.05 to 100 Ω^* cm. They are either seed by DPLW or by DPR as mentioned in section 4.2. But all the samples were grown at 700 °C with film thicknesses about 2 µm. Figure 4.7 shows the measured GF results for all these samples [92]. Discussion of the GF dependence is presented in the next section.

4.5 GF Dependence on the Film Properties

The dependence of GF on poly-C film resistivity is shown in Figure 4.7 for both DPR and DPLW seeded films with thicknesses about 2 μ m. The results show that, for films with resistivity less than 1 Ω *cm, the GF values are around 6-15 and do not change much with the resistivity and grain size. However, the GF increases substantially with resistivity for films with large grain size (grain size of 0.8 μ m by DPR seeding) and with resistivities higher than 1 Ω *cm. The GF increases substantially for film with small grain size (0.3 μ m, DPLW seeding) if the film resistivity is higher than 10 Ω *cm.

For the same resistivity higher than 1 Ω^* cm, the film with large grain size (0.8 µm by DPR seeding) gives a GF about 2-3 times higher than that of film with small grain sizes. This result is consistent with the reported GF of 283 from free standing diamond sample with grains in 50 µm range [8]. The high GF values found in large grain films are believed to be related to high sp³/sp² ratios caused by lower densities of grain boundaries.

It is also known that high resistivity will cause large thermal noise and high contact resistance [93], which will hinder the sensor application of poly-C films. By comprehensively considering the GF, film thickness, roughness, resistivity and contact resistance, we proposed the optimum condition for poly-C piezoresistive sensor application in microsystems as shown in Figure 4.7. For DPR seeded film with thickness of 2 μ m, controlling the film resistivity in the range of 20 to 80 Ω *cm will yield a GF about 50 for sensor application, which is expected to lead to unprecedented sensitivity and promising applications.

4.6 Summary

In this chapter, the measurement techniques and the equipment set up for poly-C piezoresistivity study was reported. The piezoresistivity of poly-C film with different properties was successfully measured using cantilever beam method. The result shows that for DPLW seeded small grain film, the GF begin to increase as resistivity is higher than 20 Ω^* cm. For DPR seeded large grain film with thickness about 2µm, the GF will be in the range of 30-70 if the resistivity is about 20-80 Ω^* cm. This is an optimum region to poly-C film for sensor applications.

Chapter 5

Poly-C Sensor for 2nd Generation Cochlear Probe

5.1 Introduction

Because of its high piezoresistive sensitivity, poly-C film was chosen as the position sensor material for the cochlear implant probe for the WIMS center. The first generation of the silicon thin film cochlear probe was reported by Dr. Wise's group at University of Michigan without the position sensors [84].

This chapter will report the first attempt of integrating the poly-C piezoresistive sensors into the 2nd generation cochlear probe [94]. The design, fabrication and testing of the probe with poly-C sensors will be discussed in detail.

5.2 Design and Fabrication of the Probe

5.2.1 Technology for Poly-C Process Integration

Integration is one of the important issues which limited the development and application of poly-C thin film sensors because of the special seeding and deposition requirements [95, 96]. A common procedure in integrated circuits (IC) process is insulating the active layer (poly-Si or metals) with dielectric layers (like SiO₂) both above

and below it, and making electrical connections through the contact holes [97]. Based on the design of poly-C piezoresistive position sensors [98], we developed a poly-C thin film fabrication process to integrate the poly-C technology into standard IC fabrication process as shown in Figure 5.1.



Figure 5.1 Process flow for integrating poly-C sensor into Si-based microsystems.



Figure 5.2 Cross-sectional profile of the cochlear implant probe with poly-C sensors.

The poly-C thin film will be seeded by DPLW or DPR and grown by MPCVD. Patterned metal mask will be used to pattern the poly-C film by dry etch. The poly-C deposition temperature can be varied between 400 °C to 900 °C depending on the materials used underneath the poly-C. The process insulates the micron-scale continuous poly-C films with dielectric layers both above and below it, and makes electrical connections through the contact holes such as those found in common IC processes. This method can protect the underneath materials during the diamond deposition and dry etching steps. This process ensures the compatibility of poly-C film with the typical IC or MEMS procedure and broadens the possible applications of poly-C thin films.

5.2.2 Design of the 2nd Generation Probe

The basic concept of the silicon thin film cochlear implant probe has been addressed in section 2.5. In the design of the 2nd generation probe, both poly-Si and poly-C were chosen as the position sensors for comparison. The design of the probe was mainly done by Jianbai Wang based on previous experience from Dr. Wise's group [83]. We collaborated to integrate the poly-C sensor into the design by adding one more mask for poly-C patterning. Figure 5.2 shows the detailed cross-sectional profile of the cochlear probe with poly-C sensors. Both highly doped poly-Si and Ti/TiN/Al metal stack is used as the interconnect lines. The iridium and gold are used as the stimulating sites and the pad metal, respectively. The length and width of the thin film probes are about 16 and 1.2 mm, respectively. Adding the boron diffusion and top dielectrics, the thickness of the probe backend and shank are about 16 μ m and 6 μ m, respectively. On each probe, there are eight straight poly-C position sensors uniformly distributed along the shank of the probe and one identical poly-C reference sensor located on the backend of the probe. The reference sensor is connected with each of the position sensor in tandem to form half Wheatstone bridges for testing the resistance change of position sensor [83]. The reference sensor has the same dimension as the position sensor but will not be bent while the probe is bent during the insertion. There are also one "U" shape tip sensor on the tip of the probe and one identical reference sensor on the backend to detect the deformation of probe tip. For comparison, the poly-C sensors have the same dimension as the poly-Si sensors. The length/width for the straight position sensor and the tip sensor are 450/18 μ m and 1000/18 μ m, respectively. There are also one square wave shape side wall sensors and corresponding reference sensor designed on some of the testing probes.

5.2.3 Fabrication of the 2nd Generation Probe

Figure 5.2 depicts all the layers created in the fabrication process. The fabrication began with two boron etch-stop diffusions to define the profile of the probe. The thinner probe shank (shallow boron diffusion, 4 μ m) is typically inserted into the cochlea, while the thicker (shallow and deep boron diffusion, 14 μ m) backend remains outside to support the electrical connections. A stress-compensated dielectric stack of SiO₂/Si₃N₄/SiO₂ (200/100/100 nm) was deposited by low-pressure chemical vapor deposition (LPCVD). The conducting leads were defined using ion implanted poly-Si (600 nm) [84]. Another

dielectric stack of Si_3N_4/SiO_2 (150/300 nm) was deposited to cover the poly-Si leads. Then, the poly-C film was seeded and grown on the second dielectric stack using MPCVD and patterned by plasma etching as described in Figure 5.1.

	Sample80	Sample82	Sample83
Growth time (hrs)	24	24	20
Temp (C)	580	540	560
MW power (kW)	1.55	1.55	1.55
Gas flow (H2:CH4:TMB)	100:1:1	100:1:0.2	100:1:0.1

22

1.7

0.322 / 0.332

0.06 / 0.063

20

0.7

35 / 50

2.1/3

21

1.1

86.1/122

8.09/11

Pressure (torr)

Center Thickness (µm)

Sheet resistance $(k\Omega/sq)$

[before/after surface etch]

Resistivity (Ω^* cm)

[before/after surface etch]

Table	5.1	Growth	conditions	and	properties	poly-C	films	for	the	2 nd	generation
probe.											

In this design, we expected the poly-C film thickness of 0.6-1 μ m to be comparable with poly-Si (thickness is 0.6 μ m). So DPLW was used with spin speed of 3000 rpm to achieve a seeding density of $2x10^{10}$ /cm². Due to the malfunction of our 5 kW microwave power source, a smaller microwave power source was used for the fabrication with a maximum output of 1.55 kW. Table 5.1 lists the growth conditions and properties of the poly-C films on three wafers for the 2nd generation probe.



Figure 5.3 Thickness distribution of poly-C film on the 4 inch wafers.

Due to the ball shape of microwave plasma, the heating of wafer is not uniform. The center temperature is higher than the edge temperature, which causes the diamond deposition is not uniform on the 4 inch wafer. Because of the low microwave power, low gas pressure is needed to match the power for better thickness uniformity. The poly-C film thickness distributions were monitored and shown in Figure 5.3. On the 4 inch wafers, the poly-C growths were mainly happened within the 2 inch areas and the films are not continuous on the edge areas. After the growth, all films were treated with surface etch (dry etch 30 second) to remove the surface conductive layer. The sheet resistances in Table 5.1 were all measured from the center of wafer suing 4-probe method.

After the growth, the poly-C films were patterned into position sensors using dry etch as described before. The poly-C sensors were then covered with low temperature oxide (LTO) before a metal stack of Ti/TiN/Al was sputtered and patterned by a lift-off process as interconnect between poly-C and poly-Si through contact holes. Iridium over titanium is sputtered and patterned by lift-off to define the stimulating sites. Gold bonding pads are formed on the back-end of probes in a similar manner. After this, the dielectric stack, include all the oxide and nitride layers, was patterned into the shape of probes. After finishing all the process steps on front side, the wafer was thinned from 500 μ m to about 200 μ m using HNA etch (HF:HNO₃:Acetic acid=1:3:8). Then the thinned wafer was etched using ethylene-diamine-pyrocatechol (EDP) etchant. With the boron diffused etch stop, the probes were released in EDP and cleaned for future test.



Figure 5.4 Released 2nd generation cochlear implant probe with poly-C position sensors.

Most of the process steps were done at Michigan Nanofabrication Facility (MNF) at University of Michigan by Jianbai Wang and the technicians. The diamond seeding, growth and patterning were finished at MSU lab. Due to the fabrication loss, only one wafer (sample 82) was released at last. Figure 5.4 shows one of the released cochlear probes from sample 82 with close-view of the different structures on the probe.

5.3 Testing of the 2nd Generation Probe

A testchip was fabricated on the same wafer with the implant probes to characterize the properties and interaction of all films used in the probe. Figure 5.5 shows the testchip used for film characterization and those labeled structures are all used for poly-C film characterization.

Using the 4-contact resistors on the testchip, we measured the sheet resistances of the poly-C resistors and found there are about 1-2.5 M Ω /sq, which is about 20-50 times higher than the value measured from the wafer center before we patterned it. It was believed that the discontinuity of poly-C film caused this resistance increase. Since this designed is shared with other testing probe, we only had limited amount of poly-C probes and most of the poly-C probes and testchips are located on the outer area of the 4 inch wafer. The small microwave powder led to very little poly-C growth on the outer area, as it can be seen from Figure 5.3, and caused this resistance increase.



Figure 5.5 Testchip on the 2nd generation probe wafer for process characterization.

The contact resistivity between poly-C and Ti/TiN/Al interconnect metal stack is approximately 0.2 Ω^* cm². The dry etching of poly-C produced a minimum feature size of 2 µm and showed reliable step coverage between 2 µm wide poly-C and metal layers. The resistance of dielectric layers was more than 10⁹ Ω ensuring an excellent insulation for the poly-C films.



Figure 5.6 Sketch of the testing structure for probe position sensing and the measured gauge factor result.

For measuring the GF of poly-C position sensors integrated on the cochlear probe, the probe backend was glued on a printed circuit board (PCB) substrate and electrically connected by gold wire bonding. Then the probe was bent to different curvatures using a regulator as shown in Figure 5.6. The curvature was recorded using digital camera and the corresponding radius was extracted from the stored image using geometric method.

For approximation, the probe can be treated approximately as a uniform beam with a length of 1.6 cm and a thickness of 6 μ m. The stain on poly-C sensor, ε , can be calculated by:

$$\varepsilon = \frac{h/2}{\rho} = \frac{h}{2\rho},\tag{5.1}$$

where *h* is the probe thickness and ρ is the radius of curvature. The GF was then calculated using,

$$GF = \frac{(R-R_0)/R}{\varepsilon} = \frac{2(R-R_0)\rho}{Rh}.$$
 (5.2)

A GF of 28 was achieved for a poly-C sensor on the cochlear probe as shown in Figure 5.7. It's higher than the GF of poly-Si.

5.4 Summary

A Si based thin film cochlear implant probe with poly-C piezoresistive position sensors was designed and fabricated successfully. The optimized poly-C processes, with seeding, growth, doping and etching, demonstrated a feasible integration with the bulksilicon based micro-fabrication technology although the power limitation caused the discontinuity of poly-C film. A GF of 28 was achieved for one of the probe, which is higher than the GF of poly-Si (12-18) position sensors fabricated in the same batch [83].

During the fabrication of the 2^{nd} generation cochlear probe, we have not done systematical study on the relation between GF and film properties. Further work will be presented to improve the technology and process for better integration and high sensitivity as we will see in the next chapter.

Chapter 6

Poly-C Sensor for 3rd Generation Cochlear Probe

6.1 Introduction

The 2^{nd} generation poly-C probe showed a good startup and promising future for the application of poly-C thin film although there are several issues that need to be addressed. The major problem was the low microwave power, which led to discontinuity of the poly-C film, as descried in Chapter 5. This increased the total resistance of the piezoresistor to a value of 20-60 M Ω , which is difficult for the measuring system. Based on the testing of the 2^{nd} generation probe, a new design of poly-C sensor with the cochlear probe is reported in this chapter as the 3^{rd} generation probe.

6.2 Design of the 3rd Generation Probe

The design of the new probe is an upgrade from the 2nd generation probe with many new improvements. The cross-sectional view of the 3rd generation probe is shown in Figure 6.1. Table 6.1 summarized the problems existed in the 2nd generation probe and proposed the solutions for those problems. As shown in Figure 3.9, high power and high pressure, 2.8 kW and 43 torr, were used to achieve uniform growth on 4 inch wafer. As discussed in chapter 4, to improve the piezoresistive sensitivity, large grain size and low boron doping should be used. So DPR seeding method and lightly doped poly-C film was chosen to achieve high GF. In addition, the length of piezoresistor was shorted to reduce the total resistance for better integration.

	2 nd gen. Probe	3 rd gen. Probe	Improvements
Microwave power	1.6 kW	2.8 kW	Increased uniformity
Poly-C thickness	0.6 -0 µm	2 - 1.4 μm	Increased stability
Resistor size	500/18 μm	10-200/20 μm	Reduced total resistance
Contact	Regular	Highly doped interlayer	Reduced contact resistance
Interconnect	Highly doped poly-Si	Ti/Al metal stack	Reduced total resistance
Grain size	0.2 -0.3 μm (DPLW seeding)	0.8 - 1.2 μm (DPR seeding)	Increased GF

Table 6.1 Comparison of the 2nd and 3rd generation cochlear probe.

For short resistors, the contact resistance will contribute to a large percentage to the total resistance. Thus, a new process was developed to reduce the contact resistance and hence increase the piezoresistive sensitivity by using a highly doped thin poly-C layer. This method is reported for the first time for poly-C films although similar method was used before for reducing the silicon/metal contact resistance with ion implantation in the contact area [99].



Figure 6.1 Cross-sectional view the 3rd generation cochlear probe with poly-C piezoresistive position sensors.

Another difference between the 2^{nd} and 3^{rd} generation probes is that PECVD oxide was used as the passivation layer in the new design due to the process availability, although the previously used LTO has higher quality and coverage.

The mask design of the 3rd generation probe was done in collaboration with Jianbai Wang. Table 6.2 listed all the masks used for the new design with poly-C sensors. The first 6 masks in Table 6.2 were designed by Jianbai and shared by Jianbai (with poly-Si sensors) and me (with poly-C sensors). I designed the last 4 masks in Table 6.2 to realize the poly-C sensors on the probe.

gds#	Mask name	Description
1	BDF:	deep boron diffusion, define the probe backend
2	DEL:	dielectrics pattering, define the probe profile
5	SHB:	shallow boron diffusion, define the probe shank
6	GOL:	sputtered gold pads
7	CON:	Contact hole to metal1
8	IRD:	Iridium stimulating site
21	DIA:	poly-C sensor
23	HDC:	Heavily doped poly-C for contact
24	PCC:	Contact hole to poly-C
25	DCO2:	metal1 for diamond sensor

Table 6.2 Masks used for the 3rd generation cochlear probe with poly-C sensors.

For the design of the poly-C sensors using 4 masks, different dimensions were used for the poly-C position sensors. The width of the sensors was 20 μ m and the length was designed as 5/10/20/50/200 μ m for different probes.

6.3 Fabrication of the Probe

The detail fabrication processes of the 3rd generation cochlear probe with poly-C position sensors are shown in Figure 6.2. The process is similar to that of 2nd generation probe except those differences mentioned in previous section. The first three steps (deep boron diffusion, shallow boron diffusion and dielectric deposition) were done by the technicians of Michigan Nanofabrication Facility (MNF) and I finished all the remaining steps at MNF except the poly-C growth and etching steps which were accomplished at MSU. The detail process steps are listed in the appendix.



Figure 6.2 Process flow of the 3rd generation cochlear probe with poly-C piezoresistive position sensor.

The lightly and highly doped poly-C film was grown on the dielectric stack by PECVD with the parameters shown in Table 6.3. Dry etching of the poly-C films was divided into two steps. The first step was used to define the contact areas by etching only the highly doped top layer beyond the contact areas. The second step was patterning the shape of the piezoresistors.

Poly-C growth				
H ₂ /CH ₄ gas flow (sccm)	200/3			
Trimethylboron doping (sccm)	$0.2-2 \times 10^{-3}$			
Microwave power (kW)	2.8			
Gas Pressure (torr)	43			
Growth Temperature (°C)	780			
Film thickness (µm) : lightly/highly doped	1.5-2 / 0.1-0.3			
Film resistivity (Ω^* cm) : lightly/highly doped	25-150 / 0.01-0.05			

Table 6.3 Detail growth parameters of poly-C film.

To accurately control the etch depth, the sheet resistance of the poly-C film was measured using a 4-probe station after every 30 second etch as shown in Figure 6.3. The sheet resistance was calculated by measuring the voltage and current using the 4-probe method as described by Eq. 3.1 and 3.2.



Figure 6.3 Change of the sheet resistance of poly-C film vs. the dry etch time.

As shown in Figure 6.3, after 4 minute etch, the resistance of poly-C film reach a slow changing range which means the highly doped layer was totally removed. Measured by a surface profilometer, the thicknesses of the lightly and highly doped layer are 0.2 and 1.8 μ m, respectively. The etch rate is about 50 nm/min. So for the etched film, there is only lightly doped layer with the resistivity, ρ_{lignt} , given by

$$\rho_{light} = R_{sh_etched} \cdot t_{light} = 840k\Omega / sq \times 1.8 \times 10^{-4} cm = 151\Omega \cdot cm.$$
(6.1)

For the film before etch, the conductivity is contributed from both highly and lightly doped layers, but the contribution caused by the lightly doped layer is ignorable, so the resistivity of highly doped layer, ρ_{high} equals

$$\rho_{high} \approx R_{sh_unetch} \cdot t_{high} = 1.12k\Omega / sq \times 0.2 \times 10^{-4} cm = 0.0224\Omega \cdot cm.$$
(6.2)

So from the Eq. 6.1 and 6.2, the corresponding resistivities for lightly and highly doped poly-C films are 0.0224 Ω^* cm and 151 Ω^* cm, respectively.

The poly-C sensors were then covered with $1\mu m$ PECVD SiO₂ before Ti/TiN/Al metal stack was sputtered and patterned as the interconnect lines for poly-C sensors. Another PECVD oxide layer with thickness of $1\mu m$ was deposited to cover the metal lines. The remaining steps were the same as those of 2^{nd} generation probe (section 5.2.3). Fig. 6.4 shows the detail views of the fabricated sample wafer and the cochlear probes with poly-C position sensors.


Figure 6.4 Overview of the fabricated 3rd generation cochlear implant probe with poly-C position sensors.

6.4 Testing of the Probe

6.4.1 Low-resistance poly-C contact

Figure 6.5 shows the testchip fabricated for characterizing the properties of poly-C films, which included several testing structures such as Kelvin Bridge, contact chain, cross talk and transmission lines.



Figure 6.5 Overview of the testchip for the poly-C characterization.

Kelvin Bridges with different contact sizes were used to test the contact resistance. The contact size, equals the size of contact hole on SiO₂, varies from 100, 400 to 1600 μ m².



Figure 6.6 Schematic of the Kelvin Bridge used for testing contact resistivity.

Figure 6.6 shows the schematic of the Kelvin Bridge used for measuring contact resistance with highly doped interlayer. A given current flows from the poly-C layer to the titanium metal layer by crossing the contact area. The generated voltage difference is picked from the other two electrodes shown as V1 and V2 in Figure 6.6. The SiO₂ layer and contact hole is skipped in the sketch for better view. The equation for calculating the contact resistivity is:

$$\rho_C = R_C * A = \frac{(V1 - V2)}{I} * A$$
(6.3)

where ρ_C is the contact resistivity, R_C is the contact resistance, and A is the size of contact area.

Figure 6.7 shows the SEM pictures of a Kelvin Bridge with highly doped interlayer and the close view of the poly-C surface. The average grain size of this poly-C film is about 0.8 μ m. After the dry etch, the surface shows many column-like structures. These small columns are believed to be formed by the formation of organic residue on the diamond and sputtering of the metal etching mask [100]. Although it increased the surface roughness, we expect these small and separated columns will not affect the resistance of the lightly doped film.



Figure 6.7 SEM pictures of the Kelvin Bridge and close view of the poly-C surface.

As shown in Figure 6.8, both type of contacts (with/without interlayer) show a linear I-V relationship, which means the formation of ohmic contacts by forming titanium carbide. For the films with resistivities of $0.022/151 \ \Omega^*$ cm for lightly and highly doped layers, the average contact resistivities for the contact with/without highly doped interlayer are $0.026/0.068 \ \Omega^*$ cm², respectively. For the films with resistivities of $0.016/48 \ \Omega^*$ cm, the contact resistivities are $0.0028/0.0083 \ \Omega^*$ cm² with/without interlayer. The result shows that the highly doped interlayer can reduce the contact resistances to about one third of the original values.



Figure 6.8 I-V curve of the contacts with/without highly doped interlayer.

6.4.2 Testing of the Piezoresistive GF

The application of this low-resistance contact was used in the new design as shown in Figure 6.9. The SiO₂ layer and metal layer were removed for clear view of the lightly doped diamond sensor with highly doped contact areas. The measured resistance of the 20 μ m x 20 μ m resistor is about 123 k Ω with total contact resistance about 14 k Ω . The film is seeded by DPR with a film thickness of 2 μ m and average grain size of 0.8 μ m.



Figure 6.9 SEM pictures of the lightly doped poly-C piezoresistor with highly doped contact areas.



Figure 6.10 GF of poly-C sensor on the cochlear probe achieved by measuring the resistance dependence on strain.

For testing the GF of the above poly-C position sensors on the cochlear probe, the probe was glued on an oxidized silicon wafer and measured using a cantilever beam setup as described in section 4.2. Since the thickness of the probe is very small compared to thickness of silicon wafer, we can still use Eq. 4.2 for calculating the GF. Figure 6.10 shows the calculated GF of 44 achieved from the poly-C sensor on the cochlear probe by measuring the resistance dependence on strain. The resistance of the sensor is 683 k Ω with the length and width of 50 and 20 μ m, the corresponding resistivity is 54.6 Ω *cm. This result is consistent with the conclusion from chapter 4.

6.5 Summary

Based on previous studies, the 3^{rd} generation cochlear probe with poly-C position sensors were designed, fabricated and tested with several new improvements for increasing the piezoresistive GF and compatibility with the Si-based technology. In-situ doped poly-C film, with a thickness of 2 µm, were grown and patterned on the probe as the piezoresistor. Low contact resistivity between poly-C and metal was achieved using a highly doped poly-C thin interlayer. A piezoresistive gauge factor of 44 was achieved from the poly-C sensor on the probe.

Test results shows that the poly-C process improvements, such as high microwave power, contact interlayer, short resistor and large grain size, reduced the contact resistance, total resistance and increased the GF and stability. This work demonstrated a successful integration and promising application of poly-C technology with the silicon based microsystems.

Chapter 7

Summary and Future Research

7.1 Summary of Contributions

7.1.1 Results reported for the first time

• DPLW spin-coated seeding

DPLW spin-coating was developed as the new diamond seeding method which can yield high density $(2x10^{10}/cm^2)$, non-destructive and repeatable diamond seeding on 4 inch Si wafer with dielectric surface. Dependence of seeding density on spin speeds and repeat times were studied with proof of good uniformity over 4 inch wafer.

• Low-resistance contact between poly-C and Metal

Low contact resistivity between poly-C and metal was achieved using a highly doped poly-C thin interlayer. The contact areas were defined by etching only the highly doped top layer beyond the contact areas. For same poly-C film and metal, this new design yield a contact resistivity about one third of the value of regular contact.

• Cochlear implant probe with poly-C piezoresistive position sensor

The poly-C piezoresistive position sensor was successfully integrated into two generations of cochlear probes and demonstrated its high piezoresistive GF of 44. Several improvements, such as high microwave power, contact interlayer, short resistor and large grain size, were implemented in the 3rd generation design, which led to low contact resistance, low total resistance and high GF.

7.1.2 Other significant contributions

• Study of poly-C surface conductivity

The poly-C surface conductivity, which was caused by the surface hydrogenation, was quantitatively studied by measuring the annealing caused change of sheet resistance. The calculation shows that the surface conductivity is about 0.825 (Ω^* cm)⁻¹ and is independent to the doping level of poly-C films.

• Poly-C gauge factor dependence on film resistivity and grain size

The GF dependence of poly-C on film doping and grain size was studied in detail by using different doping levels and two seeding methods (DPLW and DPR). GF increases prominently as the resistivities large than 10 Ω^* cm, and at the same resistivity level, the GF of film with grain size of 0.8 µm (DPR seeding) is about 3 times higher than that of the film with grain size of 0.3 µm (DW seeding).

• Technology for poly-C process integration

An optimum poly-C process was developed which included DPLW seeding, dry etching and metallization over SiO_2 passivation. This process is IC compatible and ensures the integration poly-C thin film with silicon based microsystems.

7.2 Future Research

Although this study improved the understanding of poly-C technology and piezoresistivity, proved the application of poly-C sensor in cochlear probe, more work is needed in the following areas in order to provide more promising applications for the poly-C thin films:

- i) Design interface circuit to test the probe for shape regeneration.
- ii) Improve the poly-C equipment and process for better film quality.
- iii) Evaluate the poly-C sensor reliability and noise level.
- iv) Study the electrochemical and biological properties of poly-C thin film for possible application as the electrode material in biomedical probe.

APPENDIX

APPENDIX

Fabrication process of the 3rd generation cochlear probe with poly-C position sensors

1. Prepare starting wafers

- P-type, <100>, 10-20 [Ω-cm]
- Scribe backsides with wafer ID's
- Rinse: DI-H2O, 2 min; spin dry
- •
- 2. Pre-furnace clean
- 3. Grow deep boron masking oxide (B2)
 - Recipe: DWD/TCA; Parameter Table: DWDSKIN

Run number: B2-

UPGAS/A	N2-3	DRY1	00:10:00	PUL-600	200
· TEMP/A	800	WET	00:00:00	SETTLE	SPKSET
TEMPRMP	10	WET/TCA	04:00:00	HIGHSET	2.0
DRY/A	00:30:00	DRY2	00:10:00	LOWSET	-2.0
UPGAS/B	O2-3	N2ANEAL	00:05:00	LON2FLO	34
TEMP/B	1100	DOWNGAS	N2-3	RAMPDWN	MAX
SETTIME	00:10:00				

- 4. Mask 1: BDF
 - Drybake: 15 min. @110°C
 - Spin: HMDS/ 1813 at 4Krpm
 - Prebake: 1 min @ 110°C on hotplate
 - Expose: MA6 for 5 sec
 - Develop: MF 319, 30-50 sec.
 - Postbake: 1 min. @110°C hotplate
- 5. Pattern deep boron masking oxide
 - Etch: BHF, 14 min. (until patterned areas are hydrophobic, 13 min.)
 - Rinse: DI-H₂O, 2 min.; spin dry

6. Strip photoresist

- Strip: PRS-2000 @65°C, 15 min.
- Rinse: DI-H₂O, 3 min.; spin dry
- Etch: BHF, 12 sec.
- Rinse: DI-H₂O, 2 min.; spin dry (inspect for traces of resist!)
- 7. Pre-furnace clean
 - Clean one monitor; store in B1 for step 9
- 8. Deep boron diffusion (A2)
 - Recipe: BEDEP99; Parameter table: BORON99

Run number: A2-

TEMP	1175	LOO2FLOW	150	HIGHSET	2.0
SETTIME	00:10:00	DLUTTIME	00:10:00	LOWSET	-2.0
DEPTIM	05:00:00	PUL-60	200		

- 9. Drive-in and dilute boron glass (A4)
 - Recipe: N2ANL/OX; Parameter table: N2ANL/OX
 - Add the bare Si monitor wafer previously stored in B1 to this run

	Run number: A2									
UPGAS	N2-3	ANLTIM E	05:00:00	N2ANEAL	00:05:00					
DOWNGAS	N2-3	TEMP2	1000	PUL-600	200					
TEMP1	1175	DRY1	00:05:00	SETTLE	SPKSET					
TEMPRMP	MAX	WET	00:20:00	LOWSET	-2.0					
SETTLE	00:10:00	DRY2	00:05:00	HIGHSET	2.0					
				UPGAS2	N2-3					

Required Monitor Data [Expected thickness = 1500 Å]

Location	Тор	Center	Left	Right	Bottom	Average
Thickness						

10. Strip deep boron masking oxide

- Etch: HF:H₂O 1:1, ____7 min. (until field/backsides are hydrophobic, ≅ 7 min.)
- Rinse: DI-H₂O, 3 min.; spin dry

Checkpoint #1

Inspect for boron pitting. Discard wafers which do not pass QC check

11. Pre-furnace clean

• Clean 1 monitor for B2

12. Grow shallow boron diffusion (A4)

• Recipe: DWDA1; Parameter Table: OXIDIZE

Run number: A4-____

UPGAS/A	N2-3	DRY1	00:10:00	PUL-600	200
DOWNGAS	N2-3	WET	01:50:00	SETTLE	SPKSET
TEMP	1000	DRY2	00:10:00	HIGHSET	2.0
TEMPRAM	MAX	N2ANEAL	00:05:00	LOWSET	-2.0
SETTIME	00:10:00				

Required Monitor Data [Expected thickness = 6,000 Å]

Location	Тор	Center	Left	Right	Bottom	Average
Thickness						

13. Mask 2: SHB

- Drybake: 15 min. @110°C
- Spin: HMDS/ 1813 @4Krpm
- Prebake: 30 min. @ 90°
- Expose:___13____ (MA6 ≅13 sec. EV420 ≅8 sec.)
- Develop: MF 319, 1.1min.
- Rinse: DI-H₂O, 2 min.; spin dry
- Postbake: 15 min. @110°C

14. Pattern shallow boron masking oxide

- Etch: BHF, __6___ min. (until patterned areas are hydrophobic, ~6 min.)
- Rinse: DI-H₂O, 2 min.; spin dry

15. Strip photoresist

- Strip: PRS-2000 @65°C, 15 min.
- Rinse: DI-H₂O, 3 min.; spin dry
- Etch: BHF, 12 sec.
- Rinse: DI-H₂O, 2 min.; spin dry (inspect for traces of resist!)

16. Pre-furnace clean

17. Shallow boron diffusion (A2)

• Recipe: BDEP99; Parameter table: BORON99

Run number: A2-

ТЕМР	1175	LOO2FLOW	150	HIGHSET	2.0
SETTIME	00:10:00	DLUTTIME	00:10:00	LOWSET	-2.0
DEPTIM	00:15:00	PUL-60	200		

18. Strip deep boron masking oxide

- Etch: HF:H₂O 1:1, ____3 min. (until field/backsides are hydrophobic, ≅ 3 min.)
- Rinse: DI-H₂O, 3 min.; spin dry

Checkpoint #2

Inspect for boron pitting. Discard wafers which do not pass QC check

19. Pre-furnace clean

• Clean 3 monitor; store 1 in B1 for step 20, 2 monitor for A4 & C4

20. Deposit LPCVD dielectrics (C4)

- Recipe: O2/NR/O2; Parameter table; CVD
- Add one of the bare Si monitor wafers previously stored in B1 to this

Run number: C4-

Oxide	3000Å	Nitride	1500 Å –	Oxide	3000 Å
HTOTIM		NITRTIM		HTOTIM	

21. Daimond seeding

- Ultrasonic diamond powder loaded water (DW) for 30 minutes (average powder size 25nm)
- Dip wafer in 5% HF for 30 sec.
- DI-H2O 2min, blow dry
- Mount wafer on spinner and drop DW on
- Spin at 2000rpm for 30 sec.

22. Growing CVD polycrystalline-diamond at MSU

	CH4/H2 (sccm)	Trimethyl- boron (sccm)	Pressure (torr)	Temp. (°C)	Time (hr)	Thickness (um)
Lightly doped	3/200	3e-4	45	740	18	1.8
Highly doped	3/200	1e-2	45	740	2.6	0.2

• Table of parameters for poly-C growth

23. Mask for Ti film lift-off: HDC (for etching highly doped diamond)

- Spin HMDS 500 4 sec/4k 10 sec
- Spin 5214 4 sec/4k 30 sec
- Oven 90°C for 30 minutes
- 1st exposure: 2-6sec MA 6
- Reversal Bake: Oven 102°C, 30 minutes
- Flood Exposure 50 sec
- Develop MIF319, 1 min (bath)
- Rinse in DI- H_2O , 5 min. spin dry

24. Deposit Ti film

- Loaded into Enerjet Evaporator, pump down to 2*10⁻⁶ Torr (1 hr)
- Evaporate 100 nm Ti

25. Lift off

- Soak in Acetone over night with wafer in holder. Wafers face down
- Ultrasound each wafer individually for a few sec the next morning
- Rinse: DI-H₂O, 5 min.; spin dry (inspect for flakes!)

26. Dry etching of highly doped diamond layer at MSU

O2/Ar/SF6 (sccm)	Pressure (mtorr)	Microwave power (W)	RF power (W)	Substrate bias (V)	Etch Rate (nm/min)	Thickness Etched (um)
30/2/2	4-6	400	100	-130	80-120	0.2 -0.3

• Diamond etching recipe:

• After each 30 second etch, check the change of sheet resistance using fourprobe method. Etch till the highly doped layer is totally removed 27. Strip Ti etching mask

- Strip: 5% HF solution, 5 min.
- Rinse: DI-H₂O, 3 min.; spin dry

28. Mask for Ti film lift-off: DIA (for etching diamond sensor)

- Spin: HMDS/ 1827 @3Krpm
- Prebake: 30 min. @ 90°
- Expose:__17__ sec using MA6
- Develop: MF 319, 1.1min.
- Rinse: DI-H₂O, 2 min.; spin dry
- Postbake: 30 min. @110°C

29. Deposit Ti film

- Loaded into Enerjet Evaporator, pump down to 2*10⁻⁶ Torr (1 hr)
- Evaporate 150 nm Ti

30. Lift off

- Soak in Acetone over night with wafer in holder. Wafers face down
- Ultrasound each wafer individually for a few sec the next morning
- Rinse: DI-H₂O, 5 min.; spin dry (inspect for flakes!)

31. Dry etching of both diamond layers at MSU

•	Diamond	etching	recipe:
-	Diamond	otoning	reeipe.

O2/Ar/SF6 (sccm)	Pressure (mtorr)	Microwave power (W)	RF power (W)	Substrate bias (V)	Etch Rate (nm/min)	Thickness Etched (um)
30/2/2	4-6	400	100	-130	80-120	~2

32. Strip Ti etching mask

- Strip: 5% HF solution, 5 min.
- Rinse: DI-H₂O, 3 min.; spin dry

33. LTO deposition (furnace C2)

- Deposit 10,000Å low temperature oxide at 425°C
- Deposition rate: ~100Å/min

34. Mask for contact to diamond: PCC

- Spin: HMDS/ 1813 @3Krpm
- Prebake: 1 min. @ 110° on hotplate
- Expose: 5-7 sec using MA6
- Develop: MF 319, 50 sec.
- Rinse: DI-H₂O, 2 min.; spin dry
- Postbake: 1 min. @110°C on hotplate

35. Etch LTO

- Pad Etchant (expect ~3000Å/min)
- Rinse: DI-H₂O, 2 min.; spin dry

36. Strip photoresist

- Strip: PRS-2000 @65°C, 15 min.
- Rinse: DI-H₂O, 3 min.; spin dry

37. Mask for interconnects: DCO2

- Spin: HMDS/ SPR220 @3Krpm
- Prebake: 1.5 min. @ 110° on hotplate
- Expose: 7_ sec using MA6
- Postbake: 1.5 min. @ 115° on hotplate
- Develop: AZ300, 60 sec.
- Rinse: DI-H₂O, 2 min.; spin dry

38. Asher for descum

39. Sputter Interconnect Metal

- Rinse DI-H₂O, 1 min. dip in BHF immediately
- Etch: BHF, 20 sec.
- Rinse: DI-H₂O, 2 min.; spin dry
- Drybake: 5 min. @110°C; load into sputtering chamber immediately
- Pump down to $3*10^{-6}$ Torr (1.5 hrs)
- Target #1, DC 550 W, 500 Å Ti _____ (10' 40'')
- Target #1, DC sputter 550W, 1200 Å TiN (N₂ gas 13.5%): _____ (16' 40")
- Target #3, DC 550 W, 800 Å Al (1% Si) _____ (4' 20")
- Target #1, DC 550 W, 300 Å Ti _____ (6' 40'')
- Target #3, DC 550 W, 800 Å Al (1% Si) _____ (4' 20")
- Target #1, DC 550 W, 300 Å Ti _____ (6' 40")
- Target #3, DC 550 W, 800 Å Al (1% Si) _____ (4' 20")

• Target #1, DC 550 W, 300 Å Ti _____ (6' 40'')

40. Lift off

Option1

- Soak in Acetone over night with wafer in holder. Wafers face down.
- Ultrasound each wafer individually for a few sec the next morning
- Rinse: DI-H₂O, 5 min.; spin dry (inspect for flakes!)

Option 2

- Liftoff: 1112A Remover @60-70°C, ≈10-15 min.
- Punctuate with 1-2 min. intervals in ultrasonic bath
- Rinse: DI-H₂O, 5 min.; spin dry (inspect for flakes!)
- 41. LTO deposition (furnace C2)
 - Deposit 10,000Å low temperature oxide at 425°C
 - Deposition rate: ~100Å/min
- 42. Mask of contact for both Ir and Au: CON
 - Spin: HMDS/ 1813 @3Krpm
 - Prebake: 1 min. @ 110° on hotplate
 - Expose: 5-7 sec using MA6
 - Develop: MF 319, 50 sec.
 - Rinse: DI-H₂O, 2 min.; spin dry
 - Postbake: 1 min. @110°C on hotplate

43. Etch LTO

- Pad Etchant
- Careful control (expect ~3000Å/min) to avoid etching of underneath Al/Ti
- Rinse: DI-H₂O, 2 min.; spin dry

44. Strip photoresist

- Strip: PRS-2000 @65°C, 15 min.
- Rinse: DI-H₂O, 3 min.; spin dry

45. Mask for patterning Ir stimulating site: IRD

- Spin: HMDS/ SPR220 @3Krpm
- Prebake: 1.5 min. @ 110° on hotplate
- Expose: 7_ sec using MA6
- Postbake: 1.5 min. @ 115° on hotplate
- Develop: AZ300, 60 sec.

• Rinse: DI-H₂O, 2 min.; spin dry

46. Asher for descum

47. Sputter Ti/ Ir

- Rinse: DI-H₂O, 2 min.; spin dry
- Drybake: 5 min. @110°C; load into sputtering chamber immediately
- Pump down to $5*10^{-6}$ Torr (1.5 hrs)
- Target #2, RF sputter 500 Å Ti @700W, 7 mT Ar, (pre-sputter 2 min.), time _____(11')
- Target#3, DC sputter 3000Å Ir @1.0 A, 7 mT Ar, (pre-sputter 2 min.), time _____(21'45")

48. Lift off

- Soak in Acetone over night with wafer in holder. Wafers face down.
- Ultrasound each wafer individually for a few sec the next morning
- Rinse: DI-H₂O, 5 min.; spin dry (inspect for flakes!)

49. Mask for patterning Au pads: GOL

- Spin: HMDS/ SPR220 @3Krpm
- Prebake: 1.5 min. @ 110° on hotplate
- Expose: 7_ sec using MA6
- Postbake: 1.5 min. @ 115° on hotplate
- Develop: AZ300, 60 sec.
- Rinse: DI-H₂O, 2 min.; spin dry

50. Asher for descum

51. Sputter Cr/Au

- Rinse: DI-H₂O, 2 min.; spin dry
- Drybake: 5 min. @110°C; load into sputtering chamber immediately
- Pump down to 5*10⁻⁶ Torr (1.5 hrs)
- Target #2, RF sputter 500 Å Cr @800W, 7 mT Ar, (pre-sputter 2 min.), time _____(6'15")
- Target#3, DC sputter 2000Å Au @0.5 A, 7 mT Ar, (pre-sputter 2 min.), time _____(8')
- 52. Pattern the profile of the probe on dielectric : DEL
 - Drybake: 15 min. @110°C

- Spin: HMDS/ 1827 @3Krpm
- Prebake: 30 min. @ 90°
- Expose: MA6 \cong 55 sec or EV420 \cong 29 sec.
- Develop: MF 319, 1.1min.
- Rinse: DI-H₂O, 2 min.; spin dry
- Postbake: 30 min. @110°C

53. RIE etch field using Semigroup RIE

- Check current recipes for etch rates and times
- Check thickness of all layer

	Kun			
Etch #1: Descum		Etch #2: Oxide	Etch#3 Nitride	
Gas/Flow	O ₂ 50 sccm	CHF3 15 sccm	CF4 20 sccm	
Gas/Flow		CF4 15 sccm	O2 1 sccm	
Pressure	300 mT	40 mT	100 mT	
Power	25 W	100 W	80 W	
Time	1 min.	(~280 Å /min)	(~100 Å /min)	

49. Strip photoresist

- Strip: PRS-2000 @65°C, 15 min.
- Rinse: DI-H₂O, 3 min.; spin dry (inspect for traces of resist)

50. Protect frontside

- Spin: HMDS/1827 @ 3Krpm
- Postbake: 15 min. @110°C

51. RIE etch backside (Semi Group)

- Check current recipes for etch rates and times
- Check thickness of all layer
- Note: recipe for etching oxide can etch nitride also

Run number:

	Etch #2A: Oxide			
Gas/Flow	CHF3 25 sccm			
Gas/Flow	CF4 25 sccm			
Pressure	40 mT			
Power	100 W			
Time				

52. Strip photoresist

- Strip: PRS-2000 @65°C, 15 min.
- Rinse: DI-H₂O, 3 min.; spin dry (inspect for traces of resist)
- 53. Thinning from backside with HF-Nitric etch
 - Mount the wafer on the hot chuck using paraffin wax, let the wafer front side facing the chuck
 - Cool down in air, clean any residue of wax on the backside of wafer using acetone
 - Prepare the etchant in Teflon tub with HNO3:HF:Acetic acid = 950:95:5 ml
 - Etch in the Teflon tub with rotating chuck and agitating N2 gas bubbles.
 - Thinning the wafer to about 200 um (etch rate is about 11.5 um/min).
 - Remove the wafer from chuck on hotplate, clean with Acetone soak and IPA.

54. EDP etch for probe releasing

• Prepare the EDP solution using the recipe in the following the table, using 6X for 4 inch wafer.

Table for various EDP mixtures.

Chemical	Single batch	Double	Triple	4X	6X
DI-H2O	48ml	96ml	144ml	192ml	288ml
Catechol	48g	96g	144g	192g	288g
Pyrazine	0.9g	1.8g	2.7g	3.6g	5.4g
Ethylenediamene	150ml	300ml	450ml	600ml	900ml

- Etch at controlled temperature of 110 °C (etch rate: ~ 80 um/hr)
- Etch till all the probes are released
- Rinse the probes gently using the beaker of hot water till water is clear
- Rinse with acetone and IPA.
- Pour the probes on the filter paper till dry.
- Store the probes for testing

BIBLIOGRAPHY

BIBLIOGRAPHY

- [1] P. Adrian, "SENSOR INDUSTRY DEVELOPMENTS AND TRENDS," Sensor Business Digest, vol. pp. 2002.
- [2] <u>http://www.sensorsportal.com/HTML/Marketplace.htm</u>, 2004.
- [3] M. Aslam, I. Taher, A. Masood, M. A. Tamor and T. J. Potter, "Piezoresistivity in Vapor-Deposited Diamond Films," Applied Physics Letters, vol. 60, pp. 2923-2925, 1992.
- [4] I. Taher, 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.
- [5] M. Werner and R. Locher, "Growth and application of undoped and doped diamond films," Reports on Progress in Physics, vol. 61, pp. 1665-1710, 1998.
- [6] J. L. Davidson, W. P. Kang, Y. Gurbuz, K. C. Holmes, L. G. Davis, A. Wisitsoraat, et al., "Diamond as an active sensor material," Diamond and Related Materials, vol. 8, pp. 1741-1747, 1999.
- [7] M. Adamschik, R. Muller, P. Gluche, A. Floter, W. Limmer, R. Sauer, et al., "Analysis of piezoresistive properties of CVD-diamond films on silicon," Diamond and Related Materials, vol. 10, pp. 1670-1675, 2001.
- [8] S. Sahli and D. M. Aslam, "Ultra-high sensitivity intra-grain poly-diamond piezoresistors," Sensors and Actuators a-Physical, vol. 71, pp. 193-197, 1998.
- [9] S. Sahli, Electronic Characterization and Fabrication of CVD Diamond Piezoresistive Pressure Sensors, Ph.D Dissertation, Michigan State University, 1997.
- [10] G. S. Yang and D. M. Aslam, "Single-structure heater and temperature sensor using a p-type polycrystalline diamond resistor," IEEE Electron Device Letters, vol. 17, pp. 250-252, 1996.

- [11] J. G. Ran, C. Q. Zheng, J. Ren and S. M. Hong, "Properties and Texture of B-Doped Diamond Films as Thermal Sensor," Diamond and Related Materials, vol. 2, pp. 793-796, 1993.
- [12] W. P. Kang, Y. Gurbuz, J. L. Davidson and D. V. Kerns, "A New Hydrogen Sensor Using a Polycrystalline Diamond-Based Schottky Diode," Journal of the Electrochemical Society, vol. 141, pp. 2231-2234, 1994.
- [13] Y. Gurbuz, W. P. Kang, J. L. Davidson, D. L. Kinser and D. V. Kerns, "Diamond microelectronic gas sensors," Sensors and Actuators B-Chemical, vol. 33, pp. 100-104, 1996.
- [14] P. Bergonzo, F. Foulon, R. D. Marshall, C. Jany, A. Brambilla, R. D. McKeag, et al., "High collection efficiency CVD diamond alpha detectors," IEEE Transactions on Nuclear Science, vol. 45, pp. 370-373, 1998.
- [15] M. Keil, W. Adam, E. Berdermann, P. Bergonzo, W. de Boer, F. Bogani, et al., "New results on diamond pixel sensors using ATLAS frontend electronics," Nuclear Instruments & Methods in Physics Research Section a-Accelerators Spectrometers Detectors and Associated Equipment, vol. 501, pp. 153-159, 2003.
- [16] N. Kumar, H. Schmidt and C. G. Xie, "Diamond-Based Field-Emission Flat-Panel Displays," Solid State Technology, vol. 38, pp. 71-73, 1995.
- [17] U. Kim and D. M. Aslam, "Field emission electroluminescence on diamond and carbon nanotube films," Journal of Vacuum Science & Technology B, vol. 21, pp. 1291-1296, 2003.
- [18] D. Hong and M. Aslam, "Field-Emission from P-Type Polycrystalline Diamond Films," Journal of Vacuum Science & Technology B, vol. 13, pp. 427-430, 1995.
- [19] X. W. Zhu, D. M. Aslam, Y. X. 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.
- [20] J. Wang, J. E. Butler, D. S. Y. Hsu and T.-C. Nguyen, "CVD polycrystalline diamond high-Q micromechanical resonators," The Fifteenth IEEE international conference on MEMS, vol. pp. 657, 2002.
- [21] N. Sepulveda-Alancastro and D. M. Aslam, "Polycrystalline diamond technology for RFMEMS resonators," Microelectronic Engineering, vol. 73-74, pp. 435-440, 2004.

- [22] A. A. Morrish and P. E. Pehrsson, "Effects of Surface Pretreatments on Nucleation and Growth of Diamond Films on a Variety of Substrates," Applied Physics Letters, vol. 59, pp. 417-419, 1991.
- [23] L. Demuynck, J. C. Arnault, C. Speisser, R. Polini and F. LeNormand, "Mechanisms of CVD diamond nucleation and growth on mechanically scratched and virgin Si(100) surfaces," Diamond and Related Materials, vol. 6, pp. 235-239, 1997.
- [24] P. A. Dennig, H. Shiomi, D. A. Stevenson and N. M. Johnson, "Influence of Substrate Treatments on Diamond Thin-Film Nucleation," Thin Solid Films, vol. 212, pp. 63-67, 1992.
- [25] E. J. Bienk and S. S. Eskildsen, "The Effect of Surface Preparation on the Nucleation of Diamond on Silicon," Diamond and Related Materials, vol. 2, pp. 432-437, 1993.
- [26] F. S. Lauten, Y. Shigesato and B. W. Sheldon, "Diamond Nucleation on Unscratched Sio2 Substrates," Applied Physics Letters, vol. 65, pp. 210-212, 1994.
- [27] M. J. Chiang and M. H. Hon, "Positive dc bias-enhanced diamond nucleation with high CH4 concentration," Diamond and Related Materials, vol. 10, pp. 1470-1476, 2001.
- [28] M. D. Irwin, C. G. Pantano, P. Gluche and E. Kohn, "Bias-enhanced nucleation of diamond on silicon dioxide," Applied Physics Letters, vol. 71, pp. 716-718, 1997.
- [29] A. Masood, M. Aslam, M. A. Tamor and T. J. Potter, "Techniques for Patterning of CVD Diamond Films on Non-Diamond Substrates," Journal of the Electrochemical Society, vol. 138, pp. L67-L68, 1991.
- [30] G. S. Yang, M. Aslam, K. P. Kuo, D. K. Reinhard and J. Asmussen, "Effect of Ultrahigh Nucleation Density on Diamond Growth at Different Growth-Rates and Temperatures," Journal of Vacuum Science & Technology B, vol. 13, pp. 1030-1036, 1995.
- [31] A. P. Malshe, R. A. Beera, A. A. Khanolkar, W. D. Brown and H. A. Naseem, "Initial results of a novel pre-deposition seeding technique for achieving an ultrahigh nucleation density for CVD diamond growth," Diamond and Related Materials, vol. 6, pp. 430-434, 1997.

- [32] F. P. Bundy, H. T. Hall, H. M. STrong and R. H. Wentroff, "Man-made diamond," Nature, vol. 176, pp. 51-54, 1955.
- [33] P. K. Bachmann, D. Leera and H. Lydtin, "Towards a General Concept of Diamond Chemical Vapour Deposition," Diamond and Related Materials, vol. 1, pp. 1-12, 1991.
- [34] D. M. Gruen, P. C. Redfern, D. A. Horner, P. Zapol and L. A. Curtiss, "Theoretical studies on nanocrystalline diamond: Nucleation by dicarbon and electronic structure of planar defects," Journal of Physical Chemistry B, vol. 103, pp. 5459-5467, 1999.
- [35] M. Kamo, Y. Sato and S. Sctaka, "(MPCVD of diamond)," Journal of Crystal Growth, vol. 62, pp. 642, 1983.
- [36] B. R. Stoner, B. E. Williams, S. D. Wolter, K. Nishimura and J. T. Glass, "Insitu Growth-Rate Measurement and Nucleation Enhancement for Microwave Plasma Cvd of Diamond," Journal of Materials Research, vol. 7, pp. 257-260, 1992.
- [37] S. Matsumoto, Y. Sato, M. Kamo and N. Setaka, "(HFCVD Diamond)," Japanese Journal of applied Physics, vol. 21, pp. 183, 1982.
- [38] R. C. Hyer, M. Green, K. K. Mishra and S. C. Sharma, "Nucleation and Growth of Diamond in Hot Filament Assisted Chemical Vapor-Deposition," Journal of Materials Science Letters, vol. 10, pp. 515-518, 1991.
- [39] A. Lettington and J. W. Steeds, "Thin Film Diamond," 1994.
- [40] V. G. Ralchenko, A. A. Smolin, V. I. Konov, K. F. Sergeichev, I. A. Sychov, I. I. Vlasov, et al., Diamond and Related Materials, vol. 6, pp. 417, 1997.
- [41] Y. Ando, Y. Yokota, T. Tachibana, A. Watanabe, Y. Nishibayashi, K. Kobashi, et al., Diamond and Related Materials, vol. 11, pp. 596, 2002.
- [42] S. Sahli and D. M. Aslam, "Effect of postdeposition anneal on the resistivity of ptype polycrystalline diamond films," Applied Physics Letters, vol. 69, pp. 2051-2052, 1996.
- [43] X. K. Zhang, J. G. Guo and Y. F. Yao, "Boron Doping of Diamond Films by B203 Vaporization," Physica Status Solidi a-Applied Research, vol. 133, pp. 377-383, 1992.

- [44] J. Ruan, K. Kobashi and W. J. Choyke, "Effect of Oxygen on Boron Doping in Chemical Vapor-Deposition of Diamond as Deduced from Cathodoluminescence Studies," Applied Physics Letters, vol. 60, pp. 1884-1886, 1992.
- [45] T. Takada, T. Fukunaga, K. Hayashi, Y. Yokota, T. Tachibana, K. Miyata, et al., "Boron-doped diamond thin film sensor for detection of various semiconductor manufacturing gases," Sensors and Actuators a-Physical, vol. 82, pp. 97-101, 2000.
- [46] J. Cifre, J. Puigdollers, M. C. Polo and J. Esteve, "Trimethylboron Doping of Cvd Diamond Thin-Films," Diamond and Related Materials, vol. 3, pp. 628-631, 1994.
- [47] J. F. Prins, "Ion-Implantation and Diamond Some Recent Results on Growth and Doping," Thin Solid Films, vol. 212, pp. 11-18, 1992.
- [48] J. F. Prins, "Implantation-doping of diamond with B+, C+, N+ and O+ ions using low temperature annealing," Diamond and Related Materials, vol. 11, pp. 612-617, 2002.
- [49] R. Kalish, "Ion-implantation in diamond and diamond films: Doping, damage effects and their applications," Applied Surface Science, vol. 117, pp. 558-569, 1997.
- [50] T. H. Borst and O. Weis, "boron-doped homoepitaxial Diamond layers: Fabrication, characterisation and electronic applications," phys. stat. sol. A, vol. 154, pp. 423, 1996.
- [51] E. Kohn, W. Ebert, A. Aleksov, M. Adamschik and P. Schmid, "Diamond Technology for Electronics and MEMS Review of Status and Perspectives," 23rd Intern'l Conf. on Microelectronics, vol. 1, pp. 59, 2002.
- [52] J. Ristein, M. Riedel, F. Maier, B. F. Mantel, M. Stammler and L. Ley, "Surface doping: a special feature of diamond," Journal of Physics-Condensed Matter, vol. 13, pp. 8979-8987, 2001.
- [53] E. Snidero, D. Tromson, C. Mer, P. Bergonzo, J. S. Foord, C. Nebel, et al.,
 "Influence of the postplasma process conditions on the surface conductivity of hydrogenated diamond surfaces," Journal of Applied Physics, vol. 93, pp. 2700-2704, 2003.
- [54] M. Werner, O. Dorsch, H. U. Baerwind, A. Ersoy, E. Obermeier, C. Johnston, et al., "Very-Low Resistivity Al-Si Ohmic Contacts to Boron-Doped Polycrystalline Diamond Films," Diamond and Related Materials, vol. 3, pp. 983-985, 1994.

- [55] M. Yokoba, Y. Koide, A. Otsuki, F. Ako, T. Oku and M. Murakami, "Carrier transport mechanism of Ohmic contact to p-type diamond," Journal of Applied Physics, vol. 81, pp. 6815-6821, 1997.
- [56] C. A. Hewett, M. J. Taylor, J. R. Zeidler and M. W. Geis, "Specific Contact Resistance Measurements of Ohmic Contacts to Semiconducting Diamond," Journal of Applied Physics, vol. 77, pp. 755-760, 1995.
- [57] Y. X. Tang and D. M. Aslam, "Technology of Polycrystalline Diamond Thin Films for Microsystems Applications," Journal of Vacuum Science & Technology B, vol. 23, pp. 1088, 2005.
- [58] P. E. Viljoen, E. S. Lambers and P. H. Holloway, "Reaction between Diamond and Titanium for Ohmic Contact and Metallization Adhesion Layers," Journal of Vacuum Science & Technology B, vol. 12, pp. 2997-3005, 1994.
- [59] T. Iwasaki, K. Okano, Y. Matsumae, E. Matsushima, H. Maekawa, H. Kiyota, et al., "Formation of Ohmic Contacts on Semiconducting Diamond Grown by Chemical-Vapor-Deposition," Diamond and Related Materials, vol. 3, pp. 30-34, 1994.
- [60] S. Katsumata, Y. Oobuchi and T. Asano, "Patterning of Cvd Diamond Films by Seeding and Their Field-Emission Properties," Diamond and Related Materials, vol. 3, pp. 1296-1300, 1994.
- [61] X. Jiang, E. Boettger, M. Paul and C. P. Klages, "Approach of Selective Nucleation and Epitaxy of Diamond Films on Si(100)," Applied Physics Letters, vol. 65, pp. 1519-1521, 1994.
- [62] H. W. Liu, C. X. Gao, X. Li, C. X. Wang, Y. H. Han and G. T. Zou, "Selective deposition of diamond films on insulators by selective seeding with a double-layer mask," Diamond and Related Materials, vol. 10, pp. 1573-1577, 2001.
- [63] O. Dorsch, M. Werner and E. Obermeier, "Dry-Etching of Undoped and Boron-Doped Polycrystalline Diamond Films," Diamond and Related Materials, vol. 4, pp. 456-459, 1995.
- [64] M. Bernard, A. Deneuville, T. Lagarde, E. Treboux, J. Pelletier, P. Muret, et al., "Etching of p- and n-type doped monocrystalline diamond using an ECR oxygen plasma source," Diamond and Related Materials, vol. 11, pp. 828-832, 2002.

- [65] C. Vivensang, L. FerlazzoManin, M. F. Ravet, G. Turban, F. Rousseaux and A. Gicquel, "Surface smoothing of diamond membranes by reactive ion etching process," Diamond and Related Materials, vol. 5, pp. 840-844, 1996.
- [66] R. Otterbach and U. Hilleringmann, "Reactive ion etching of CVD-diamond for piezoresistive pressure sensors," Diamond and Related Materials, vol. 11, pp. 841-844, 2002.
- [67] H. Shiomi, "Reactive ion etching of diamond in O-2 and CF4 plasma, and fabrication of porous diamond for field emitter cathodes," Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers, vol. 36, pp. 7745-7748, 1997.
- [68] F. Silva, R. S. Sussmann, F. Benedic and A. Gicquel, "Reactive ion etching of diamond using microwave assisted plasmas," Diamond and Related Materials, vol. 12, pp. 369-373, 2003.
- [69] C. S. Smith, "Piezoresistance effect in germanium and silicon," Physical Review., vol. 94, pp. 22, 1954.
- [70] G. C. Kucznski, "Effect of elastic strain on the electrical resisstance of metals," Physical Review, vol. 94, pp. 1954.
- [71] F. Conti, B. Morten, C. Nobili and A. Taroni, "Piezoresistive Coefficients in Silicon Diffused Layers," Physica Status Solidi a-Applied Research, vol. 17, pp. K29-K31, 1973.
- [72] Y. Onuma and K. Sekiya, "Piezoresistive Properties of Polycrystalline Silicon Thin-Film," Japanese Journal of Applied Physics, vol. 11, pp. 20-&, 1972.
- [73] J. S. Shor, D. Goldstein and A. D. Kurtz, "Evaluation of β-SiC for sensors," IEEE Proceedings, Intl. Conf. on Solid-State Sensors and Actuators (Transducers'91), vol. pp. 912, 1991.
- [74] D. R. Wur and J. L. Davidson, "Piezoresistivity of Polycrystalline Diamond Films," 1992 MRS meeting, vol. pp. 1992.
- [75] M. Deguchi, N. Hase, M. Kitabatake, H. Kotera, S. Shima and H. Sakakima, "Piezoresistive properties of p-type CVD diamond films," Diamond Films and Technology, vol. 6, pp. 77-85, 1996.
- [76] I. Taher, CVD Diamond Piezoresistive Microsensors, Ph.D. Dissertation, Michigan State University, 1994.

- [77] C. Herring and E. Vogt, "Transport and deformation-potential theory for manyvalley semiconductors with anisotropic scattering," Physical Review, vol. 101, pp. 944-961, 1956.
- [78] R. N. Thurston, "Use of Semiconductor Transducers in Measuring Strain, Accelerations, and Displacemnts," Physical Acoustics, vol. 1, pp. 215, 1964.
- [79] 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.
- [80] P. J. French, "Piezoresistance in polysilicon," Electronics Letter, vol. 24, pp. 999-1000, 1984.
- [81] O. Dorsch, K. Holzner, M. Werner, E. Obermeier, R. E. Harper, C. Johnston, et al., "Piezoresistive Effect of Boron-Doped Diamond Thin-Films," Diamond and Related Materials, vol. 2, pp. 1096-1099, 1993.
- [82] Cochlear Implant Program, University of Michigan, http://www.med.umich.edu/oto/ci/implant.htm.,
- [83] J. Wang, M. Gulari, P. T. Bhatti, B. Y. Arcand, K. Beach, C. R. Friedrich, et al., "A Cochlear Electrode Array with Build-in Position Sensing," 18th IEEE Intl. Conf. on Micro Electro Mechanical Systems (MEMS 2005), vol. 2, pp. 786-789, 2005.
- [84] P. T. Bhatti, B. Y. Arcand, J. Wang, N. V. Butala, C. R. Friedrich and K. D. Wise, "A High-Density Electrode Array for A Cochlear Prosthesis," IEEE Int. Conf. on Solid-State Sensors and Actuators (TRANSDUCERS'03), vol. 2, pp. 1750-1753, 2003.
- [85] R. R. Thomas, F. B. Kaufman, J. T. Kirleis and R. A. Belsky, "Wettability of polished silicon oxide surfaces," Journal of the Electrochemical Society, vol. 143, pp. 643-648, 1996.
- [86] S. Schelz, C. F. M. Borges, L. Martinu and M. Moisan, "Diamond nucleation enhancement by hydrofluoric acid etching of silicon substrate," Diamond and Related Materials, vol. 6, pp. 440-443, 1997.
- [87] J. Stiegler, Y. vonKaenel, M. Cans and E. Blank, "Space filling by nucleation and growth in chemical vapor deposition of diamond," Journal of Materials Research, vol. 11, pp. 716-726, 1996.

- [88] K. Hayashi, S. Yamanaka, H. Watanabe, T. Sekiguchi, H. Okushi and K. Kajimura, Journal of Applied Physics, vol. 81, pp. 744, 1997.
- [89] 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, 1995.
- [90] N. Sepulveda-Alancastro, *Polycrystalline Diamond RF MEMS Resonator Techonology and Chracterization*, Ph.D. Dissertation, Michgan State University, 2005.
- [91] G. S. Yang and M. Aslam, "Ultrahigh Nucleation Density for Growth of Smooth Diamond Films," Applied Physics Letters, vol. 66, pp. 311-313, 1995.
- [92] Y. X. Tang, D. M. Aslam, J. B. Wang and K. D. Wise, "Study of polycrystalline diamond piezoresistive position sensors for application in cochlear implant probe," Diamond and Related Materials, vol. 15, pp. 199-202, 2006.
- [93] X. M. Yu, J. Thaysen, O. Hansen and A. Boisen, "Optimization of sensitivity and noise in piezoresistive cantilevers," Journal of Applied Physics, vol. 92, pp. 6296-6301, 2002.
- [94] Y. X. Tang, D. M. Aslam, J. B. Wang and K. D. Wise, "Technology and Integration of Poly-Crystalline Diamond Piezoresistive Position Sensor for Cochlear Implant Probe," the 13th International Conference on Solid-State Sensors, Actuators and Microsystems (TRANSDUCERS '05), vol. 1, pp. 543-546, 2005.
- [95] O. Auciello, J. Birrell, J. A. Carlisle, J. E. Gerbi, X. C. Xiao, B. Peng, et al., "Materials science and fabrication processes for a new MEMS technoloey based on ultrananocrystalline diamond thin films," Journal of Physics-Condensed Matter, vol. 16, pp. R539-R552, 2004.
- [96] M. Adamschik, M. Hinz, C. Maier, P. Schmid, H. Seliger, E. P. Hofer, et al.,
 "Diamond micro system for bio-chemistry," Diamond and Related Materials, vol. 10, pp. 722-730, 2001.
- [97] J. Rabaey, "Digital Integrated circuits: A Design Perspective," vol. pp. 2003.
- [98] Y. X. Tang, S. Sahli, D. M. Aslam, D. Merriam and K. D. Wise, "Poly-diamond inter-and intra-grain piezoresis ive position sensor design for WIMS," AICHE Annual Conference on Sensors, vol. pp. 186, 2002.

- [99] J. H. Lee, J. Y. Lee, I. S. Yeo and S. K. Lee, "Lower sheet/contact resistance in shallower junction obtained by F+B mixed implant," Journal of Vacuum Science & Technology B, vol. 20, pp. 396-399, 2002.
- [100] H. Shiomi, "Reactive Ion Etching of Diamond in O2 and CF4 plasma, and Fabrication of Porous Diamond for Field Emitter Cathodes," Japanese Journal of applied Physics, vol. 36, pp. 7745-7748, 1997.

